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THE EFFECT OF MIXING TIME ON THE HOMOGENEITY OF MULTI-COMPONENT GRANULAR SYSTEMS

Summary

Mixing of granular materials is unquestionably important. Mixing solids is common in industrial applications and frequently represents a critical stage of the processes. The effect of mixing determines the quality of products. The objective of this study was to determine the effect of mixing time on the quality of multi-component granular mixtures. Experimental studies were conducted at the mixing process line in an industrial feed mill. The applied granular systems are feed mixtures with different weight proportions of individual components, varying in their diameter dimensions and bulk densities. Estimation of the degree of homogeneity was performed based on the scale of the mixture quality proposed by Boss according to the relation suggested by Rose. The mixing degree *M* as a function of time for granular mixtures was extended by presenting it according to different levels of collecting samples. Studies were performed for four mixtures varying according to their granular mixture formulas for five different mixing times. The quality of granular mixtures consisting of eight components was defined as 'very good' for mixing times of 30, 40, 50, and 60 minutes and as 'good' for the 20-minute mixing time. For the 12-component mixture BF, the quality was considered as 'excellent' for the 40-minute mixing time, and as 'very good' for mixing times of 20, 30, 50, and 60 minutes. For the 12-component mixture WM and the 14-component mixture WW, the quality was considered as 'excellent' for all mixing times (M>0.96).

Key words: *granular mixture, homogeneity, quality, multi-component mixture, mixer*

1. Introduction

The mixing process is a particular unit operation which takes place in a great number of practical applications and in every processing industry such as mining, pharmaceutical, energy, and food industries and agriculture [7, 11, 13, 14, 15, 17, 19, 24]. The effect of mixing determines the quality of products. This is a complex process dependent on a number of parameters such as: characteristics of mixed materials [4, 5, 17, 29], mixing device type and conditions under which the process takes place [3, 7, 23, 28]. The mixing process is a result of mechanisms like diffusion, convection, and shear [4, 16].

The objective of mixing is homogeneous distribution of each component of the mixture throughout the entire volume. In contrast to liquid and gaseous systems, granular mixtures are never perfectly homogeneous and the status of such systems can be assessed based on collected samples [3]. Assessment of homogeneity of the mixture should be numerical in nature.

According to Boss [3], the numerical index defined as a degree or index of mixing should meet certain conditions, such as: it should be closely associated with the main characteristic of the mixture at each stage of production; it should characterize the final state of the mixture in a constant and unambiguous manner; it should be independent of the mixing method and of the method of mixture composition analysis; additionally, it should be easily determined or calculated in various cases of performing the mixing process. Most of the criteria for assessing the state of the mixture are based on the statistical analysis of the mixed system. This is due to the fact that the process of mixing granular materials is random and stochastic in nature [3]. Statistics is the main tool for determining the state of a granular system.

Homogeneity assessment of granular mixtures is a key aspect in assessing its state. Examples of multi-component granular systems are compound feeds and fodders. Several methods for assessing the results on the quality of mixing to control the technological process and to supervise the feed and fodder quality are used in industrial conditions. These methods include: examination of the degree of mixing based on the key component (such as chloride, calcium carbonate, zinc, copper and manganese, protein) [6, 27]; indicator method [1, 10]; internal quality control procedures in companies captured by the HACCP system. More and more often, the computer-aided image analysis is used for analysing the quality of granular mixtures [18, 25].

An extremely significant factor is precise mixing of all mixture components. Improper mixing of components, especially in the case of additives or active ingredients added to feeds for medical purposes may have adverse effects. Inhomogeneous distribution of components in the mixture, the segregation process during mixing or in transport, and during filling the mixer with the components can have negative side effects [20]. The manufacturer of animal feeds pays attention to the quality of the mixture on the one hand, and to the costs of running the process on the other, striving to optimise the process [2, 9, 10, 20]. A satisfactory mixing process should produce a uniform mixture in minimum time and at minimum cost for overheads, power, and labour. Nowadays companies are focusing on decreasing the production time while maintaining the high quality manufacturing [21].

The objective of this study is to determine the effect of the mixing time on the quality of multi-component granular mixtures. Homogeneity assessment was based on the scale of the mixture quality proposed by Boss according to the relation suggested by Rose.

2. Material and methods

Experimental studies were conducted at the mixing process line in an industrial feed mill. A schematic diagram of experimental arrangement is presented in Fig. 1. The mixing process was carried out in a vertical mixer equipped with a worm agitator. An accurate description, dimensions, and parameters of the mixer are given in other papers of the author [13, 14]. The process of particle mixing took place owing to the movements of the mixing screw and the recirculation through the bucket elevator. The poured granular material (raw material-grains) was emptied in the place of discharge, and then directed to the bucket elevator from which it went back to the mixer. The reason for recirculating the mixture was to separate grains from small impurities (husks, shells, dust). Composition of the tested granular mixtures is shown in Table 1 and Fig. 2 (the selected mixture is named UN).

