Studies on the screw granulation of fertilizers manufactured on the basis of urea and calcium sulphate adduct

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Introduction

The screw granulation technology, referred to also as extrusion, means a process that is commonly applied to production of plastics [1]. It has also found application to the pharmaceutical and food sectors [2] and is also frequently used to produce catalytic agents and carriers. The Fertilizer Research Institute applied that method to granulation of hop [3]. The process involves extruders of various types, including those with a single or two screws. The results of former investigations carried out by scientists from the Fertilizer Research Institute on screw granulation of fertilizers classified to the type of urea and calcium sulphate adduct indicated that that method is suitable to obtain products with high degree of urea conversion into the form of adduct [4]. However, these granular products were manufactured by extraction of a paste prepared by amalgamation of urea and phosphogypsum with the water content of about 30%. The reaction of adduct formation took place chiefly during the phase of paste preparation. Therefore the decision was made to investigate whether it is possible to skip the phase of adduct paste preparation and to produce fertilizers of the urea and calcium sulphate adduct type in a direct manner.

Methodology

The investigations were carried out with use of a plant for screw granulation (Fig.1) manufactured by the Factory of Cable Machines (Zakład Maszyn Kablowych) 'ZAMAK'

Sp. z o.o. (Co. Ltd.) with the maximum productivity of 45 kg/h. The major component of the plant is an extruder with the structure as shown in Figure 2.

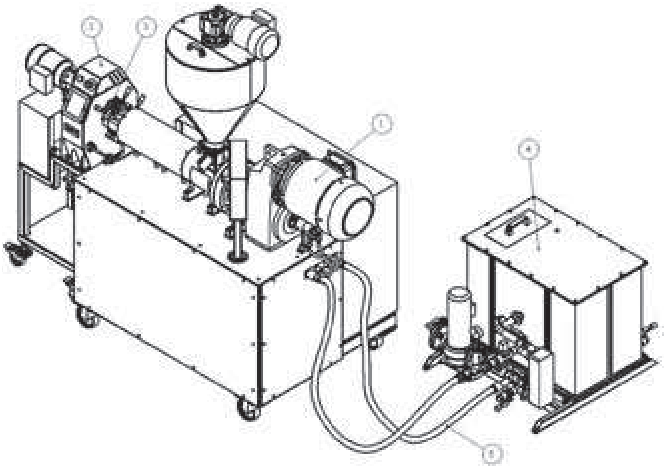

Fig.1. The plant for screw granulation
EHR-45 extruder, 2 – G-45 INS granulating unit, 3 – GM-45 head, 4 – power supply system for heating and cooling units, 5 – connection of the power supply system

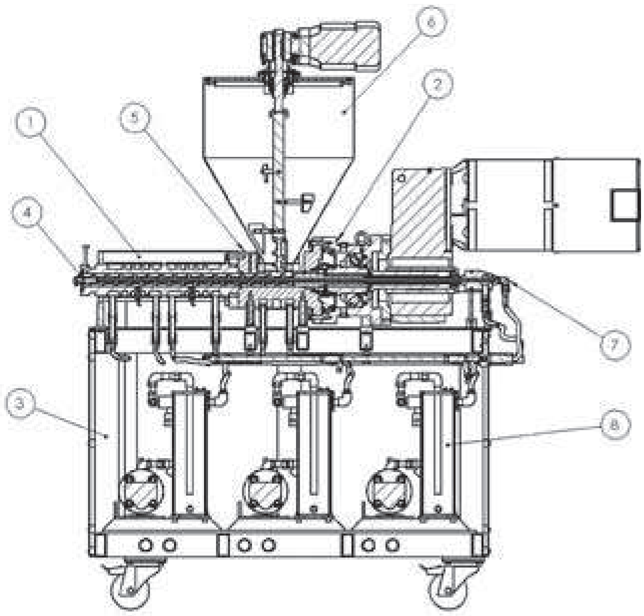

Fig. 2. The EHR – 45 extruder
1 – cylinder, 2 – thrust bearing support, 3 – frame, 4 – coupler, 5 – screw, 6 – charging hopper, 7 – water distribution system, 8 – cooling system for the screw

