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FLUORESCENCE OPTICAL ANALYSIS METHOD FOR ASSESSING HOMOGENEITY OF GRANULAR MIXTURES

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Abstract

This paper presents a method of optical fluorescence analysis for the evaluation of homogeneity of multicomponent grain mixtures. This method is based on the evaluation of the content of fluorescent marker. Maize with two degrees of fineness $d_1 = 1.25$ mm and $d_2 = 2.00$ mm was used as a tracer. Maize was covered with Rhodamine B, which emits red light under the influence of ultraviolet radiation. The tracer was introduced into the mixture before the mixing process began. Nine multicomponent grain mixtures were used. The proportion of fluorescent maize was evaluated on the basis of computer image analysis. Additionally, the fraction of the tracer was evaluated using a control method (validation of the accuracy of the proposed method). The results indicate that the degree of the tracer's fineness influences the results obtained. The use of fluorescent maize with particle size $d_2 = 2.00$ mm allowed to obtain results which differed less from the control method. The average size of the difference in results ranged from 0.20–0.38 for the 2.00 mm tracer and 0.38–1.34 for the 1.25 mm tracer.

Keywords: fluorescence, ultraviolet radiation, homogeneity, multicomponent granular mixtures, tracer.

1. Introduction

Mixing of granular components is one of individual operations that significantly affects the quality of the final product [1]. This process can be found in many industries such as: construction, ceramics, pharmaceutical or agri-food [2, 3]. Currently, many ingredients are mixed (even up to 20 ingredients) which creates problems of a completely new sort [4]. Królczyk [5] presents an attempt to predict quality changes in a ten-component non-homogeneous granular system during mixing carried out on the mixing line. The variety of ingredients is one of the main factors influencing segregation, defined as the opposite of mixing. Segregation may still take place during mixing but also during packaging or during transport of the final product [6]. The quality of the mixture and the end-point of the process should be done by analysing samples taken from

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the mixture. The samples taken should be representative and their quantity and size should be appropriate to the volume of the mixed material [7]. Traditionally, the sampling is done on the basis of interference with the mixed bed [8]. Matuszek [9] sampled by the special construction of flow mixer – consisted of ten rings, so it was possible to observe the distribution of particles in the entire mixed bed. Królczyk [10] sampled in a different way – samples were taken during the mixing with recirculation every 30 seconds in the place of discharge from the mixer. However, one can find publications describing non-invasive methods such as near-infrared spectroscopy (NIR) [11], laser induced fluorescence LIF [12], broadband acoustic emission [13], real-time on-line image analysis [14]. Generally, the measurements of granular descriptors, such as nature, size, morphology *etc.*, are fundamental problems in grain mass production and/or transformation, transportation and packaging [15].

Analysis of the mixing process is usually carried out on the basis of determining the share of the selected key component. So far, the content of active substances such as chlorine or sulfur [16] amoxicillin, chlortetracycline, doxycycline, lincomycin, tiamulin or tylosin [17] has been determined. Among others, the Microtracer iron filings method is well known [18]. This method was examined in industrial horizontal band [19]. However, most often the content of a specially coloured component was analysed. In this range good results were obtained only in the case of binary [9] or at most ternary systems [20]. This is due, among other things, to the fact that in the case of multiple components, it is almost impossible to analyze the share of a particular component without its manual separation. The studies in which the content of each component of a multicomponent mixture was determined are very time consuming and rare.

Optical methods are used in metrology in many scientific areas. Marciniak *et al.* [21] presents measurement of retina vessels by segmentation of images reconstructed from optical coherence tomography data. Isaza *et al.* [22] presents the development of an acousto-optic system for hyperspectral image segmentation. Optical methods, especially in the mixing of granular systems, due to the fact they are well known and researched, are characterized by simplicity and low cost in comparison with other methods. However, they require the router to be visually different than the bulk materials mixed. The use of optical methods for multi-component mixtures (even 20 ingredients) is still poorly tested. Moreover, it is with multi-component systems with ingredients differing in colours and fragmentation level that these methods show their drawbacks. One of them is the possibility of covering the tracer by bulk materials during mixing, which leads to underestimation of its fraction. The methods of this type are most often based on the analysis of samples taken [23], which requires intervention in the mixed material. Non-invasive solutions can also be found [24]. Cho *et al.* [25] were trying to solve granular segregation problems using a biaxial rotary mixer. Olaofe *et al.* [26] improved the digital image analysis technique for evaluation of segregation in pseudo-2D beds. However, when using the analysis continuously on line, interference may occur due to reflections or scratches in the mixing vessel which affects the quality of the images taken. Moreover, it may be difficult to obtain a representative sample [27].

