

ESTIMATION OF FOOD AGGLOMERATES PRODUCED BY MEANS OF PRESSURELESS GRANULATION

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The present study was aimed at analysing the pressureless agglomeration process using various food mixtures. It additionally took into account the opportunities for agglomerating different biological materials at different percentage and using different binding liquids. The analysis of the results achieved revealed that pressureless granulation method is appropriate for fine ground materials. Determinations were carried out for: compression resistance, kinetic durability and physical properties according to the obligatory standards. For vegetable mixtures, the largest granules were achieved using 30% of potato syrup (about 2 mm). In addition, potato syrup appeared to be the best binding liquid for that type of mixture – the most durable granules. For dried mushrooms, the largest granules were achieved using 10% of a potato starch solution (1.8 mm). Half of starch concentration (5%) resulted in a decrease of granules diameter by about 40%. The smallest granules were produced for mixtures with distilled water as a binding agent (0.81 mm).

INTRODUCTION

The pressureless granulation method allows for achieving agglomerates characterised by a low granulation level and high internal porosity of granules [Korpala, 1998]. The mechanism of small particles combining into larger ones is very complex and depends on many technical and technological parameters. The condition for agglomerate formation is the presence of cohesion force between adjacent particles. Based on the studies by Schubert [1975, 1993], Litster [2003], Iveson *et al.* [2001], Hoge Kamp [1996], and Gluba [2003], it was found that for very fine particles (1 to 10 μm), the addition of any binding agent is not necessary; on the other hand, larger particles can form stable agglomerates only due to such an agent. There are materials that are very vulnerable to pressureless granulation; they are hydrophilic materials that can retain moisture on their surface.

After the granulation process, a product contains much moisture hence it should be dried. The selection of an appropriate drier depends on process intensity and local conditions.

To enable the granulator's continuous work, auxiliary devices are required; among others: a set for dosing, mixing and supplying properly ground loose products, dosing devices, granulating liquid sprayers, granulate receivers, sieves, and a drier [Grochowicz 1999].

The present study was aimed at analysing the pressureless agglomeration process using various food mixtures. It additionally took into account the opportunities for agglomerating different biological materials at different percentage and using different binding liquids.

MATERIAL AND METHODS

The pressureless granulation was examined on an example of dried mushrooms and mixtures, the compositions of which are presented in Table 1. The whole mass of the mixture was 2 kg. Dried vegetables and mushrooms originated from a plant factory near Lublin. Potato syrup and starch were produced in a syrup factory in Lublin.

Particular components were ground in a breaker equipped in 1.5 mm mesh sieve, and then mixed in a mixer to achieve homogenous matter.

The composition of the examined mixtures was selected to use them in gastronomy for food flavoring (mixtures I, II, III) as well as granulated sweets for direct consumption or to dissolve in water. Therefore, different binding liquids were used, the list of which is presented in Table 2.

The study included: (1) Measurement of material's physical properties: moisture content [PN 91/A 74010], chute angle [PN-65/2-04005], angle of repose [PN 65/2 04004], bulk density [PN-73/R-74007], shaken density [PN-65/2-04003], average particle's diameter [PN-89/R-64798]; (2) Pressureless agglomeration using various binding liquids; (3) Measurement of granulate physical properties: shaken density, average granulate diameter, compression resistance, and kinetic durability [PN – R – 64834].

Measurements of basic physical properties were made in accordance to obligatory standards in the three trials.

The moisture content was calculated on the base of the formula:

TABLE 1. Composition of the mixtures examined.

Mixture I	Content (%)	Mixture II	Content (%)	Mixture III	Content (%)	Mixture IV	Content (%)
starch	42	starch	25	flour	40	powdered milk	70
salt	20	flour	25	salt	20	cocoa	30
dried carrots	15	dried carrots	17	dried carrots	15		
dried parsnip	10	dried parsnip	15	dried parsnip	10		
basil	5	dried cabbage	12	dried cabbage	8		
parsley leaves	5	basil	3	caraway	3		
oregano	3	juniper	1.5	marjoram	3		
		thyme	1.5	oregano	1		

TABLE 2. Binding liquids used in the experiments.