The cylinder, the coupler body and the screw of the extruder are made of high-alloy special steel. All surfaces that are exposed to wear during the manufacturing process are nitride hardened. The cylinder and the coupler are provided with a heating and cooling jacket. The cylinder is furnished with sockets for installation of temperature sensors and a pressure sensor. The cylinder end at the side of its head is adapted for connection with other heads with use of a taper key joint. The screw with its rated diameter D=45 mm has the working length of 15D and is terminated with a tapered end. The screw was modified during the investigations by providing a cutter that was attached to the screw end in order to make the extrusion process easier. The extruder frame is a welded structure made from steel profiles with a control panel attached thereon. The material to be extruded is supplied to the plastification system from a charging hopper that is made of stainless steel. The stirrer and a supplying worm are aligned to the central line of the machine and driven by means of a gear motor since the supplying worm keeps the plastification system constantly full. Water is distributed to specific areas of the machine owing to application of three heating and cooling units. These units are installed inside the extruder, below the plastification system. The tests involved application of a screen with a mesh diameter of 8 mm.

The raw material used for investigations comprised phosphogypsum acquired from the Chemical Factory (ZCh – Zakład Chemiczny) 'POLICE' S.A. as well as from the Factory of Phosphor Fertilizers 'FOSFORY' Sp. z o.o. (Co. Ltd.) (Gdański Zakład Nawozów

Fosforowych - GZNF) in Gdańsk. Due to high content of water the phosphogypsum from 'POLICE' was pre-dried to achieve two samples with the total water content of 30% w/w and 23.4 w/w respectively. The crystallized urea necessary for the investigations was made by the Nitrogen Plant 'KĘDZIERZYN' S.A. (Zakłady Azotowe Kędzierzyn - ZAK) whilst kieserite with the content of 27% MgO was purchased from K+S Kali GmbH.

Prior to each granulation attempt the raw products were premixed with use of a mechanical stirrer and then poured into the charging hopper of the extruder. The extrusion process was carried out at the respective temperatures of the heating and cooling system equal to 20°C, 40°C and 60°C. During each test the rotation speed of the extruder shaft was switched within the range of 20, 40 and 60 rpm. Figure 3 presents measurement results for the residence time how long the mixture made up of urea and phosphogypsum was processed by the extruder as a function of the screw rpm.

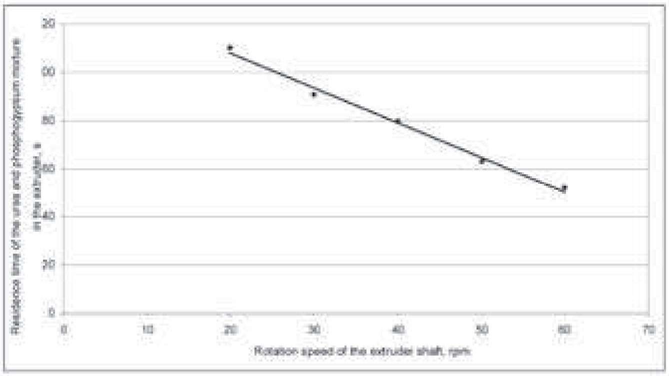

Fig. 3. Residence time of the urea and phosphogypsum mixture in the extruder

The final products obtained in the form of granules were then dried at the temperature of 105°C and subjected to the further analysis in order to find out how much urea was transformed into the form of adduct and to determine the crushing strength of the final product. The degree of urea reaction into the form of adduct was determined by means of the method based on the differential solubility in *n*-butanol demonstrated by free urea and the urea bound into adduct. The crushing strength achievable for granules of produced fertilizers was measured by means of the ERWEKA TBH 200D instrument. Each series of measurement was carried out for 20-30 granules.