As we know, some specific chemicals (dyes) may absorb radiation and then emit fluorescence [28]. The use of fluorescence, namely UV-visible spectrophotometry in the assessment of homogeneity of loose mixtures has been the subject of many authors' studies and concerned mainly pharmaceutical products. Karumanchi *et al.* [29] used this method to assess the end point of mixing and to identify the dead zones in the mixer based on distribution of fluorescent API granules. Mendez *et al.* [30] proved that UV spectrophotometry can be used in industry during production of acetaminophen tablets. This is due to the fact that many components of drugs used and available on the market tend to be fluorescent when excited by a wave of appropriate length. Placing a sample between a light source and a photodetector allows to determine the share

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of a given absorber after selecting the proper wavelength. The UV-visible spectrophotometry method is widely used as a quick reference method for determining the content of active ingredient during powder mixing [31]. One of its main disadvantages is that absorbency spectra of soluble components are measured all together. Therefore, it is sometimes impossible to determine the proportion of selected fractions. Moreover, this method is insensitive to substances of low concentration [32].

Due to current and new problems related to mixing granular materials, it is advisable to look for tools that will allow to analyse the mixing process in a quick and easy, yet precise way. The method of optical fluorescence analysis proposed in the article is based on the well-known field of optics and computer image analysis. Although the literature (presented in this section) presents methods based on the analysis of the dyed component, dyeing with a fluorescent substance allows to eliminate erroneous readings of its content in the case of multi-component mixtures. It is also worth noting that the most common mixing was carried out in two-component systems where each component differed in color. In such systems, image analysis does not pose any problems. In the proposed method, only one component of the feed (maize) was dyed and then its content was assessed against the background of a multi-colored sample. This has resulted in an innovative tool that gives new possibilities to analyse the process of mixing granular materials. What is more, this method has its potential and its development may be still ahead, *e.g.* by upgrading it to on-line sampling. This work brings new knowledge about the evaluation method for multi-component granular mixtures. The proposed approach can be used as a tool to assess technical parameters (such as mixing time, working capacity, speed of agitator, *etc.*) for the industrial mixers.

2. Research methodology

The research was carried out using nine multicomponent grain mixtures with a degree of fineness from 0.54 to 1.26 mm (industrial feeds). They were characterized by differentiation in terms of composition and particle size (Table 1). The particle size distribution of used mixtures was estimated on the basis of PN-R-64798:2009 Feeds – Determination of particle size.

The mixing process was carried out in a flow mixer. Technical parameters of this mixer are presented in Matuszek and Tukiendorf [33]. The process consisted in pouring the mixture from the feeding tank to the receiving tank. Before the mixing process started, the feeding tank was filled with 900 g of ready feed mixture (90% of mixed material). In the upper part of the same tank, 100 g (10% of the mixed material) of tracer was placed. The number of flows was 10 each time. After the mixing was completed, sample taking was carried out. Samples were taken from each ring of the receiving tank. In this way, 10 samples ($N = 10$) with a single sample weight of 10 g were obtained. For each of the nine mixtures, analogous mixing was performed in three repetitions. Thus, 27 tests were performed for a tracer with an average particle size of 1.25 mm and 27 tests for a tracer with an average particle size of 2.00 mm.

The study used a crushed yellow maize tracer with an average particle size of $d_1 = 1.25$ mm and $d_2 = 2.00$ mm. Crushing was carried out in a 580 W grain grinder. Material prepared in this way was wet treated with 0.01% Rhodamine B solution ($C_{28}H_{31}ClN_2O_3$, excitation 553 nm, emission 627 nm, molecular weight 479 g/mol). The choice of dye is the result of previous analyses [34]. Dried maize (mean moisture content of grain weight 13.2% ± 1.5%) was stored in a dry place in closed containers protecting it against light.

The samples taken were placed on 120 × 20 mm Petri dishes and then in a chamber (Fig. 1). The image collection stand was made of black metal sheet. This material protected against light from outside. The sample was placed in the lower part, on a pull-out "tray". A source of ultraviolet

Table 1. Composition and particle size of the mixtures.