Material	Binding liquid
	Water
Dried mushrooms	5% potato starch
	10% potato starch
Mixtures I, II, III	Water
	30% potato syrup
Mixture IV	20% honey in water

$$W = \frac{a-b}{a-c} \cdot 100\% \quad (1)$$

where: W – product's moisture content (%), a – weight of a vessel plus material before drying (g), b – weight of a vessel plus material after drying (g), and c – weight of an empty vessel (g).

The angle of repose was measured in a device equipped in a metal plate.

The granulometric composition was determined using a laboratory sieve device of SZ-1. Mean geometrical diameter of a particle was calculated applying appropriate standards.

The compression resistance was tested in Instron 4302. The device is equipped in a tensometric head with 1kN pressure force and 50 mm/min velocity. Samples were subjected to compression, while the maximum force was recorded. The test was conducted in 10 samples for granules from dried mushrooms and 30 samples for granules from mixtures.

The kinetic durability was measured according to the obligatory norm.

Description of the testing stand

The stand for pressureless granulation is presented in Figure 1. The disc granulator, in which granulation process occurs, is a general device. Raw material is directed through the inlet 2 to the screen 4, that due to reciprocating motion, disperses the mixture onto the granulator's disc. The binding liquid is directed from the reservoir 6 through the nozzle 3 in a form of a mist to granulator's disc 1. Produced granules are accumulated on the granulator's inlet surface and get out into the basket 5. Angle of granulator's disc bottom inclination can be regulated (α). Angle α is selected on the basis

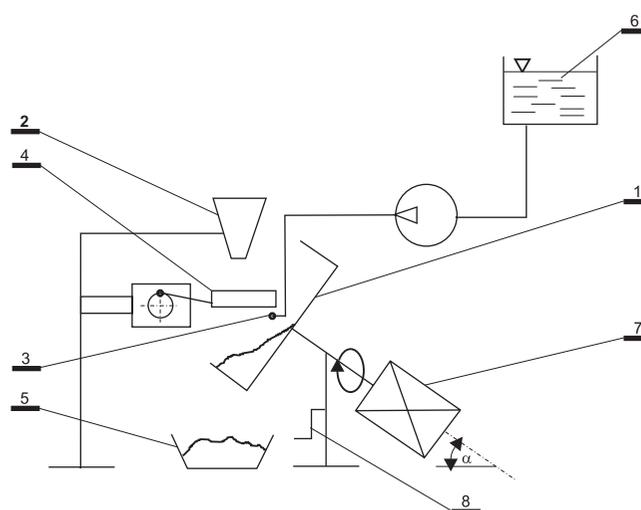


FIGURE 1. Scheme of the stand for pressureless granulation (1 – granulator's tray, 2 – supply of the material, 3 – spraying nozzle, 4 – sieve, 5 – final granulate, 6 – binding liquid, 7 – motor, 8 – crank for adjusting the tray inclination).

of preliminary studies for every mixture in such a way that the mixture fulfils all the granulator's disc surface when working. The angle α for the studied mixtures was as follows: M I, M II, M III – 34°; M IV – 30°; and for dried mushrooms – 31°.

Statistical analysis

The statistical analysis was carried out with the use of STATISTICA PL software. The results were verified statistically by determined basic statistical measures, *i.e.*: average and standard deviation. Analysis of variance at the significance level of $\alpha=0.05$ and Tukey's test were applied as well.

RESULTS AND DISCUSSION

The analysis of the results achieved revealed that the pressureless granulation method is suitable for fine ground materials. Granulation of such substances improves their physical properties, *e.g.* by decreasing their bulk and shaken density, which makes that material does not agglomerate during storage [Pietsch, 2005, 2002; Iveson *et al.*, 2001]. The granulated mixtures achieved may serve for precise dosing to other products (*e.g.* additives improving fodder mixtures) providing with

the uniform contents of particular components. That method enables also coating the achieved granules, which can be made on specially prepared granulator's drum [Pietsch, 2005; Hoge Kamp *et al.*, 1996].

Table 3 presents basic physical properties of materials for granulation. The data indicate significant differentiation of the determined physical traits of particular materials and mixtures, namely weight characteristics (bulk and shaken densities). Minor differences were observed in values characterising mean particle diameter and dump and embankment angles. Initial moisture contents of the studied materials varied within the range from 10.0 to 14.7%. That parameter determines the initial conditions of pressureless agglomerating process.

