

## Molasses Influence on Ash Granulation Process and Quality Parameters

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Rape straw ash (RSA) and sunflower husk ash (SHA) containing the primary and secondary plant nutrients – phosphorus, potassium, calcium, magnesium and also some micronutrients – zinc, copper, cobalt, manganese, iron and molybdenum may be used for fertilization of variety plants. The RSA and SHA have low plasticity and very difficult to granulate. Granulation process occurs better when binding materials are used. A molasses as a binding material influence on the optimal parameter of granulations process and granulated ash properties was investigated. The main product properties such as static crushing strength of granules, granulometric composition, pH of 10 % solution by standard method were examined. It was found that the best yield of granulated rape straw ash marketable fraction is equal 46.43 % and the greatest strength of granules – 33.9 N/gran., when approximately 19 % of molasses amount was used in the granulation mixture. Respectively marketable fraction was equal 65.18 % and the greatest strength of granules – 21.1 N/gran., when approximately 9 % of molasses amount was used in the granulation mixture of sunflower husk ash. The results indicate a positive impact of molasses on the ash granulation process and product quality parameters.

**Keywords:** rape straw ash, sunflower husk ash, molasses, plasticity, granulation process, product properties.

### INTRODUCTION

Only free-flowing materials allow mechanized handling and distribution. Granules often require less storage space because of their greater bulk density. They are stored and transported more economically. A further advantage of granular fertilizers over powdered and crystalline product is that they produce less dust, and product losses are reduced. A granular product with a definite particle size distribution is a prerequisite for uniform mechanical application with field equipment and granules with diameter between 1 mm and 5 mm are most suitable [1, 2].

Agglomeration – sticking together of particles is a major growth mechanism in granulation processes. The growth process begins as soon as liquid is added to the agitated powder mass. The growth of granules, which occurs during liquid addition, has been divided into three phases [3–6], as shown in Fig. 1:

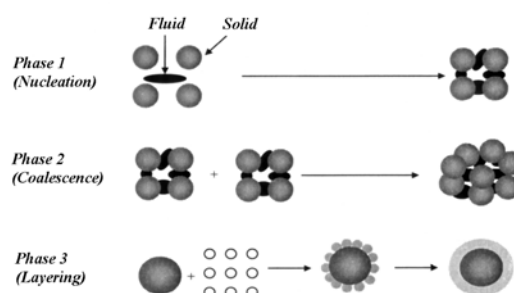
- 1 – nucleation of particles,
- 2 – coalescence between colliding agglomerates,
- 3 – layering of smaller particles onto established agglomerates.

During the initial fluid spraying, bridges are formed between the primary particles and this process is named „nucleation” [8–11]. The sticking of two large granules is referred to as „coalescence” [11, 12], whereas the sticking of the fine materials onto the surface of large pre-existing granules is often termed „layering” [10, 12].

The development of processes for granulation by agglomeration of dry materials takes place in the world. The granulation is controlled by the addition of water and/or steam.

Theoretically, for each mixture there is a percentage of liquid phase, at which granulation efficiency is optimal

[13]. The liquid phase consists of the moisture content plus the salts that dissolve in that amount of water. Since the solubility of fertilizers salts increases with temperature, the higher temperature is required.



**Fig. 1.** Phases of wet granulation: 1 – nucleation, 2 – coalescence, 3 – layering [7]

Thus, for any given mixture there is an optimum moisture content for each temperature, which may be described by a curve [13] such as Fig. 2.

The plasticity of the mixture components is very important property for granulation. Ash has a low plasticity and it is very difficult to granulate [13]. Composition that are lacking in plasticity can be granulated by addition additives such as clay and a less content of water is required in the mixture.

The data using ash for plant fertilization and for improving of soil properties are found in literature [14–18].

Equipment for ash granulation is created and produced at Germany company “Lödige” [19].

