Studies on transport and granulation properties of superphosphate pulps

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Introduction

In the manufacturing process of phosphate and multicomponent fertilizers the main component or one of the components included in the NPK blend subjected to granulation may be present in the form of a pulp. This pulp is usually a suspension of inorganic salts in a saturated solution of these salts, which on contact with the recycle (undersize and oversize particles after size reduction) or with the recycle and a fresh solid mixture crystallizes forming product granulation nuclei, and also enlarges the existing circulating granules. The study of physicochemical properties of the pulp is necessary if the efficiency of the granulation process is to be maintained for an extended period of time. During the investigations it must be taken into account that the properties of the pulp may often change after extended storage, and problems may arise with pulp feeding or with changes in its chemical composition.

The pulp is prepared in reactors of various design, equipped with means for agitating the suspension, and with a cooling/heating jacket for maintaining temperature at the required level. In a study on mixing processes a stand for measuring the mixing power in vibration mixers was presented [1].

This setup was used to determine the properties of process pulps. Data presented in literature indicate that the measurement of the mixing power, as well as the mixing processes themselves, are very complex and are affected by many parameters, such as the type of substance being mixed, mixing vessel geometry, type of stirrer, etc. [2]. Our case is that of non-homogenous mixtures comprising solid particles suspended in a saturated solution of pulp components. In addition, pulps usually tend to change their properties with time and with temperature variations. In view of the fact that the values of power, torque of the shaft, and rotational speed are strictly interdependent, it was assumed that the operating principle of the measuring system of the laboratory setup for investigating changes in consistency may be based on the measurement of stirrer torque. The value of the torque at a given moment was determined from the difference of readouts made under load and when idling at the same rotational speed of the stirrer [3]. This method of measurement is not very accurate, but it proved sufficient for this type of systems.

This paper presents the results of investigations on the variation of the consistency of pulps: TSP, MSP and USP, with water content, temperature and phosphate/pulping agent ratio, based on the measurement of mixing torque in the dedicated setup. Based on the results obtained in each of the tests, graphs were plotted illustrating changes in torque and temperature with time, and the content of water necessary to ensure adequate pulp consistency was determined. Laboratory granulation tests were performed on selected pulps.

Laboratory setup

The setup for determining the consistency of granulation pulps is shown in Figure 1. The setup comprises a variable speed stirrer with torque measuring means (fitted with an electronic system for maintaining constant speed irrespective of the changing load), a reactor with a heating jacket and a temperature sensor. A paddle stirrer was used (number of blades: 2, diameter 70 mm, blade inclination angle 10°).

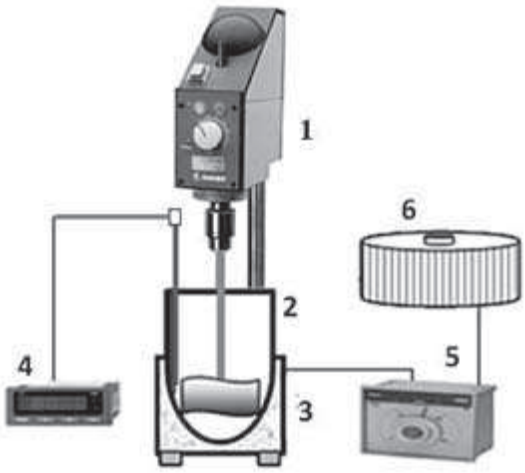

Fig. 1. Laboratory set diagram for pulp consistency test. 1 – mechanical stirrer with torque measurement and speed regulation HEIDOLPH RZR 2052, 2 – reactor, 3 – heating jacket, 4 – temperature indicator, 5 – temperature controller, 6 – autotransformer

The examination of pulp consistency was based on recording the values of stirrer torque. The mixing speed was 200 rpm. Based on granulation experiments and visual evaluation, it was assumed that a torque of less than 4 Nm corresponded to adequate pulp consistency. Every time 500 g of pulp were prepared in the reactor and stirrer torque was measured within a period of several hours during the digestion of phosphate rock.

Test result

Investigations of the consistency of selected granulation pulps.

Here we present the results of investigations on the changes in the consistency of the pulps used in the manufacture of phosphate and multicomponent fertilizers. The pulps tested included TSP 48, MSP 30 and USP.