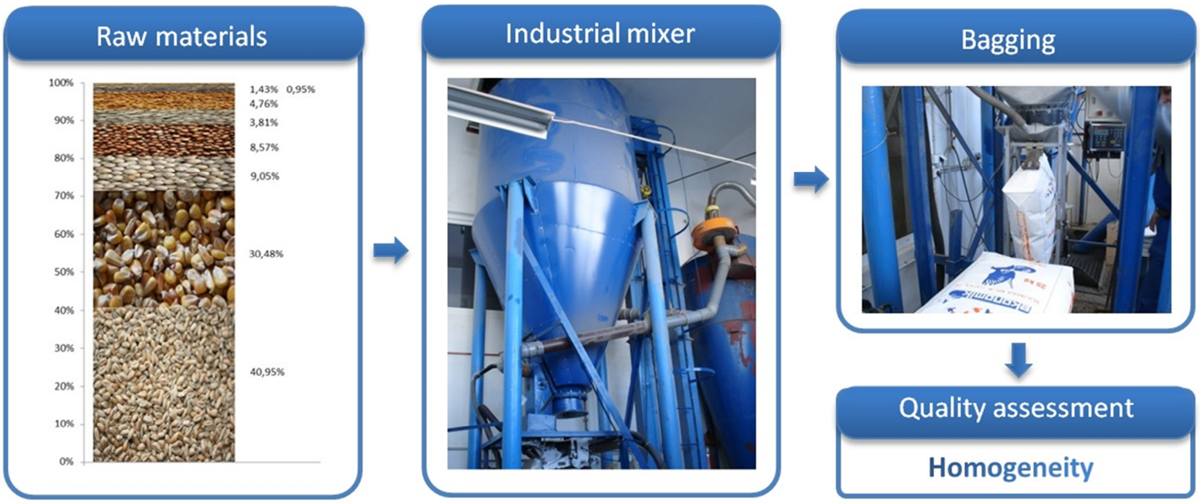

Fig. 1 Schematic diagram of experimental arrangement

Table 1 Composition of the examined granular mixtures

	Components of the mixture	8-element granular mixture (UN)		12-element granular mixture (BF)		12-element granular mixture (WM)		14-element granular mixture (WW)	
		[kg]	[%]	[kg]	[%]	[kg]	[%]	[kg]	[%]
1	Black sunflower	30	1.43	30	1.36	90	4.29	60	2.86
2	Brown vetch	-	-	-	-	50	2.38	-	-
3	Carthamus	-	-	-	-	80	3.81	40	1.91
4	Field peas	-	-	20	0.91	170	8.10	80	3.81
5	Green peas	80	3.81	80	3.64	510	24.28	120	5.71
6	Hulled oat	-	-	-	-	100	4.76	50	2.38
7	Light vetch	-	-	30	1.36	-	-	30	1.43
8	Red maize	-	-	100	4.55	-	-	100	4.76
9	Red millet	-	-	25	1.14	25	1.19	25	1.19
10	Sorghum	180	8.57	220	10.00	320	15.24	265	12.62
11	Wheat	840	40.95	660	30.00	-	-	-	-
12	White rice	-	-	-	-	50	2.38	25	1.19
13	White sorghum	20	0.95	50	2.27	250	11.90	150	7.14
14	Yellow maize	640	30.48	700	31.82	-	-	800	38.10
15	Yellow millet	100	4.76	145	6.59	155	7.38	155	7.38
16	Yellow peas	190	9.05	140	6.36	300	14.29	200	9.52
	Total	**2100**	**100**	**2200**	**100**	**2200**	**100**	**2100**	**100**