Figure 2 presents granulometric composition of mixtures with dried vegetables and herbs.

An important parameter determining pellet's quality is also the type of binding liquids. The study was conducted on dried mushroom fruiting bodies of *Lentinula edodes* with the use of different binding liquids: *i.e.*: distilled water, potato starch solution, and potato syrup solution [Sobczak, 2007].

The suitable binding liquid turned out to be a 5-10% potato starch solution. But the sweet aftertaste is not approve for all mixtures. A preliminary study is, thus, necessary to elaborate the operation parameters of granulation and to select the binding liquid for every single mixture. According to the procedure of the determination of mean size granulates the study was tested into three trials. Arithmetic mean and standard deviation were calculated. Granules achieved using 10% of potato starch were the largest, while those agglomerated using water as a binding agent – the smallest (Figure 3).

Figure 4 presents the mean size of granulate for particular mixtures. Referring to granulometric composition, the granulates with particles of 2.0–3.15 mm diameter were the most numerous. The largest granulates were achieved applying 30% potato syrup solution as a binding agent. For all three mixtures, the average granulate size was about 2 mm. The granulates produced with water as a binding liquid were characterised by mean size of about 0.56–0.72 mm.

Figure 5 presents the compression resistance of granulate achieved from dried mushrooms depending on the binding

TABLE 3. Physical properties of the studied materials.

Material	Moisture content (%)	Angle of repose (°)	Chute angle (°)	Bulk density (kg·m ⁻³)	Shaken density (kg·m ⁻³)	Mean particle diameter (mm)
Dried mushrooms	13.4±0.21	40±1	33±1	463.1 ±7.2	683.9 ±7.8	0.22 ±0.01
Mixture I	12.2±0.13	38±1	45±1	808.3±11.3	850±10.4	0.19±0.02
Mixture II	14.7±0.11	39±1	43±1	696.2±10.6	783.3±11.7	0.19±0.02
Mixture III	10.7±0.11	34±1	44±1	713.3±8.5	798±9.6	0.28±0.03
Mixture IV	10±0.13	41±1	56±1	543.412.1	567±10.9	0.11±0.01

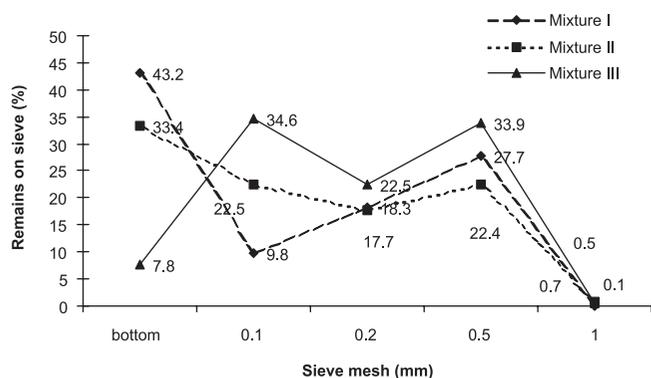


FIGURE 2. Granulometric composition of the mixtures.

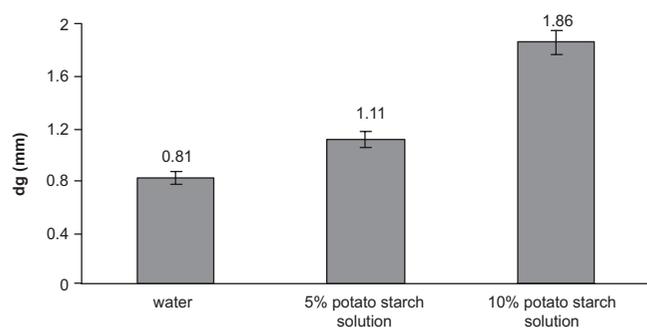


FIGURE 3. Mean size of dried mushrooms granulate.