TSP pulp

The TSP 48 pulp enables the production of triple superphosphate with a P_2O_5 content of 48wt.%. The pulp is obtained by decomposing phosphate rock with phosphoric acid. Water is also added in a quantity that ensures proper consistency of the pulp enabling it to be fed (pumped) to the granulator. Phosphate rock was fed in portions of 50 g.

TSP 48 pulp composition:

- "Tunisia" phosphate rock (29% P_2O_5) – 200 g
- phosphoric acid (48% P_2O_5) – 250 g

Test results are presented by a graph illustrating the changes in mixing torque and temperature during the decomposition process (Fig. 2). The data obtained showed that the addition of the first portion of phosphate rock did not change the mixing torque. Addition of a second portion of phosphate rock increased the temperature

to 37°C. After 20 minutes from the moment of feeding phosphate rock, 15 g of water were added to the reactor to reduce the torque. It decreased from 8 Nm to 2 Nm. Addition of another portion of phosphate rock caused a rapid increase of torque to 18.5 Nm and problems in mixing the pulp. After adding more water the pulp was liquefied and the mixing power again dropped to about 2 Nm. The second portion of water of 15 g was insufficient, the pulp thickened further (increase in mixing power) and therefore additional amount of water was added. After adding a third portion of phosphate rock, the temperature increased to about 41°C. The fourth portion of phosphate rock did not change the temperature. However, there was further thickening of the pulp, and to counteract that, a total of 30 g of water were added. The stirrer torque decreased to about 2.5 Nm and the pulp was considered pumpable. The total amount of water added in the process of obtaining TSP pulp by digesting phosphate rock with phosphoric acid was 100 g, the total water content in pulp was about 41 wt.%, and the maximum temperature was 41°C.

Another test consisted in obtaining the TSP pulp by decomposing phosphate rock with phosphoric acid at the temperature of 55°C. Phosphate rock was fed in four portions of 50 g each. Changes in torque and temperature during the decomposition are presented in Figure 3. The mass fraction of water in the pulp, as in test no. 1, was about 41 wt.%. Towards the end of the process (after 130 minutes) pulp heating was discontinued in order to determine the effect of temperature on changes in pulp consistency. The torque and temperature variation curve shows that after 30 minutes from the moment of stopping heating, the temperature in the reactor dropped from 64°C to 42°C, and the torque increased by about 2 Nm, indicating that while the temperature decreases, the obtained pulp becomes thicker to such degree that it is no longer transportable.

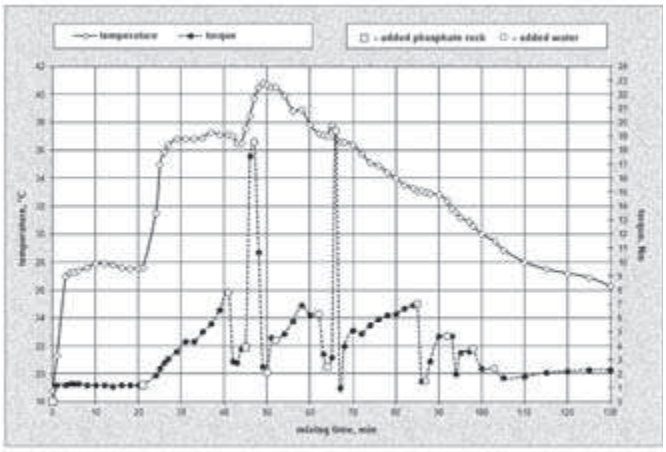

Fig. 2. Torque and temperature changes during production of TSP 48 pulp (test no. 1)

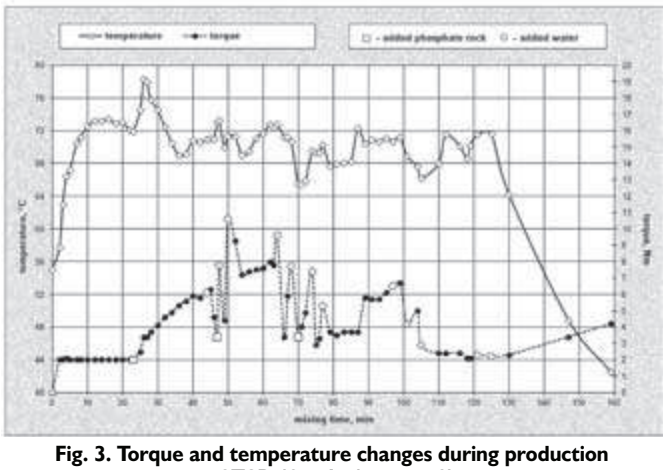

Fig. 3. Torque and temperature changes during production of TSP 48 pulp (test no. 2)