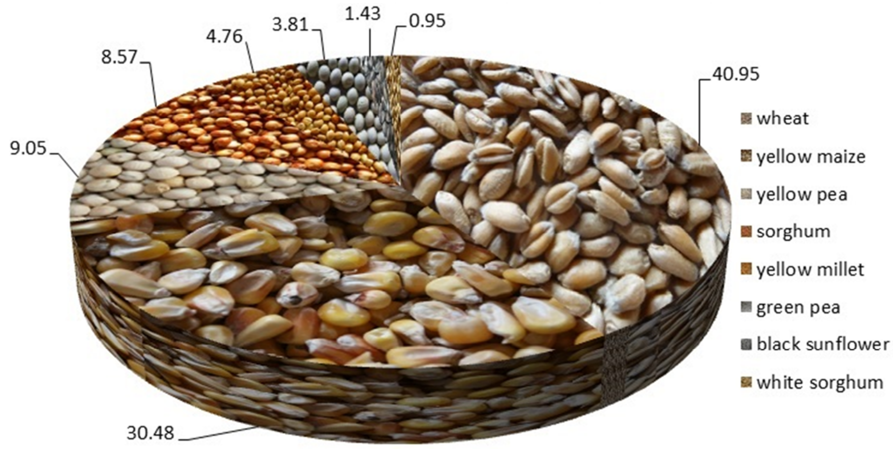

Fig. 2 Mixture composition for the fodder named UN

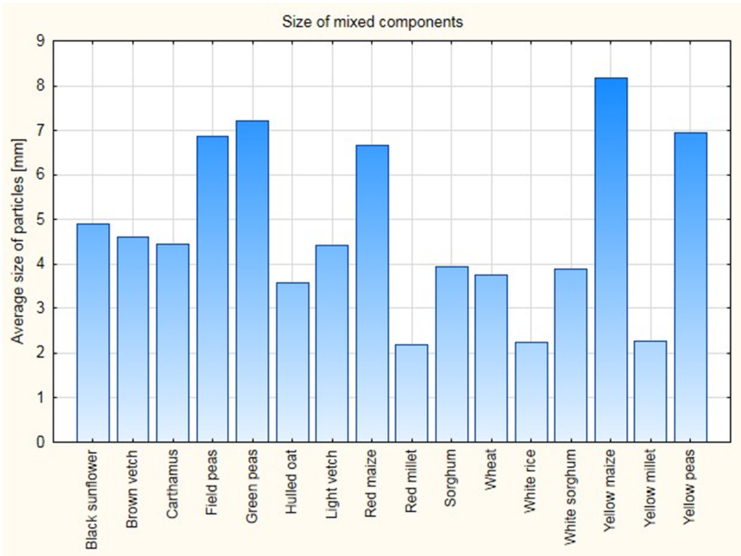

Fig. 3 Characteristic properties of mixed granular materials – average size of particles

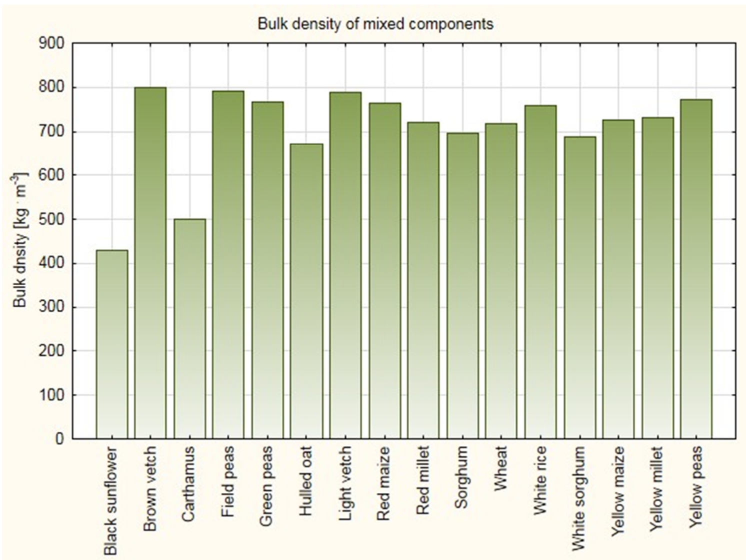

Fig. 4 Characteristic properties of mixed granular materials – bulk density

The quality of the obtained granular mixture can be determined by means of various methods like tests, quality indicators or mixing degrees. The most commonly applied method is the mixing degree proposed by Rose [26]:

$$M = 1 - \frac{S}{S_0} \tag{1}$$

where:

$$\mathrm{S} = \sqrt{\frac{\sum_{i=1}^{n}(x_i - p)^2}{n}} \tag{2}$$

$$\mathrm{S_0} = \sqrt{p(1-p)} \tag{3}$$

M – mixing degree,

n – number of samples,

p – probability of the dispersed phase occurring in the mixture,

S – estimator of the standard deviation after mixing,

S_0 – standard deviation before mixing,

x_i – concentration of the dispersed phase in the i^{-th} sample.

It is extremely important to determine times for which the obtained degree of mixing M of the mixture is of the required quality. On the basis of the relation proposed by Rose, the assessment of the mixture quality was suggested by Boss [3]. The range of the scale is from 0 to 1. According to the cited suggestion, the proposed scale of quality is as follows:

- M in the range of 0-0.70 – the quality of mixture is marked as 'failed',
- M 0.70-0.80 – unsatisfactory,
- M 0.80-0.90 – quite good,
- M 0.90-0.94 – good,
- M 0.94-0.96 – very good,
- M > 0.96 – excellent.

3. Results and discussion

The above mentioned classification of the mixture quality regarding the degree of mixing *M* defined by the formula (1) was used to determine the effect of mixing time on the homogeneity of the tested multi-component granular mixtures. The input data were the percentages of components in subsequent research trials. Sample test results of the selected series of research tests (20 and 60 minutes of mixing) carried out on the granular mixtures called UN are presented in Fig. 5. The graph shows three selected components: wheat, sorghum and maize. Results of the analysis were presented for three levels of collecting samples in the bag 'a', 'b' and 'c'.

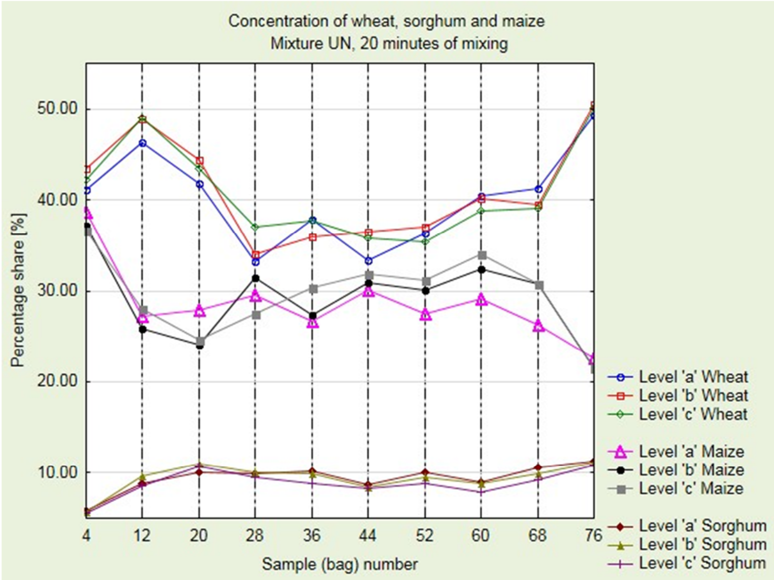

Fig. 5 Concentration of selected components of the UN granular mixture after 20-minute period of mixing – wheat, sorghum and maize