Unfortunately, data and technological parameters of ash granulation and an influence of additives on physico-chemical properties of granulated ash were not found in literature.

The aim of this work is to determine an influence of molasses on ash granulation parameters, which can be used as fertilizer. The molasses has high plasticity and as a waste is obtained in a sugar industry.

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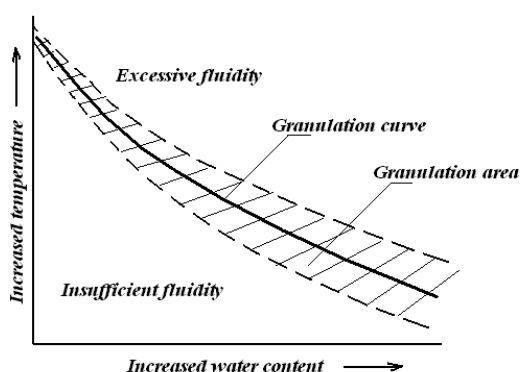


Fig. 2. The granulation curve [13]

The particle size distribution and the particle strength that are two main parameters defining the efficiency of granulation are analyzed.

## METHODS AND MATERIALS

The nitrogen content in the samples was determined by the standard methods [20], the phosphorus content was determined using a photo colorimeter KFK-2 [21], magnesium and calcium content were determined by the complexometric titration method [22], potassium and sodium content was determined with a flame photometer PFP-7 [23].

The atomic absorption spectrometry method (the A Analyst 400 device from Perkin Elmer Company) was applied for the determination of microelements content [24]. In all cases acetylene ( $C_2H_2$ ) was used to produce a flame and for the determination of the concentration of molybdenum –  $N_2O$  was applied. Chemically pure or analytically pure materials were used and the samples were prepared by decomposition of dry material with unconcentrated (1:1) HCl acid and filtering the obtained solution and diluting it in water.

Ash X-ray radiation diffraction analysis [25] was carried out by the X-ray diffractometer DRON-6 ( $Cu K_{\alpha}$  radiation, Ni filter, detector's step length –  $0.02^{\circ}$ , duration of intensity measurement in the step –  $0.5$  s, voltage  $U = 30$  kV, current  $I = 20$  mA).

Simultaneous thermogravimetric and differential scanning calorimetry (TG-DSC) [22, 25] was carried out by the thermal analyzer NETZCH STA 409 PC Luxx. Samples were heated up to the temperature of  $500^{\circ}C$ , rate of rising the temperature was  $10^{\circ}C/min$ , at air medium.

IR spectra [26] were obtained on a Perkin Elmer FT-IR spectrometer. The samples were produced by pressing the tablets from the ashes and the optically pure dried KBr. The tablet was prepared by mixing 1 mg of the test material and 200 mg of potassium bromide.

Ash was granulated in the laboratory drum-type granulator-dryer, at 3 degrees of tilt angle and at constant (26 rpm) rotation speed. The raw materials were supplied to the granulator preheated up to  $70^{\circ}C$ , hot air was supplied for drying the granules into the drum type granulator by air fan. For irrigation water was used, which was injected into the raw material mixture upstream the drum-type granulator-dryer [27].

The granular product, depending on the used content of water, was dried in an oven from 7 to 21 hours at a

temperature of  $60^{\circ}C - 70^{\circ}C$ , then physical chemical properties were identified.

The static strength of granules, as the average value of granules crushing was determined by using a device IPG-2. Its measurement range was 5 N – 200 N, instrumental error was equal  $\pm 2.00\%$  from the upper limit of measurement (when temperature is  $20^{\circ}C \pm 5^{\circ}C$ ). Calculations of strength were made using a standard methodology [23].

Hygroscopic moisture of granulated fertilizer, pH, granulometric composition and bulk density of ash were determined using the standard procedures [23].