Name of the raw material	**Fraction of the raw material**								
	Mixture no.								
	1	2	3	4	5	6	7	8	9
Fodder chalk	1.5	2	8	1.9	9.8	12	9	1.5	7.1
Barley	–	–	2	–	–	–	–	30	–
Maize	36.5	30	10	15.2	20	22	8	9	7
Triticale	15	–	–	5	24.18	19	–	20	–
Soya meal	9	5	60	28	14	12	65.55	12	72
Rape meal	27	35	7	–	–	–	–	5	–
Sunflower meal	–	–	–	–	8	9	–	–	–
Dry maize decoction	10	25	–	–	–	–	5.45	–	4.3
Wheat	–	–	–	46.9	22	24.19	–	20	–
Sodium chloride	0.5	0.5	3	0.5	0.3	0.32	2.5	0.5	2
Phosphate	–	–	3	1	0.7	0.5	3.5	1	2.8
Premix	0.5	2.5	5	0.6	0.5	0.6	2.5	1	2
Methionine	–	–	–	0.24	0.19	0.18	0.5	–	0.4
Lysine	–	–	–	0.1	0.12	0.2	2.5	–	1.8
Phytase	–	–	–	0.01	0.01	0.01	0.05	–	0.05
NEU-SOL	–	–	–	0.3	0.2	–	–	–	–
Acidic sodium carbonate	–	–	–	0.06	–	–	–	–	–
Grindazym	–	–	–	0.01	–	–	0.05	–	0.05
Aroma	–	–	–	0.18	–	–	0.05	–	0.05
Threonine	–	–	–	–	–	–	0.35	–	0.3
Neubaciol	–	–	–	–	–	–	–	–	0.15
Number of components	8	7	8	15	13	12	13	10	14
Particle size [mm]	0.54	0.57	0.58	1.08	1.26	0.62	0.64	0.61	0.63

light was located inside the chamber. After the chamber was closed, the sample was illuminated with UV light through a remote control. As a result of the excitation agent the photoluminescence (fluorescence) of Rhodamine B, covering the tracer was obtained (intense red, Fig. 1). Next, photos of illuminated samples were taken in the BMP file format and 1600×1200 pixel resolution. For this purpose, a 20.1 Mpix digital camera with a standard lens was used. The obtained images were entered into PATAN® software by Marek Krótkiewicz, working in the RGB 256 range. In the studied area the objects responsible for the given component of the mixture sample (tracer and background) were identified by assigning RGB scale values to the pixels. In this way three classes were determined. One for the tracer (fluorescent maize) and two for the background (industrial feed mixture). In this way, information was obtained on the fraction of each class. For further analysis, the results on the fraction of the tracer were used. During this stage of the study it was observed that the use of a fluorescent dye resulted in a sharp distinction of the tracer against the multicolored sample (Fig. 1). This was possible only after Rhodamine B was treated with a photoluminescent inducing agent.

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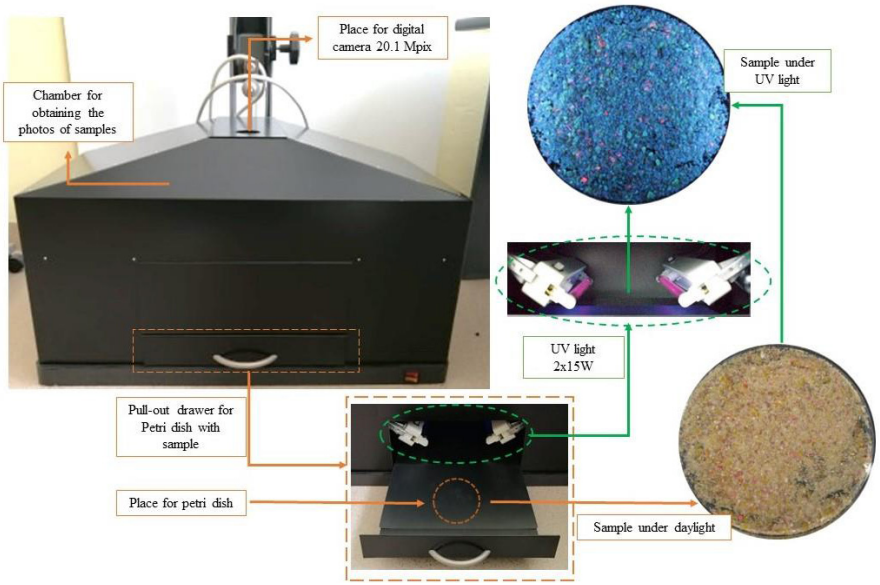

Fig. 1. Test stand – a chamber with UV lighting for taking sample images.

In the last stage, the samples were separated on sieves and the fluorescent maize was manually separated. The tracer was then weighed on an analytical balance with the accuracy of ±0.01 g and its fraction was determined. This method was a validation tool (accuracy verification). In this way the share of the tracer was finally determined by two methods:

– using optical fluorescence analysis – method 1,
– using a balance – method 2 (control).

The coefficient of variation (as 100×standard deviation/mean) was calculated on the basis of the tracer fraction. This parameter can be used to determine the homogeneity of the mixture. It was assumed that for CV < 15% the mixture is considered homogeneous while CV ≥ 15% indicates abnormal concentration of tracer distribution in the mixture (poor homogeneity) [17].