It is clear that the concentration of components varies depending on the order (number) of the bag. This is related to segregation processes that occur when mixing granular materials with very different properties. It has been proven that significant variation in relative diameter dimensions of mixed particles has the strongest effect on the process of segregation. The order of adding components into the mixer affects the mixing process. In industrial practice, firstly, larger components like maize, peas, field peas are added and then components with smaller grains. Another observation made by people working in this field is to turn on the worm agitator placed in the mixer during the processes of pouring and packing into bags (Fig. 1). During the experiment, it was noted that a secondary segregation occurs at the end of bagging, which can be noted as poorer mixing of the components. Commonly, small grains are observed more frequently in final bags. Improvement in the process technology achieved by launching a worm agitator during the process of bagging is a method for mitigating the observed effect of secondary segregation. The agitator is run for approx. 1 minute, and then the bagging process is continued. Usually such mixing occurs in the middle of the bagging process and at the end of the process. The process of secondary segregation is frequently observed in many situations and conditions [3, 12, 22]. This is one of the reasons for the variation of mixing degree with mixing time. After some time, the quality of mixing achieves its maximum and then the quality becomes worse. The probable state of granular mixture which will be obtained after a sufficiently long period of mixing components with similar properties is a random state. The probability of finding a particle of a specific component in the mixtures is the same at all places of the mixture. However, in the case of heterogeneous and multi-element granular systems, the description of the state of the mixture is one of fundamental problems in mixing [3, 8, 9]. In such mixtures we have significant differences in density, shape or average grain size. In Fig. 5, one can note that the percentage of wheat is larger than the target share (40.95%) for the first and final bags. The share of maize in most bags is similar to the target share (30.48%). In the first bag subjected to analysis (no 4) there is significantly more maize than it is specified in the recipe, while in the final bag (no 76) the amount of this component is much lower. When comparing the next component, which is sorghum, there is less of this component in the first bag than it is specified in the recipe (8.57%) and more of it in the final bag (no 76). The trends described above are observed in all sections (levels) of bags 'a', 'b' and 'c'; variation curves for each component have similar waveforms.

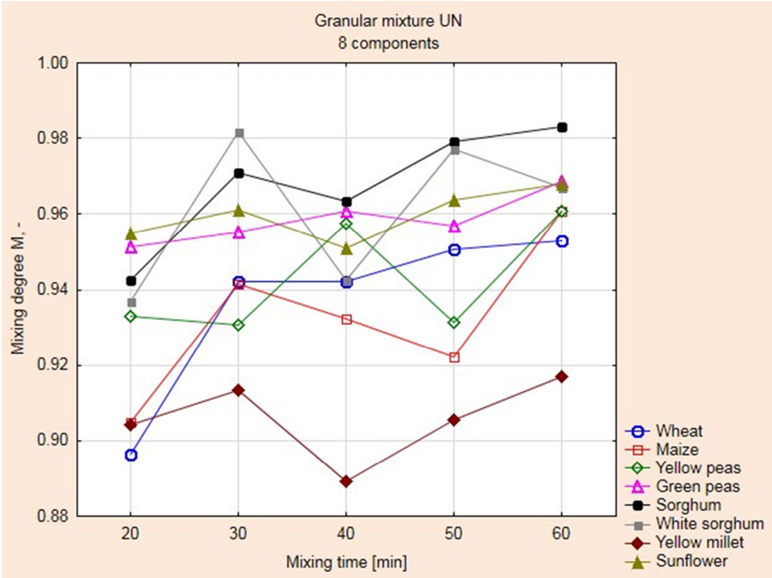

Fig. 6 The degree of mixing M as a function of mixing time for components of the UN granular mixture

Fig. 6 presents the degree of mixing M calculated for individual components of the UN granular mixture. Degrees of mixing M are shown separately for each component. The degree of mixing M obtained for this mixture was in the range of 0.89-0.98. This indicates that the individual components of this quality can be defined as 'quite good' for $0.80<M<0.90$, 'good' for $<M<0.94$, 'very good' for $0.94<M<0.96$, and 'excellent' for $M>0.96$. Analysing the graph shown in Fig. 6, it can be said that 'yellow millet' is the least capable of mixing , while the range of the obtained values is the lowest and it amounts to 0.889-0.917, whereas sorghum has the best mixing capabilities (M_{mean}= 0.967- the highest mean value derived from the degree of mixing M). The case of 'red and yellow millet' from the BF mixture (not presented in the form of graph) is also the one which mixes unsatisfactorily (M_{mean}= 0.914 - the lowest value of the mean M obtained for the range of 20-60 minutes). Similarly, regarding the mixtures WW and WM, the component which mixes the worst was 'yellow and red millet'. As for the mixture WW, the range of the obtained values is the lowest and it amounts to 0.937-0.952, while for the mixture WM it amounts to 0.939-0.948. 'Red and yellow millet' are components with the smallest diameter (Fig. 3), hence they are the worst ones to mix. It can be assumed that this component moves in a 'favourable' manner in the bed as it passes through the lower parts of the agitator through inter-granular spaces in the bed.