Examination results

The attempts to manufacture fertilizers classified to the type of the urea and calcium sulphate adduct were carried out by means of the screw granulation method. Composition of mixtures subjected to the granulation process is summarized in Table 1.

Table 1

Compositions of mixtures for screw granulation

No.	Content in the mixture. % w/w			
	phosphogypsum	urea	kieserite	water in total
1	45.3	54.7	-	13.59
2	43.8	56.2	-	10.25
3	45.3	54.7	-	12.68
4	43.3	50.2	6.5	12.93

For the examined mixtures the molar ratio between urea and calcium sulphate obtained from phosphogypsum was 4:1. The materials used for attempts 1-3 enabled to produce fertilizers with the respective content of N, S and CaO amounting to 29.0, 8.0 and 14.0 percent. For the attempt #4 the content of individual components, i.e. N, S, CaO and MgO in the final fertilizers amounted to 26.6, 9.4, 13.8 and 2.0 percent. The specified formulas correspond to fertilizers with the 1% content of water w/w.

Table 2 brings together key parameters of the extrusion process, the maximum temperature the mixture of reagents reached, the maximum pressure recorded inside the extruder in the area upstream the screen as well as properties of final products.

The first attempt of granulation was made with use of phosphogypsum from 'POLICE' preliminarily dried to the water content of 30% w/w. The process was carried out at the temperature of 20°C maintained within the heating and cooling system of the extruder. The obtained results made it possible to find out that the final product demonstrated high degree of urea conversion into the form of adduct, as high as above 78% for the rotation speed of the machine shaft equal to 20 rpm. The conversion degree slightly decreased in pace with the growth of the rotation speed. The granulate exhibited high crushing strength of above 120 N/gran. that dropped to the level of 105 N/gran. at higher values of the rotation speed. Faster rotation of the machine shaft was accompanied by the growth of the mixture temperature (from 47.3°C to 60.7°C) and pressure inside the extruder (from 2.07 MPa to 3.16 MPa).

Table 2

Test parameters and properties of final products

Sample #	Rotation speed of the extruder screw, rpm	Temperature of the heating and cooling system, °C	Maximum temperature of products °C	Maximum pressure of products, MPa	Conversion degree of urea, %	Crushing strength, N/gran.
Attempt #1						
1.1	20	20	47.3	2.07	78.71	123.5
1.2	40	20	55.9	2.69	77.31	109.8
1.3	60	20	60.7	3.16	77.06	105.2
Attempt #2						
2.1	20	20	72.6	8.81	76.67	111.6
2.2	40	20	88.3	5.87	71.37	114.0
2.3	60	20	107.1	5.79	72.64	104.6
Attempt #3						
3.1	20	20	52.5	5.32	81.57	135.2
3.2	40	20	63.3	5.28	67.72	119.8
3.3	60	20	70.2	2.10	68.68	105.2
3.4	20	40	47.7	3.00	76.41	111.6
3.5	40	40	51.3	2.06	77.23	118.1
3.6	60	40	46.8	1.37	77.51	114.0
3.7	20	60	43.8	0.29	89.14	127.8
3.8	40	60	43.8	0.21	71.40	123.5
3.9	60	60	The feed caked in the charging hopper and failed to travel to the screw			
Attempt #4						
4.1	20	20	41.0	6.07	68.92	72.9
4.2	40	20	43.6	3.04	66.45	63.4
4.3	60	20	41.5	1.78	61.77	75.2
4.4	20	40	35.7	0.57	58.07	103.4
4.5	40	40	35.8	0.51	74.03	114.5
4.6	60	40	37.2	0.55	70.95	98.7
4.7	20	60	The feed caked in the charging hopper and failed to travel to the screw			