MSP 30 pulp

The MSP 30 pulp enables the production of superphosphate with a P_2O_5 content of 30 wt.%. The pulp is obtained by decomposing phosphate rock with a mixture of phosphoric and sulphuric acids. Water is also added in a quantity that enables the pulp to be fed (transported). Phosphate rock was fed in portions of about 82 g each, every 10-12 minutes. Changes in torque and temperature during the production of the MSP 30 pulp are presented in Figure 4.

MSP 30 pulp composition:

- "Tunisia" phosphate rock (29 % P2O5) – 247 g
- phosphoric acid (48% P2O5) – 110,2 g
- sulphuric acid (95% H_2SO_4) – 137,9 g

In the first step, solutions of sulphuric and phosphoric acids were introduced into the reactor, which caused temperature increase to 63°C. In the subsequent step of pulp production (phosphate material decomposition) the temperature was retained at 70-80°C with no additional heating. A total of 150 g of water was added portionwise during the test. The total water content in pulp was about 32 wt.%. That amount of water helped retain proper consistency of the pulp and constant mixing conditions, also after decreasing pulp temperature to 28°C.

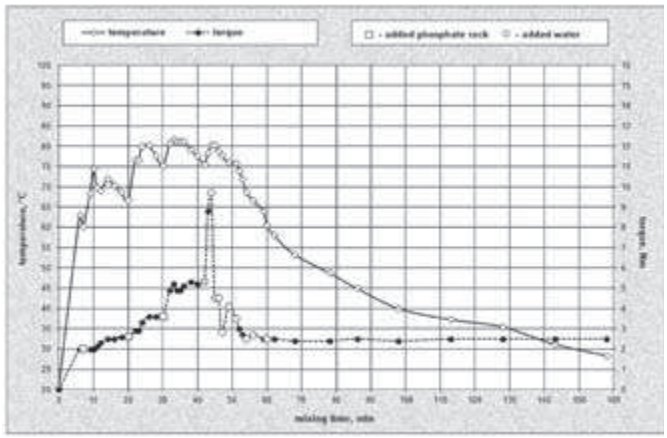

Fig. 4. Torque and temperature changes during production of MSP 30 pulp (test no. 1)

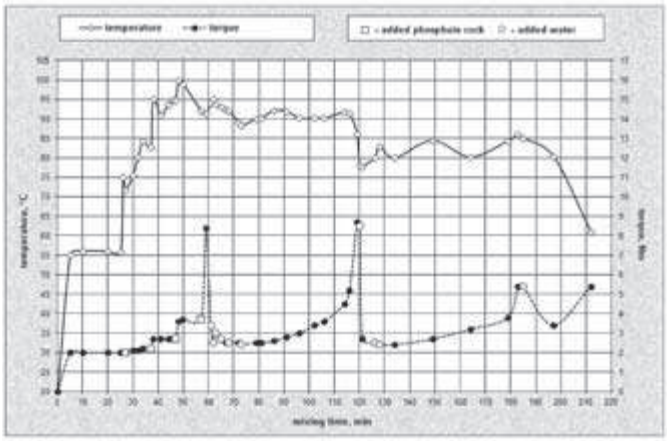

Fig. 5. Torque and temperature changes during production of MSP 30 pulp (test no. 2)

Another test consisted in obtaining MSP pulp with the initial decomposition temperature being 90°C, subsequently reduced to 80°C. Test results are shown in Figure 5. Upon mixing the solutions of phosphoric and sulphuric acids the temperature increased to 55°C. The reactor was heated further to the desired temperature and portionwise feeding of phosphate rock was commenced. The elevated temperature in the reactor resulted in water losses due to evaporation. A total of 250 g of water was added during the process. The theoretical total water content in the system was about 46 wt.%, but because of evaporation losses it was lower. The graph shows that after the pulp was liquefied (75th and 130th minutes) there was a 15 minutes period

of stabilised mixing power, after which the torque tended to increase due to water evaporation. In relation to the previous test, to make up for the high reaction temperature, an additional amount of 14% of water was added to maintain proper consistency of the pulp.