However, the quality of the mixture should be determined by taking into account all the components together, as it is shown in Figs. 7-11.

The quality of the 8-component mixture UN (Fig. 7) can be defined as 'good' for the mixing time of 20 minutes (M=0.928 – we consider in this part of the paper only 'mean' values) and 'very good' for other mixing times of 30, 40, 50, and 60 minutes. Values of the degree of mixing M amounted to: M_{30}=0.950, M_{40}=0.942, M_{50}=0.948, and M_{60}=0.960. In the case of this mixture, it cannot be clearly said that as the mixing time increases, the homogeneity of the granular mixture increases as well. The values of the degree of mixing M for 40 and 50 minutes are lower than those obtained for the 30-minute mixing time. For this mixture, it is not justified to mix it for longer than 30 minutes (after 30 minutes we obtained a mixture of 'very good' quality).

In the case of the 12-component mixture BF (Fig. 7), values of the degree of mixing M amounted to 0.949-0.961. The quality of the BF mixture can be considered as 'very good' for the mixing time of 20, 30, 50, and 60 minutes. These values were: M_{20}=0.949, M_{30}=0.949, M_{50}=0.955, M_{60}=0.957. For the mixing time of 40 minutes, the quality of the mixture can be considered as 'excellent' (M_{40}=0.961). However, mixing periods longer than 20 minutes are in this case not justified since the value of the degree of mixing *M* set for 20 minutes was M_{20}=0.949, and for 30 minutes M_{30}=0.949.

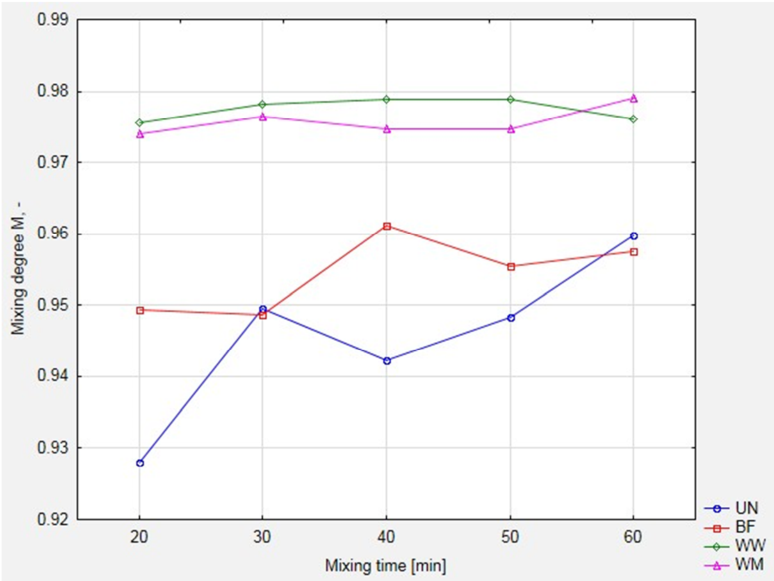

Fig. 7 Comparison of the degree of mixing M (mean values) as a function of mixing time

In the case of the 12-component mixture WW (Fig. 7), values of the degree of mixing *M* amounted to 0.976-0.979. The quality of the WW mixture can be considered as 'excellent' for all mixing times M>0.96 (M_{20}=0.976, M_{30}=0.978, M_{40}=0.979, M_{50}=0.979, M_{60}=0.976). Trends of quality improvement can be noted as the time period is being extended to 40 minutes, but in this case the improvement in quality is extremely small, so 20 minutes of mixing is enough to obtain ‘excellent’ quality of this mixture.

The quality of the 14-component mixture WM (Fig. 7) can be defined as 'excellent' for all mixing times. Values of the degree of mixing *M* amounted to 0.974-0.979 (M_{20}=0.974, M_{30}=0.976, M_{40}=0.975, M_{50}=0.975, M_{60}=0.979). Extending the mixing time period above 20 minutes is not economically justified.

The degree of mixing *M* of granular mixtures as a function of time was also presented in relation to different levels of sampling- 'bottom'-'a', 'middle'-'b' and 'top'- 'c' (Figs. 8-11). Waveforms are similar for all levels of sampling for the 8-component granular mixture (Fig. 8). However, greater variability is noted with the 12-component mixture (Fig. 9). In the cases of granular mixtures WW and WM we can observe a lower mixture quality for the ‘bottom-a’ sampling level (Fig. 10-11). The quality of this level is ‘very good’ in all mixing times for the WW mixture and also for the WM mixture in the mixing time of 20-50 minutes (M_{60}=0.964 – ‘excellent’).

The benefit of this research is a possibility to assess the quality of mixtures by controlling the technological process and to determine the exact mixing time for multi-component granular mixtures. Manufacturers of animal feed pay attention to the quality of the mixture and to the costs of running the process, striving to optimise the process. The key issue is to produce a uniform mixture in minimum time and at a minimum cost for power. In the case of this research, for mixtures consisting of 12 and 14 components, it was not justified to extend the mixing time for a time period longer than 20 minutes and longer than 30 minutes in the case of the 8 components mixture. In practice, the full mixing cycle in the fodder mixing plant is 30 minutes. This research has proven that we can shorten the mixing time with no loss in the mixture quality.