The following materials were used for the production of granular fertilizers: rape straw ash (RSA), sunflower husk ash (SHA), Marijampolė's (Lithuania) sugar factory waste: sugar factory lime (SFL) and molasses (M). Water (W) was used for the mixture moisturizing.

The experimental granulation results are presented as an average of three measurements. The Student's criterion (t-test) was applied to determine statistical reliability of the granulation results. To determine a statistical significance of physical-chemical properties a Fischer's criterion (F-test) was applied.

By using the ash (fraction  $< 2$  mm) and sugar factory waste – SFL and M, samples were granulated in the laboratory technique. The quantities of components used in the granulation and additives improving the characteristics of fertilizers were analyzed.

## RAW MATERIAL ANALYSIS

Rape straw ash (RSA) was obtained by burning stalk in a laboratory furnace at a temperature  $400^{\circ}C - 500^{\circ}C$ .

Sunflower husk ash (SHA) was obtained in an industrial equipment. The chemical composition of sunflower ash and some properties have been investigated and presented previously [28]. In this research rape straw ash and sunflower husk ash was analysed. The concentration of primary and secondary nutrients and microelements was identified in dry substance (DS) and the results are given in Table 1.

Table 1. Content of primary, secondary nutrients and microelements in ash

	Primary and secondary nutrients (DS), %		Microelements(DS), mg/kg		
	RSA	SHA		RSA	SHA
N	0.01	0.01	Fe	1201.87	2940.46
$P_2O_5$	6.23	10.94	Cu	7.03	405.61
$K_2O$	20.72	25.84	Zn	125.06	167.23
CaO	23.24	19.07	Mn	475.88	410.45
MgO	2.12	18.58	Co	90.40	0.44
$Na_2O$	0.19	0.03	Mo	806.80	472.17

As shown in Table 1, the significant content of primary, secondary and micronutrients is present in the ash. For example,  $P_2O_5$  varies from 6.23 % up to 10.94 %,  $K_2O$  – from 20.72 % up to 25.84 %, CaO – from 12.29 % up to 23.24 % and MgO – from 2.12 % up to 18.58 %. Only traces of  $Na_2O$  and N were found. Microelements content varies in a broad range. Iron content found in ashes varies from 1201.87 mg/kg up to 2940.46 mg/kg. Reduced

content of Mo – 806.80 mg/kg, of Mn (410.45 mg/kg–475.88 mg/kg), of Zn (125.06 mg/kg–167.23 mg/kg) in RSA were determined. Content of Cu in ashes is very different: 7.03 mg/kg of Cu was found in rape straw ash, but 405.61 mg/kg of Cu in sunflower husk ash. From 0.44 mg/kg up to 90.40 mg/kg of cobalt was determined.

Chemical composition of ashes is very different and it depends on burning conditions such as oxygen excess ratio, temperature, furnace type as well as plant fertilization conditions.

The main physical properties as granulometric composition and bulk density of ash are given in Table 2.

**Table 2.** The physical properties of ash

Ashes	Granulometric composition, %					Bulk density, g/cm <sup>3</sup>
	<1 mm	(1–2) mm	(2–3) mm	(3–5) mm	>5 mm	
RSA	31.09	57.44	8.00	2.31	1.16	0.618
SHA	38.84	31.49	7.71	12.08	9.88	0.708

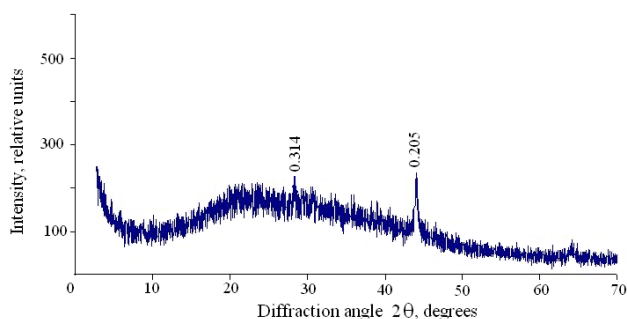
As shown in the Table 2, granulometric composition of ash is very different. The greatest content of fine fraction (up to 2 mm) – 88.53 % in rape straw ash and 70.33 % in sunflower husk ash was found. Bulk density was similar in both ash as – 0.618 g/cm<sup>3</sup> and 0.708 g/cm<sup>3</sup>.