USP pulp

The USP pulp enables the production of a fertilizer with N content of 21 wt.% and P_2O_5 content of 10 wt.%. The pulp is obtained by decomposing phosphate rock with a solution of urea in sulphuric acid (3.6:1 molar ratio) and adding water in a quantity required to provide proper consistency. Water was introduced at an initial stage of preparing the reaction solution. Due to the properties of the USP pulp, adding small amount of water to the pulp becoming increasingly thick during decomposition, produced an opposite effect to the intended and caused deterioration of consistency (this was caused by the binding of water in the crystalline structure of calcium sulphate included in the pulp). The results presented in the paper illustrate the consistency changes during USP pulp production with water mass fraction amounting to 16 wt.% (test no. 1) and 18% wt.% (test no. 2), respectively.

Pulp composition:

- urea – 187.1 g
- 95% sulphuric acid – 90.5 g
- "Morocco" phosphate rock (31% P2O5) – 127.1 g
- water – 71.5 g (test no. 1)
- water – 88 g (test no. 2)

An aqueous solution of sulphuric acid and urea was prepared initially. The solution obtained was used upon heating to 65°C in the second step of pulp preparation – digestion of ground phosphate rock. Phosphate rock was fed in 5 portions at intervals of 3-5 minutes. The temperature and stirrer torque were recorded. The results of the tests performed are given in Figures 6 and 7.

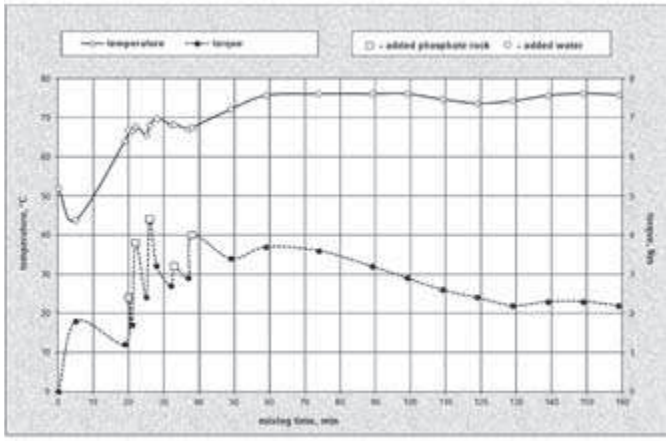

Fig. 6. Torque and temperature changes during production of USP pulp (test no. 1)

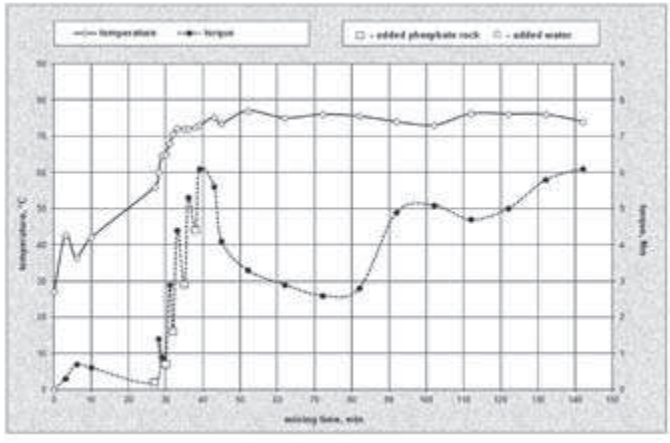

Fig. 7. Torque and temperature changes during production of USP pulp (test no. 2)

In both tests the digestion process proceeded in a stable manner, there was a temporary abrupt increase of torque of 1-2 Nm every time a new portion of phosphate rock was added. After all of phosphate rock was added, the pulp was maintained at about 75°C. In test no.1, an increase of torque (by more than 6 Nm) and thickening of the pulp were observed after 80 minutes. Despite the fluid consistency of the pulp, with water content at about 16 wt.%, problems with feeding the pulp might have occurred. In test no. 2, where the water content in the pulp was 18 wt.%, there was no effect of concentration, and the torque of the stirrer decreased to below 2 Nm after 2 hours of conducting the process. The pulp retained its pumpability and fluid consistency.