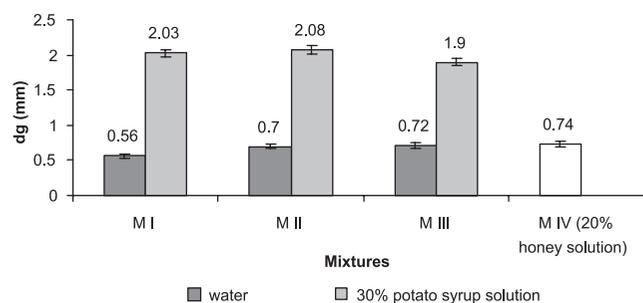


FIGURE 4. Mean size of granulate for particular mixtures.

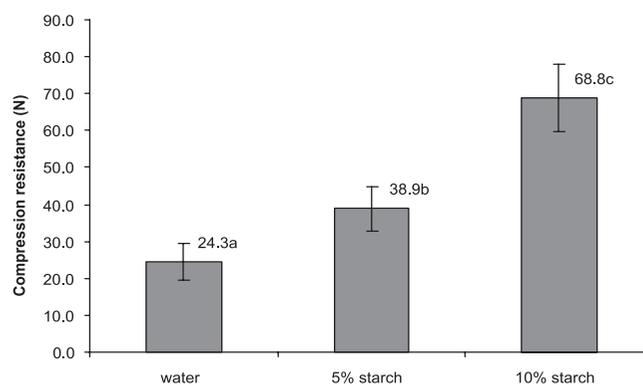


FIGURE 5. Compression resistance of granulated dried mushrooms depending on binding liquid.

TABLE 4. Variance analysis of compression resistance for dried granulated mushrooms at the significance level of 0.05.

Variability source	Freedom levels	Square sum	Square mean	Testing function F value	F α	p
Type of liquid	2	10000.58	5000.290	44.19	3.35	p< α
Error	27	3055.126	113.1528			

TABLE 5. Variance analysis of compression resistance for granulated mixtures at the significance level of 0.05.

Variability source	Freedom levels	Square sum	Square mean	Testing function F value	F α	p
Type of liquid	3	20286.91	6762.304	73.414	2.7	p< α
Error	101	9303.205	92.11094			

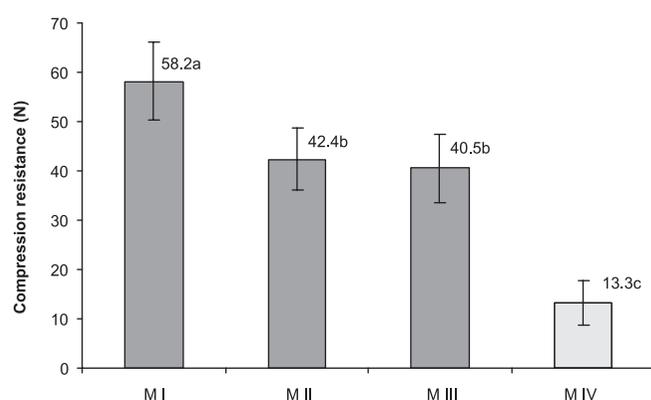


FIGURE 6. Compression resistance of granules for particular mixtures.

liquid type. On the basis of the variance analysis, significant differences were found in the compression resistance depending on the binding liquid type (starch addition), (Table 4). Results were verified by performing the Tukey's test. Statistically significant differences were present at $p < \alpha$, *i.e.* for three examined liquids. Granules made of 10% of starch solution had the highest compression resistance, while those made with water as a binding agent – the lowest.

Figure 6 presents the compression resistance of granules prepared from various mixtures and potato starch solution as a binding agent. Variance analysis at the significance level of $\alpha = 0.05$ (Table 5) confirmed significant differences in the compression resistance depending on the mixture composition. In order to evaluate in details, between which mixtures the differences occurs, Tukey's test was carried out. Statistically significant differences were shown in Figure 6 as uniform groups. Granules made using water were too small to determine the compression resistance applying the selected method. The granulates achieved from M IV mixture and honey as a binding liquid showed lower compression resistance than those made of mixtures I, II, and III. The granulate prepared from M I mixture was characterised by the highest compression resistance. There were no differences between M II and M III in compression resistance. The mixture composed of starch as the main component was characterised by elevated compression resistance as compared to that produced of flour or flour with a starch mixture. The mixture composition, *i.e.* both flour (40%) and flour (25%) plus starch (25%) mixture, did not affect significantly the compression resistance.