The chemical composition of sugar factory lime and some properties have been investigated and presented previously [29].

The chemical composition of molasses was investigated and published by us earlier [30]. Molasses composition varies within the range (%): dry particles content 76–82; non-sugar substances 32–34; reducing materials 0.5–2.5; raffinose 0.6–1.4; lactic acid, 4–6; acetic acid 4–8; conductometric ash 6–10. Molasses also contain microelements (Fe, Mn, Zn, Cu, Co, Mo).

Molasses is very viscous liquid and use of the instrumental methods is difficult or even impossible. As molasses is used as a fertilizer granulation additive it is necessary to evaluate the possible material variations in the temperature range of fertilizer technological parameters. To study these changes, molasses samples were heated at temperature of 200 °C and XRD and IR analysis were carried out.

The typical results of XRD analysis of molasses are given in Fig. 3.

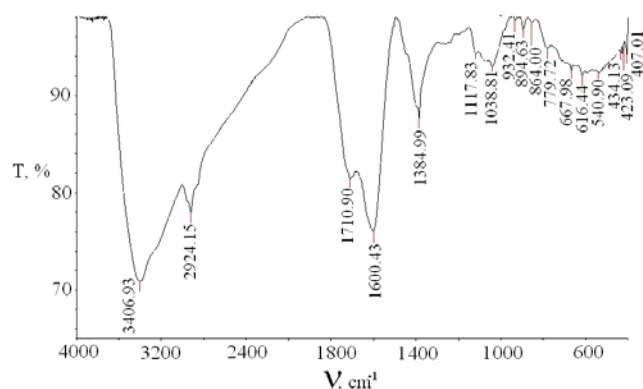


**Fig. 3.** X-ray diffraction pattern of molasses

In the spectrum obtained during the IR spectral analysis (Fig. 4) in the area 3405.93 cm<sup>-1</sup> one can distinguish low intensity typical OH valence band vibration. As the peak is broad and intensive it can be assumed that the

–OH group exists in the main structure of the investigated material. Absorption band in the frequency range of 2924.15 cm<sup>-1</sup> can be attributed to the C–H group valence vibrations. The peaks in the frequency range of 1710.90 cm<sup>-1</sup> and 1600.43 cm<sup>-1</sup> are typical to the –COO– group. The vibration in the area of 1384.99 cm<sup>-1</sup> indicates that CH group exists.

The low intensity peaks in the area of 1117.83 cm<sup>-1</sup> and 1038.81 cm<sup>-1</sup> can be attributed to the valence vibration of C–N group. The peak in the range of 932.41 cm<sup>-1</sup> is typical to the C=C–H group. The vibration in the frequency range of 894.63 cm<sup>-1</sup>, 854.00 cm<sup>-1</sup> and 779.72 cm<sup>-1</sup> can be attributed to the NH group vibration. The peaks in the range of 667.98 cm<sup>-1</sup> and 616.44 cm<sup>-1</sup> can be attributed to the CH group vibration.



**Fig. 4.** IR spectrum of molasses heated at 200 °C temperature

The peak in far IR spectrum in the range of 540.90 cm<sup>-1</sup> can be depending to the amines. The low intensity peaks in the range of 434.13 cm<sup>-1</sup>, 423.09 cm<sup>-1</sup> and 407.01 cm<sup>-1</sup> show that the inorganic salts are present.

By using the ash (fraction <2 mm) and sugar factory waste – SFL and M, samples were granulated applying the laboratory technique [27].