Results of the investigations of laboratory scale granulation

In all methods of granulation, the formation of granules is based on the thickening of the structure of the substance resulting from the forces acting between particles or crystals. In wet granulation, which is the case when pulp is used, the binder is the liquid phase. In this type of granulation capillary forces play the major role [4].

The condition for the proper conduct of the granulation process on an industrial scale is the attaining and maintaining constant values of process parameters. Optimum pulp consistency, in terms of transport and granulation properties, enables attaining high quality of the product and high efficiency. From the viewpoint of attaining maximum efficiency of the granulation process, and consequently improvement of economic indicators of manufacture, it is desirable to obtain a granulation pulp which has the lowest possible water content and still retains good granulation purposes. Proper granulation conditions can be attained by appropriately setting the ratio between granulation pulp fed to the granulator and the recycle stream.

This paper presents the results of measurements of average crushing strength of granules and of sieve analysis of granular product obtained in the laboratory scale granulation process with the use of TSP and USP pulps. Granulation tests were performed on a laboratory setup for granulating multicomponent fertilizers (Fig. 8).

The granulation setup comprised a blade granulator, granulation disk, reactor with stirrer, peristaltic pump and vibrating feeder. Tests were conducted in periodical manner. The granulator was fed with the recycle and raw material – fertilizer pulp, which additionally acted as a binder. The recycle comprised ca. 60-65% of the proper product (particle diameter 2-5 mm) and 35-40% undersize particles (< 2 mm).

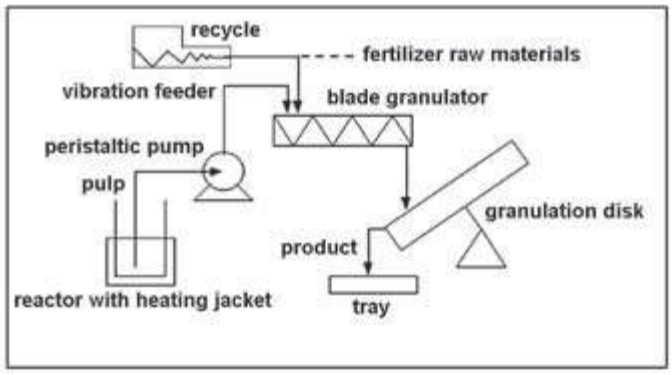

Fig. 8. Diagram of laboratory plant for fertilizer granulation

The prepared recycle was fed to the blade granulator together with hot pulp (ca. 80°C) by means of the peristaltic pump. Granulation pulps of different water content were produced in the laboratory setup for determining pulp consistency (Fig. 1.). The amount of recycle fed was varied depending on the property of the pulp. The pulp was granulated in batches of 500 g. The ratio of pulp quantity to recycle quantity varied from 1:5 to 1:9, while care was taken to

avoid clogging of the granulator. The granules produced were dried in an air-circulated drying chamber for 30 minutes at 90°C. Then, the granulate was left to cool down. Next, a 2-5 mm fraction was isolated, the strength of the granules was determined and sieve analysis was conducted to determine the equivalent diameter, size guide number (SGN) and uniformity index (UI) [5]. The results of measurements made are listed in Tables 2 and 3.

Table 1

List of granulation pulps which were granulated

Item	Sample type	Water content, % wt	Pulp:recycle ratio
1	Test no. 1 - USP pulp	22	1:6
2	Test no. 2 - USP pulp	20	1:5,2
3	Test no. 3 - USP pulp	18	1:4,5
4	Test no. 4 - TSP pulp	40	1:9
5	Test no. 5 - TSP pulp	35	1:8

Table 2

Compressive strength of fertilizer granules obtained during laboratory tests

Item	Sample type	Average size of tested granules (mm)	Average strength: N/gran.
1	Test no. 1 - USP pulp, H_2O content 22 wt.%	3.91	24.2
2	Test no. 2 - USP pulp, H_2O content 20 wt.%	4.05	23.7
3	Test no. 3 - USP pulp, H_2O content 18 wt.%	3.98	25.6
4	Test no. 4 - TSP pulp, H_2O content 40 wt.%	3.79	29.8
5	Test no. 5 - TSP pulp, H_2O content 35 wt.%	3.40	31.6

Table 3

Indicators of fertilizer particle size distribution obtained during laboratory tests