TABLE 6. Kinetic durability of granulate made of mixtures with syrup

Mixture	Granule diameter	Kinetic durability (%)
I	2.0–2.5 mm	71.58±2.56
	2.5–3.15 mm	91.44±1.25
	3.15–4.0 mm	91.48±1.56
II	2.0–2.5 mm	64.14±1.20
	2.5–3.15 mm	83.92±1.25
	3.15–4.0 mm	88.32±1.01
III	2.0–2.5 mm	86.04±1.69
	2.5–3.15 mm	95.64±1.55
	3.15–4.0 mm	94.32±1.29

An important property is also kinetic durability for granulates. There is not relation between the compression resistance and kinetic durability. Some very hard granules may have lower kinetic durability and the other way [Grochowicz, 1996; Gluba, 2003]. The size of the achieved granulates allows to measure the kinetic durability only in the granules achieved from mixtures I, II and III with syrup as a binding liquid. The granules were fractioned first into three sizes: 2.0–2.5; 2.5–3.15; and 3.15–4.0 and then tested in the apparatus. The results are presented in Table 6. The mixture III was characterised by the highest kinetic durability of about 95% at the lowest compression resistance.

CONCLUSIONS

1. For dried mushrooms, the largest granules were achieved using 10% of potato starch solution (1.8 mm). Half of starch concentration (5%) resulted in the decrease of granules diameter by about 40%. The smallest granules were produced for mixtures with distilled water as a binding agent (0.81 mm).

2. For vegetable mixtures, the largest granules were achieved using 30% of potato syrup (about 2 mm). In addition, potato syrup appeared to be the best binding liquid for that type of mixture – the most durable granules.

3. The compression resistance of agglomerates made of vegetable mixtures depended on that mixture composition. Addition of wheat flour caused the decrease of the force for sample damage.

REFERENCES

1. Gluba T. Compression of agglomerates in the wet granulation process. *Inżynieria i Aparatura Chemiczna*, 2003, 3, 45–47 (in Polish).
2. Grochowicz J. (ed.), Premixes and concentration mixtures. Ingredients, production techniques and the application. 1999, Pa-gros S.C., Lublin.
3. Grochowicz J., Technologia produkcji mieszanek paszowych. 1996, PWRiL (in Polish).
4. Hoge-kamp S., Schubert H.S., Wolf S., Steam jet agglomeration of water soluble material. *Powder Technol.*, 1996, 86, 49–57.
5. Iveson S.M., Litster J.D., Hapgood K., Ennis B.J., Nucleation, growth and breakage in agitated wet agglomeration processes. *Powder Technol.*, 2001, 117, 3–39.
6. Korpala W., Weiner W., Application of non-pressure granulation in utilisation of dust refuse. *Ekologia i Technika*, 1998, 6, 44–49 (in Polish).
7. Litster J.D., Scale up of wet granulation processes: science not art. *Powder Technol.*, 2003, 130, 35–40.
8. Pietsch W. Agglomeration Processes. Phenomena, Technologies, Equipment. 2002, Wiley-VCH. Verlag GmbH Weinheim Germany.
9. Pietsch W., Agglomeration in industry: occurrence and applications. 2005, Vol. 1. Wiley-VCH.
10. Polish Standard PN-91/A-74010. Determination of moisture content (in Polish).
11. Polish Standard PN-65/2-04004. Determination of dust repose angle (in Polish).
12. Polish Standard PN-65/2-04005. Determination of chute angle (in Polish).
13. Polish Standard PN-73/R-74007. Determination of bulk density (in Polish).
14. Polish Standard PN-65/2-04003. Determination of shaken density (in Polish).
15. Polish Standard PN-89/R-64798. Determination of granulation (in Polish).
16. Polish Standard PN – R – 64834. Determination of kinetic durability (in Polish).
17. Schubert H., Tensile strength of agglomerates. *Powder Technol.*, 1973, 11, 107-119.
18. Schubert H., Instantization of powdered food products. *Int. Chem. Eng.*, 1993, 33, 28-45.
19. Sobczak P., Kalbarczyk J., Żukiewicz-Koc W., Non-pressure granulation of mushroom powder. *Pol. J. Food Nutr. Sci.* 2007, 57, 161-164.

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