## GRANULATION PARAMETERS AND PRODUCT QUALITY

A rape straw ash and sunflower husk ash has a low plasticity, and granulation by agglomeration practically is impossible. Very small particles are produced and particles strength is low. To improve granulation process parameters, molasses and sugar factory lime as binding materials were used. The raw materials were supplied to the granulator that was preheated up to 70 °C, hot air was supplied for drying the granules into the drum granulator by air fan. Molasses and molasses water solution were used for irrigation that waste injected into the raw material mixture upstream into the granulator.

### Rape straw ash

12 samples were granulated using rape straw ash, sugar factory lime, molasses and water. The quantities of components used in the granulation and additives improving the characteristics of fertilizers are presented in Table 3.

The granulated product was dried at temperature of 60 °C–70 °C from 7 h to 56 h, depending on humidity and molasses content in the product. The fraction composition, pH of 10 % solution and static crushing strength of 3 mm–

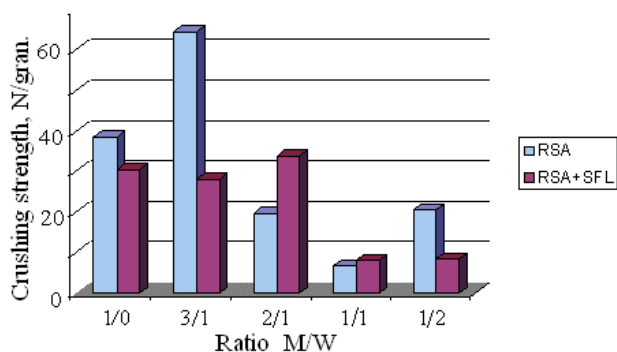
5 mm granules were measured. The results are presented in Table 4.

Our investigation has shown that static strength of the granulated rape straw ash depends on the quantity of water and recycle and varies from 1.9 N/gran. to 6.0 N/gran. When recycle was used in the granulation, less marketable product fraction was obtained.

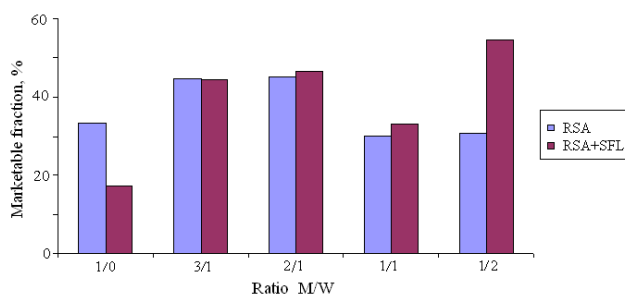
To improve physical properties of product, molasses or molasses water solutions as additives were used. The ratio molasses water varied from 3 : 1 to 1 : 2 (R1–R5 samples). Strength value of the product granules (Table 4 and Figure 5) and marketable fraction value (Figure 6) depending on molasses quantity has been increased.

**Table 3.** Composition of granulation mixture with RSA

Sample	Quantities of components, %					Ratio M : W
	RSA	SFL	Molasses	Water	Recycle	
R1	58.82	0.00	41.18	0.00	0.00	1 : 0
R2	58.82	0.00	30.88	10.29	0.00	3 : 1
R3	58.82	0.00	27.41	13.76	0.00	2 : 1
R4	50.00	0.00	25.00	25.00	0.00	1 : 1
R5	58.82	0.00	13.76	27.41	0.00	1 : 2
R6	35.71	0.00	14.29	14.29	35.71	1 : 1
R7	35.71	35.71	28.57	0.00	0.00	1 : 0
R8	35.71	35.71	21.43	7.14	0.00	3 : 1
R9	35.74	35.74	19.01	9.51	0.00	2 : 1
R10	35.71	35.71	14.29	14.29	0.00	1 : 1
R11	35.74	35.74	9.51	19.01	0.00	1 : 2
R12	19.23	19.23	11.54	11.54	38.46	1 : 1



**Fig. 5.** Dependence of granules strength on molasses/water ratio



**Fig. 6.** Dependence of marketable fraction on molasses/water ratio

The greatest granules crushing strength was obtained in R1 and R2 samples (38.5 N/gran. and 64.5 N/gran.), when molasses or molasses water solutions with ratio 3 : 1 were used for moisturizing of the raw mixture.