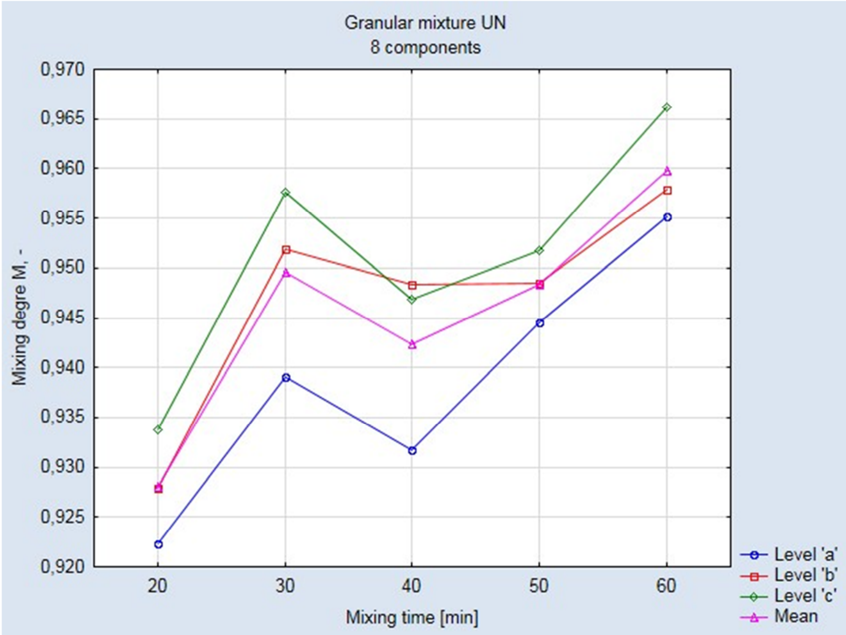

Fig. 8 The degree of mixing M as a function of mixing time for the UN granular mixture on different levels of sampling (bottom, middle, top)

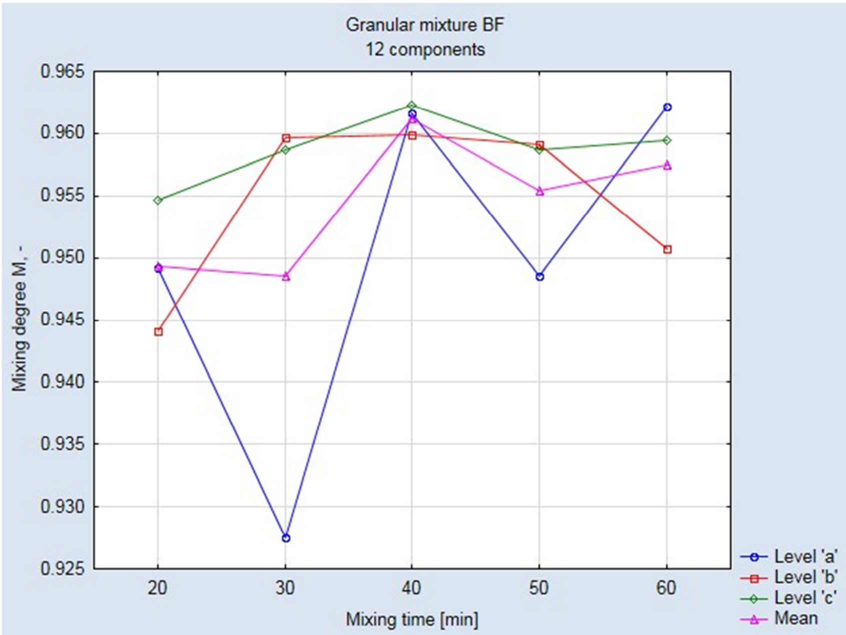

Fig. 9 The degree of mixing M as a function of mixing time for the BF granular mixture on different levels of sampling (bottom, middle, top)