The second attempt of extrusion was carried out with use of phosphogypsum from 'POLICE' but that time it was preliminarily dried to the water content of 23.4% w/w. and mixed with crystallized urea from ZAK. The tests were completed at the temperatures of 20°C and 40°C. The measured results revealed that lower content of water in the reactive mixture led to drop of the urea conversion degree into the form of adduct and the crushing strength of granules alike. Similarly to the attempt #1, higher rpm of the extruder screw entailed visible drop of the degree of how intensely urea could be conversed into adduct. The crushing strength of final product granules remained at the high level and exceeded 100 N/gran. for each sample. Reduced water content in the mixture of reagents clearly gave rise to the mixture temperature and pressure as compared to the parameters recorded during the attempt #1. The increase in rotation speed during the attempt resulted in remarkable temperature growth from 72.6°C to 107.1°C. It was also revealed that the pressure inside the extruder decreased from 8.81 MPa to 5.79 MPa as a response to the increased rpm, which was probably caused by improved plasticity of the reagent mixture at elevated temperatures.

The third attempt of extrusion was carried out with use of phosphogypsum with the water content of 28% w/w. and received from GZNF 'FOSFORY'. The extrusion process was run at temperatures of 20°C, 40°C and 60°C maintained in the heating system of the extruder. It was found out that the temperature growth entails drop of the maximum pressure measured for the product inside the extruder and its maximum temperature, which almost certainly is associated with acceleration of the processes when crystallizing water is released from gypsum and urea is being gradually dissolved. The attempt to run the test at the temperature of 60°C was prevented by problems due to clogging of the extruder inlet by a cake of solidified reactants. The final product demonstrated excellent crushing strength that for the fixed temperature only slightly declined in pace with speeding up of the extruder screw rotation. The drop of crushing strength of granules was also revealed when the system temperature was being increased. However it must be emphasized that the crushing strength never dropped below 100 N/gran. for either of examined samples. The degree of urea conversion into the form of adduct ranged from 68% to 89% for all produced samples.

The forth attempt of extrusion involved phosphogypsum with the water content of 28.0% w/w acquired from GZNF 'FOSFORY' with the admixture of crystallized urea from ZAK and kieserite from K+S GmbH with the water content of 12.4% w/w. The extrusion process was carried out at temperatures of 20°C, 40°C and 60°C maintained within the heating system of the extruder.

The attempt at the temperature of 60°C was disabled by obstacles with supplying the reactive mixture into the screw duct as the mixture caked in the charging hopper of the extruder and solidified at the screw inlet, which made the further investigations infeasible.

The admixture of kieserite entailed visible temperature drop of the mixture inside the extruder. When the temperature of the heating system was 40°C the substantial drop of the product pressure was recorded, which was caused by alteration of the product consistence, i.e. the final product at the extruder outlet was plastic. The crushing strength of final product obtained at the temperature of 20°C was significantly lower (by ca. 30 N/gran.) as compared to the previous attempts. The product obtained at the temperature of 40°C demonstrated crushing strength exceeding 100 N/gran. The degree of urea conversion into the form of adduct also decreased as it exceeded 70% only for two samples produced at the temperature of 40°C.

Recapitulation and conclusions

The completed investigations demonstrated that fertilisers classified to the type of calcium sulphate and urea can be produced with use of screw granulation. Fertilizers with the high degree of urea conversion into the form of adduct can be produced from such raw materials as crystallized urea and phosphogypsum with reduced total content of water (28-30% H_2O w/w). For phosphogypsum grades with high water content (phosphogypsum from ZCh 'POLICE') it is first necessary to pre-dry the material or use of a feedback loop to supply dried and milled final product at the amount sufficient to achieve production of the final mixture with the total content of water ranging from 12% to 14%. It was revealed that reduction of the total water content in the reactive mixture to the level of 10% entailed considerable growth of pressure inside the extruder and, in turn, increased energy consumption of the manufacturing process. It is beneficial to run the extrusion process at the elevated temperature (ca. 40°C) since it enables to reduce pressure inside the extruder. The granulated materials produced under the foregoing conditions demonstrated high degree of urea conversion into adduct as well as high mechanical (crushing) strength, exceeding 110 N/gran. It was found out that admixture of kieserite to the blend of urea and phosphogypsum led to increased plasticity of final granulates and drop of their crushing strength, similarly to the degree of urea conversion.

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