The best yield of marketable product was obtained in the sample R3, when approximately 27 % of molasses amount was used in the granulation mixture. The granules with a diameter from 2 mm up to 5 mm composed 45.15 % of the product.

When 50 % of the recycle was used in the granulation (R6 sample) positive influence on the product properties no observed. In this case, granules strength value is sufficient (24.4 N/gran.), but the yield of marketable fraction decreased down to approximately 22 %.

The pH value of 10 % concentration solutions of all samples varied slightly in the range of 10.5–11.3.

The mixture of ash and sugar factory lime (R7–R11 samples) as well as mixture of ash and SFL with recycling (R12 sample) was granulated. To improve properties of the granulated product a molasses or molasses water solutions of different concentration were added into mixture of RSA and SFL.

A strength of granules produced by granulating a mixture containing 28.5 % of molasses was high and equal 30.4 N/gran. (R7 sample), but large fraction of the product (diameter more than 5 mm) is predominant and approximately is equal 81 %. Marketable fraction is equal approximately 17 %. Higher marketable fraction yield was obtained when moisturizing of the initial mixture by molasses water solutions was used. The greatest strength of granules (33.9 N/gran.) and sufficient yield of the marketable fraction (46.43 %) was determined in the R9 sample. In this case, molasses water solution with ratio M : W equal 2 : 1 for moisturizing was used. The greatest marketable fraction yield (54.58 %) was obtained in R11 sample, but the granules strength value was low and equal to 8.4 N/gran.

The granulation of mixture ash and SFL with recycling (R12 sample) and using moisturizing by molasses water solution (ratio M:W equals 1:1) gives poorer granulometric composition and marketable fraction concludes approximately 32 %, but strength of granules is sufficient and equals 20.5 N/gran.

The pH value of granulated product from RSA and SFL was measured and it was found that the pH value of 10 % concentration solutions varies slightly in the range of 10.3–10.9.

#### **Sunflowers husk ash.**

Composition of the granulation mixture is presented in Table 5 and properties of the product are given in Table 6.

Sunflower husk ash was granulated by moisturizing with molasses (S1 sample) or molasses water solutions of different concentration (S2–S5 samples). Concentration of molasses in the granulation was different and varied in the range from 9.5 % up to approximately 22 %.

It was found that a crushing strength of granules depends on the content of molasses in the mixture that is presented (Table 6, Fig. 7). The strength of granulated product was greatest in the S1 and S5 samples, when concentration of molasses in the mixture was equal approximately 21.9 % and 9.5 % respectively.

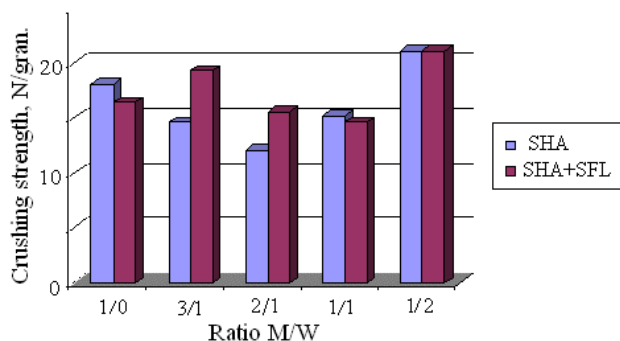
**Table 4.** RSA and additives granulation conditions and properties of product