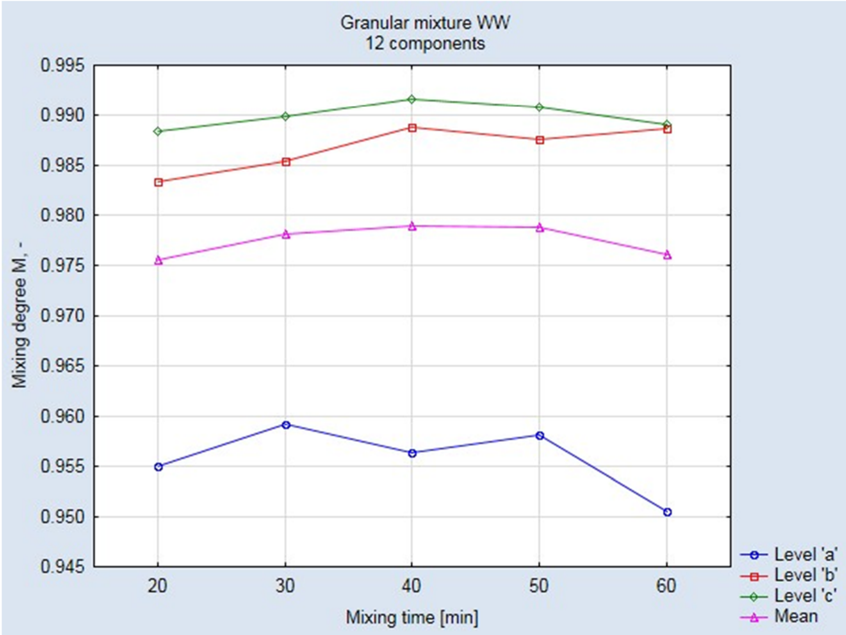

Fig. 10 The degree of mixing M as a function of mixing time for the WW granular mixture on different levels of sampling (bottom, middle, top)

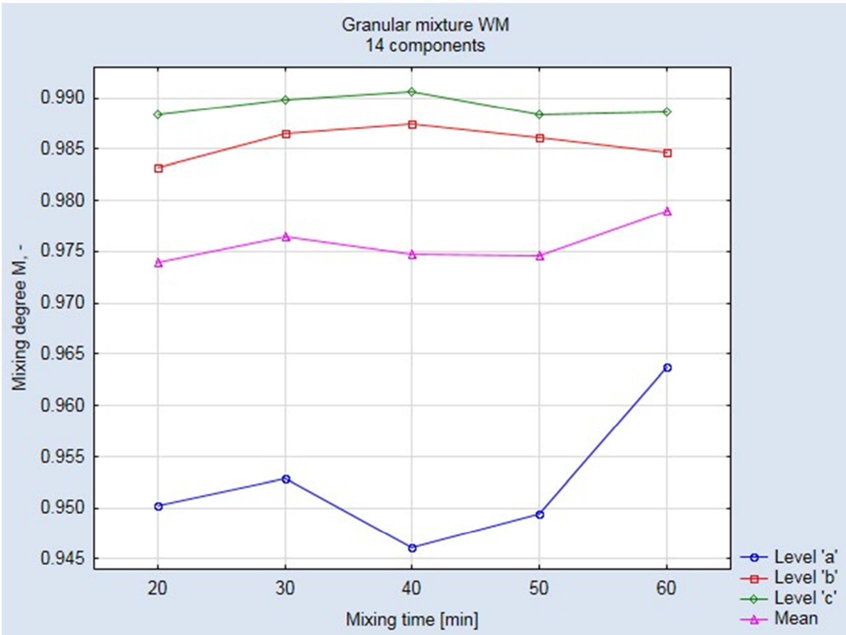

Fig. 11 The degree of mixing M as a function of mixing time for the WM granular mixture on different levels of sampling (bottom, middle, top)

4. Conclusions

1. For the granular mixture consisting of eight components designated as UN, the quality can be defined as 'good' for the mixing time of 20 minutes (M=0.928) (according to the scale of mixing time proposed by Rose) and as 'very good' for other mixing times of 30, 40, 50, and 60 minutes (M=0.950, M=0.942, M=0.948, M=0.960, respectively). In the case of this mixture, it cannot be clearly said that as the mixing time increases, the homogeneity of the granular mixture increases too. The values of the degree of mixing M for 40 and 50 minutes are lower than those obtained for the time of 30 minutes; therefore, the quality of

the mixture is lower. For this mixture, it is not justified to extend the mixing time beyond the time period of 30 minutes.

2. For the BF mixture consisting of twelve components, the values of the degree of mixing M range between 0.949 and 0.961. The quality of the BF mixture can be defined as 'very good' for the mixing times of 20, 30, 50, and 60 minutes (M=0.949, M=0.949, M=0.955, M=9.957, respectively), whereas, for the mixing time of 40 minutes the mixture can be considered as 'excellent' (M=0.961). In the latter case, the mixing time of more than 20 minutes is not reasonable due to the fact that the value of the degree of mixing M for 20 minutes is the same as that for 30 minutes.
3. For the WW mixture consisting of twelve components, the values of the degree of mixing M range between 0.976 and 0.979. The quality of the WW mixture can be considered as 'excellent' for all mixing times, M>0.96 (M=0.976, M=0.978, M=0.979, M=0.979, M=0.976). In this case, the improvement in quality is extremely small, so a 20 minute period of mixing is sufficient for obtaining the 'excellent' quality of this mixture.
4. For the WM mixture consisting of fourteen components, the values of the degree of mixing M range between 0.974 and 0.979. The quality of the WM mixture can be considered as 'excellent' for all mixing times, M>0.96. Here we can observe trends of quality improvement as the time is being extended, but the improvement is small. Extending the mixing time above 20 minutes is not economically justified.
5. In the experiment, it was noted that secondary segregation occurs at the end of bagging, which results in the deterioration of mixing of the components. Often, fine grains can be more often found in last bags. Improving the technology of the process by launching a worm agitator in the time of bagging is a method for mitigating the observed effect.
6. Components with smallest diameters, such as 'yellow millet' (for the UN mixture) and 'red and yellow millet' (for the BF, WM, WW mixtures) have the lowest mixing capability (the lowest degree of mixing M was derived). It can be assumed that these components move in a 'favourable' manner in the bed, penetrating into lower parts of the agitator and going through the inter-granular gaps that are present in the bed.