Item	Sample type	Mass fraction retained on sieve (% wt.)						Equivalent diameter (mm)	SGN	UI
		5.00	4.00	3.15	2.50	2.00	<2.00			
1	Test no. 1 - USP	5.28	17.25	35.12	26.86	13.43	2.07	3.36	333.5	48.34
2	Test no. 2 - USP	3.13	25.00	38.54	22.92	8.33	2.08	3.46	351.8	49.86
3	Test no. 3 - USP	5.66	37.74	33.02	14.15	7.55	1.89	3.69	383.0	49.66
4	Test no. 4 - TSP pulp	4.64	18.84	22.23	33.57	17.95	2.77	3.25	306.7	47.32
5	Test no. 5 - TSP pulp	5.37	38.11	25.41	9.77	16.61	4.73	3.51	378.2	45.24

The granulation tests performed on a laboratory setup show that it is possible to modify the pulp preparation step by lowering water content without affecting the granulation process. In such cases it is necessary to reduce the ratio of recycle in the granulation system. The strengths of the obtained granules ranged from 23.7 to 25.6 N/granule in the case of USP fertilizer, and from 29.8 to 31.6 N/granule in the case of TSP fertilizer. The results of the sieve analysis show that decreasing water content in the pulp increases the tendency to form granules larger in diameter and of slightly more irregular shape. However, the equivalent diameter of the granulates was proper and remained within the range of 3 to 4 mm. To summarize, the differences in strength and grain size distribution measurement results of the various granulate samples were small, indicating that reduced water content in the pulps did not deteriorate the physicochemical properties of the granulates.

Summary

The aim of this study was to determine the effect of water content and temperature on the consistency of pulps used in the production of phosphate and multicomponent fertilizers, as well as to assess the suitability of such pulps for granulation. Tests were conducted on a laboratory setup that enabled measurement of pulp mixing torque and temperature. The results obtained indicate that the system proposed can be used in tests of this type. The conducted experiments enable the assessment of the effect of water content and temperature on the properties of the various pulp types.

Results of tests of TSP 48 pulp preparation show that additional water is necessary during digestion in an amount of about 40 wt.%. This amount of water helps retain the pulp in fluid state in the reactor and avoid problems when transferring it to the granulator. Heating of the mixture obtained by digesting phosphate rock with phosphoric acid seems advantageous, as it adds to the fluidity of the pulp. While the temperature of the obtained pulp drops, the pulp slowly thickens, and continuous agitating is required.

For the preparation of MSP 30 pulp conducted with no additional heating, water was added in an amount of about 32 wt.%. This amount sufficed to obtain the required pulp consistency. This pulp did not thicken abruptly after cooling down. When pulp of the same composition was prepared while heating it to 80-90°C, the amount of water added was about 46 wt.%. This pulp tended to thicken at elevated temperatures as well as upon cooling.

Preparation of the USP pulp, due to rapid increase of mixing resistance during the feeding of phosphate rock to the solution of urea in sulphuric acid, requires great care. Phosphate rock should be fed in small portions. Adding excessive amount of material at one time could cause the pulp to lose its fluidity and clog the reactor. Stable conditions of mixing and granulating the USP pulp were attained when the pulp contained ca. 18 wt.% water.

Beneficial effect of reduced water content in pulp on recycle ratio was observed during the individual granulation tests. It may therefore be said that reduction of water content in pulp decreases the amount of water that has to be evaporated and the amount of energy required for recycling off-spec product, which translates into reduced overall power consumption.

The tests conducted have shown that granules of moist TSP and USP had different properties. In the case of USP fertilizer the tendency of the granules to agglomerate was much lower than in the case of TSP fertilizer.

The two-stage granulation system, comprising mixing the raw materials in a blade granulator followed by shaping the granules on a disk, enabled the formation of nearly spherical smooth granules. The physicochemical properties of the obtained granulates (granule size and strength) were at the mean level for the fertilizers tested. Reducing water content in pulp within the range studied, causes

slight increase of granule diameter and deterioration of its shape. However, it does not affect the granulation process and does not reduce granule strength.

Depending on the raw materials used and conditions under which they are prepared, granulation pulps have diverse physicochemical properties. The tests performed are of an illustrative nature and constitute a reference for further work aimed at improving the operating efficiency of the reactor unit and the production economics by reducing energy requirements for the process of manufacturing granulated fertilizers.

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