Sample	Humidity of raw materials, %	Recycle, %	Granulometric composition of product, %					Mass change after drying, %	pH (10 %)	Crushing strength, N/gran.
			>5	3–5	2–3	1–2	<1			
R1	8.24	–	61.35	<b>21.90</b>	<b>11.54</b>	4.52	0.69	8.815	10.89	38.5
R2	16.47	–	39.39	<b>25.27</b>	<b>19.44</b>	13.01	2.88	11.317	10.89	64.5
R3	19.25	–	25.52	<b>25.01</b>	<b>20.14</b>	22.61	6.72	7.259	10.86	19.6
R4	30.00	–	25.46	<b>14.45</b>	<b>15.60</b>	24.22	20.27	17.282	10.48	6.6
R5	30.16	–	22.84	<b>14.44</b>	<b>16.43</b>	23.62	22.67	15.765	11.33	20.4
R6	17.14	50	42.90	<b>10.04</b>	<b>12.02</b>	25.42	9.62	0.455	11.19	24.4
R7	15.60	–	81.05	<b>12.86</b>	<b>4.36</b>	1.46	0.26	14.163	10.64	30.4
R8	21.31	–	48.43	<b>25.26</b>	<b>19.21</b>	6.28	0.82	14.934	10.84	28.0
R9	23.20	–	44.89	<b>26.18</b>	<b>20.25</b>	7.84	0.84	15.672	10.46	33.9
R10	27.03	–	16.55	<b>15.29</b>	<b>17.98</b>	30.49	19.69	15.051	10.25	8.0
R11	30.80	–	24.77	<b>27.08</b>	<b>27.50</b>	17.54	3.12	24.231	10.74	8.4
R12	19.17	50	25.81	<b>16.31</b>	<b>16.14</b>	36.70	5.03	11.914	10.88	20.5

**Table 5.** Composition of granulation mixture with SHA

Sample	Quantities of components, %					Ratio M:W
	SHA	SFL	Molasses	Water	Recycle	
S1	78.13	0.00	21.88	0.00	0.00	1:0
S2	71.43	0.00	21.43	7.14	0.00	3:1
S3	71.43	0.00	19.05	9.52	0.00	2:1
S4	71.43	0.00	14.29	14.29	0.00	1:1
S5	71.43	0.00	9.52	19.05	0.00	1:2
S6	35.71	0.00	14.29	14.29	35.71	1:1
S7	43.35	43.35	13.29	0.00	0.00	1:0
S8	40.54	40.54	14.19	4.73	0.00	3:1
S9	39.47	39.47	14.00	7.05	0.00	2:1
S10	41.67	41.67	8.33	8.33	0.00	1:1
S11	41.21	41.21	5.86	11.72	0.00	1:2
S12	20.49	20.49	9.02	9.02	40.98	1:1

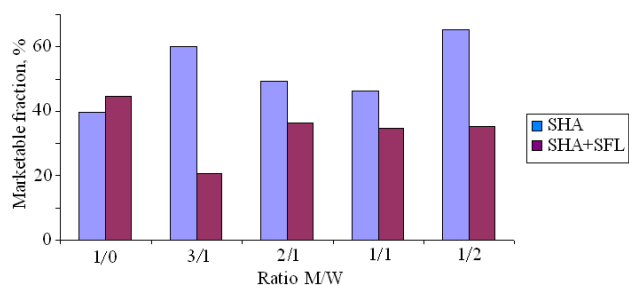
The content of marketable granules fraction (Table 6, Fig. 8) directly depends on the concentration of binding material such as molasses as well as on the humidity of granulation mixture.



**Fig. 7.** Dependence of granules strength on molasses/water ratio

The best yield of marketable fraction (60 % and 65 %) by granulating of sunflower husk ash with additive – molasses water solution was obtained in S2 and S5 samples, when content of molasses was equal approxi-

mately 21 % and 9.5 %. The granules with a diameter from 2 mm up to 5 mm conclude more than a half of the product. Some influence at mixture humidity on the content of marketable granules fraction was also observed.