REFERENCES

[1] N. Barashkov, D. Eisenberg, S. Eisenberg, J. Mohnke: *Ferromagnetic microtracers and their use in feed applications*. XII Int. Feed Technol. Symp. Novi Sad, 2008

[2] I. Bauman, D. Ćurić, M. Boban: *Mixing of solids in different mixing devices*. Sadhana, 33, 6, 721-731. 2008

[3] J. Boss: *Solid mixing process* (in Polish). PWN, Warszawa – Wrocław, 1987

[4] J. Bridgwater: *Mixing of powders and granular materials by mechanical means-A perspective*. Particuology, 10, 4, 397–427, 2012

[5] B. Chaudhuri, A. Mehrotra, F.J. Muzzio, M.S. Tomassone: *Cohesive effects in powder mixing in a tumbling blender*. Powder Technology, 165(2), 105-114, 2006

[6] M. Chyłek-Purchała, B. Kozak, M.P. Siwczyńska, A. Grelik, K. Kwiatek: *Determination of active substances and homogeneity of medicated feeds – results for 2006 – 2013* (in Polish). Pasze Przem. 3, 71–76, 2014

[7] P. W. Cleary, M. D. Sinnott: *Assessing mixing characteristics of particle-mixing and granulation devices*. Particuology 6 (6), 419–444, 2008

[8] P. J. Cullen: *Food Mixing: Principles and Applications*. Hardcover, Wiley-Blackwell, 2009

[9] B. H. Daumann, H. Nirschl: *Assessment of the mixing efficiency of solid mixtures by means of image analysis*. Powder Technology, 182, 415–423, 2008

[10] O. Djuragic, J. Levic, S. Sredanovic, L. Lević: *Evaluation of homogeneity in feed by method of microtracers®*. Archiva Zootechnica, 12, 4, 85-91, 2009

[11] N Harnby: *An engineering view of pharmaceutical powder mixing*. Pharmaceutical science & technology today, 3, 9, 303-309, 2000

[12] R. Hogg: *Mixing and Segregation in Powders: Evaluation, Mechanisms and Processes*. KONA Powder and Particle Journal, 27, 3-17, 2009

[13] J. B. Krolczyk: *An attempt to predict quality changes in a ten-component granular system*. Tehnički vjesnik - Technical Gazette 21, 2, 255-261, 2014

[14] J. B. Krolczyk: *The use of cluster analysis method to describe the mixing process of the multi-element granular mixture*. Transactions of FAMENA. Volume XXXVII No. 4, 43-54, 2014

[15] J. B. Królczyk, A. Rezwiakow, M. Tukiendorf: *Mixing of biomass and coal in a static mixer as an example of technological solutions involving implementation of renewable energy sources*. Ecological Chemistry and Engineering S, 21, 4, 685-696, 2014

[16] B. F. C. Laurent, J. Bridgwater, D. J. Parker: *Convection and segregation in a horizontal mixer*. Powder Technology, 123(1), 9-18, 2002

[17] A.S. Mendez, G. de Carli, C.V. Garcia: *Evaluation of powder mixing operation during batch production: application to operational qualification procedure in the pharmaceutical industry*. Powder Technology, 198, 2, 310-313, 2010

[18] S. Muerza, H. Berthiaux, S. Massol-Chaudeur, G. Thomas: *A dynamic study of static mixing using on-line image analysis*. Powder Technol. 128, 195–204, 2002

[19] R. M. Nedderman: *Statics and Kinematics of Granular Materials*. Cambridge University Press, 2005

[20] K. D. Neumann: *Work of the mixer is crucial for additives*. Kraftfutter, 2000

[21] L. Obregón, A. Realpe, C. Rinaldi, C. Velázquez: *Mixing of granular materials. Part I: Effect of periodic shear*. Powder Technology, 197, 9-16, 2010

[22] J. M. Ottino, D.V. Khakhar: *Mixing and segregation of granular materials*. Annu. Rev. Fluid Mech, 32, 55–91, 2000

[23] T. Pöschel, T. Schwager: *Computational Granular Dynamics: Models and Algorithms*. Springer, 2010

[24] S. Radl, E. Kalvoda, B.J. Glasser, J.G. Khinast: *Mixing characteristics of wet granular matter in a bladed mixer*. Powder Technology, 200, 3, 171-189, 2010

[25] A. Realpe, C. Velázquez: *Image processing and analysis for determination of concentrations of powder mixtures*. Powder Technol. 134, 193–200, 2003

[26] H. E. Rose: *A suggested equation relating to the mixing of powders and its application to the study of the performance of certain types of machine*. Trans. Instn Chem. Engrs 37, 47-64, 1959

[27] S. Walczyński, W. Korol: *Inter-laboratory investigations on evaluation of industrial fodder mixtures homogeneity*. Polish J. food Nutr. Sci. 57, 187–190, 2007.

[28] E. Y. Wornyoh, V. K. Jasti, C. F., Higgs: *A review of dry particulate lubrication: powder and granular materials*. Journal of Tribology, 129(2), 438-449, 2007

[29] C. Yang: *Density effect on mixing and segregation processes in a vibrated binary granular mixture*. Powder Technology, 164 (2) 65–74, 2006

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