**Fig. 8.** Dependence of marketable fraction on molasses/water ratio

Mixture of SHA and SFL was prepared in ratio 50 : 50 and granulated by moisturizing with molasses (S7 sample) or molasses water solutions (S8–S11 samples). The concentration of molasses in mixture was different and varied from 5.8 % up to 14.2 %.

A crushing strength of granules directly depends on the molasses concentration in mixture.

The greatest strength of granules was obtained in S11 sample and it was equal 21.1 N/gran. The concentration of molasses in this sample was approximately 6 %. The best yield of marketable granules fraction (approximately 45 %) by granulation of SHA with molasses as additive was obtained in the sample S7. In this case, concentration of molasses was approximately 13 %. It is notable, that volume of marketable granules fraction is directly linked not only to the molasses content, but also to the humidity of mixture.

Ash (S6 sample) as well as ash and SFL mixture (S12 sample) was granulated using 50 % of recycle. For moisturizing of the mixture molasses water solution was used, prepared in ratio M:W equal 1:1. The positive results were obtained: the yield of marketable granules fraction and strength of granules was equal approximately 32 % and 16.9 N/gran. in S6 sample and approximately 48 % 19.5 N/gran. in S12 sample.

**Table 6.** SHA and additives granulation conditions and properties of product

Sample	Humidity of raw materials, %	Recycle, %	Granulometric composition of product, %					Mass change after drying, %	pH (10%)	Crushing strength, N/gran.
			>5	3–5	2–3	1–2	<1			
S1	4.38	–	27.45	<b>22.30</b>	<b>17.53</b>	19.55	13.17	1.655	12.22	18.1
S2	11.43	–	29.71	<b>26.72</b>	<b>33.42</b>	7.66	2.49	5.127	12.28	14.6
S3	13.33	–	20.10	<b>15.62</b>	<b>33.82</b>	15.23	15.23	7.430	12.44	12.1
S4	17.14	–	18.69	<b>15.95</b>	<b>30.46</b>	30.25	4.66	6.496	12.28	15.2
S5	20.95	–	15.44	<b>25.17</b>	<b>40.01</b>	14.85	4.53	9.184	12.32	21.1
S6	17.14	50	65.02	<b>30.29</b>	<b>1.80</b>	1.42	1.47	11.841	12.07	16.9
S7	14.65	–	19.77	<b>24.15</b>	<b>20.59</b>	22.83	12.65	7.642	11.42	16.4
S9	20.78	–	73.59	<b>16.58</b>	<b>4.28</b>	2.98	2.57	14.022	12.1	19.3
S8	18.79	–	51.33	<b>23.70</b>	<b>12.73</b>	7.47	4.77	11.299	11.98	15.6
S10	21.53	–	35.43	<b>18.20</b>	<b>16.55</b>	23.58	6.25	11.922	11.69	14.6
S11	24.29	–	59.66	<b>26.23</b>	<b>9.06</b>	2.80	2.25	16.123	11.75	21.1
S12	16.49	50	49.36	<b>32.93</b>	<b>15.26</b>	1.83	0.63	13.643	11.87	19.5

The results indicate a positive impact of molasses on the ash granulation process and product quality parameters.

## CONCLUSIONS

1. It was found that rape straw ash and sunflower husk ash contain the primary (P, K), secondary (Ca, Mg) plant nutrients and micronutrients (Zn, Cu, Co, Mn, Fe Mo).

2. The best yield of granulated rape straw ash marketable fraction (46.43 %) and the greatest strength of granules (33.9 N/gran.) were obtained, when approximately 19 % of molasses quantity was used in the granulation mixture.

3. The best yield of granulated sunflower husk ash marketable fraction (65.18 %) and the greatest strength of granules (21.1 N/gran.) were obtained, when approximately 9 % of molasses quantity was used in the granulation mixture.

4. The results indicate a positive impact of molasses on the ash granulation process and product quality parameters.

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