The main aim of the study was to verify whether particle size of the tracer affects the accuracy of the method (the degree of the difference between the results and the control method – hereinafter referred to as "differential modulus"). In other words, whether there is such a diameter of the tracer, which is best suited to the proposed method of determining the homogeneity of mixtures – allows to obtain a lower level of difference in relation to the control method. Therefore, a statistical comparative analysis was made of the results of the fraction of the tracer obtained when using a tracer of average particle size d_1 = 1.25 mm and d_2 = 2.00 mm and the differences obtained in relation to the control method. The main research problems were formulated in questions:

– Are the proposed tracer diameters (1.25 and 2.00 mm) correctly selected in relation to the presented mixtures with the particle size from 0.54 to 1.26 mm?
– Which of the diameters used for computer image analysis gives more accurate results of analysis?

3. Results and discussion

The results of the tracer fraction obtained by the two methods and the degree of mixing are shown in Table 2.

Table 2. Results of the tracer and coefficient of variation obtained using fluorescent maize with average particle size d_1 = 1.25 mm and d_2 = 2.00 mm.

Parameter	Tracer fraction[a] [%]		Differential modulus[a.b]	Tracer fraction[a] [%]		Differential modulus[a.b]
	Method 1	Method 2		Method 1	Method 2	
	Maize d_1 = 1.25 mm			Maize d_2 = 2.00 mm		
	Mixture 1					
Mean, %	8.94 ± 1.26	9.03 ± 1.15	0.60 ± 0.43	9.52 ± 1.62	9.45 ± 1.53	0.32 ± 0.20
CV, %	13.38 ± 2.79	12.26 ± 1.70	1.39 ± 0.77	14.99 ± 7.49	14.85 ± 6.02	1.43 ± 0.86
	Mixture 2					
Mean, %	8.83 ± 1.14	8.76 ± 1.04	0.81 ± 0.61	9.17 ± 1.09	9.17 ± 1.14	0.29 ± 0.13
CV, %	12.58 ± 1.81	11.67 ± 0.41	1.67 ± 0.56	11.52 ± 1.44	11.88 ± 2.09	0.73 ± 0.65
	Mixture 3					
Mean, %	9.32 ± 1.26	9.36 ± 1.11	0.93 ± 0.57	9.03 ± 1.08	8.98 ± 1.12	0.27 ± 0.15
CV, %	12.43 ± 0.87	10.76 ± 1.35	0.60 ± 0.06	11.58 ± 1.41	12.02 ± 1.83	0.89 ± 0.18
	Mixture 4					
Mean, %	8.37 ± 0.42	8.64 ± 0.45	1.34 ± 0.99	5.61 ± 0.56	5.56 ± 0.53	0.20 ± 0.11
CV, %	22.25 ± 1.87	16.94 ± 3.41	5.31 ± 1.63	21.39 ± 5.64	21.05 ± 5.26	0.67 ± 0.15
	Mixture 5					
Mean, %	8.83 ± 0.24	8.97 ± 0.15	0.89 ± 0.32	8.20 ± 0.76	8.25 ± 0.83	0.26 ± 0.14
CV, %	9.94 ± 1.91	11.80 ± 1.74	1.98 ± 1.75	6.96 ± 1.38	7.00 ± 1.02	0.38 ± 0.09
	Mixture 6					
Mean, %	9.37 ± 0.22	9.19 ± 0.33	0.8 ± 0.35	8.12 ± 0.14	8.14 ± 0.08	0.30 ± 0.16
CV, %	7.21 ± 0.98	7.34 ± 0.39	0.79 ± 0.15	6.42 ± 0.28	6.58 ± 0.57	0.35 ± 0.15
	Mixture 7					
Mean, %	9.18 ± 1.01	9.23 ± 0.96	0.50 ± 0.23	8.67 ± 1.08	8.81 ± 1.06	0.34 ± 0.19
CV, %	10.50 ± 1.19	9.83 ± 1.07	0.92 ± 0.34	10.83 ± 1.62	10.37 ± 1.66	0.82 ± 0.28
	Mixture 8					
Mean, %	9.39 ± 0.80	9.29 ± 0.87	0.38 ± 0.23	10.10 ± 1.41	10.04 ± 1.37	0.26 ± 0.12
CV, %	8.25 ± 0.16	9.04 ± 0.28	0.80 ± 0.15	13.46 ± 1.32	13.17 ± 1.60	0.38 ± 0.35
	Mixture 9					
Mean, %	9.19 ± 0.92	9.33 ± .89	0.59 ± 0.30	9.15 ± 0.96	9.23 ± 1.06	0.38 ± 0.22
CV, %	9.75 ± 1.33	9.15 ± 0.76	1.15 ± 0.34	9.55 ± 1.10	10.87 ± 0.87	0.38 ± 0.21

[a] arithmetic mean of 10 samples and 3 series ± standard deviation
[b] differential modulus between the results obtained by the two methods

Graphical interpretation of the tracer fraction obtained by the two methods using the tracer d_1 = 1.25 mm and d_2 = 2.00 mm is presented on selected examples (mixtures 1, 6 and 8) in Figs. 2 and 3.

The analysis of the obtained results for the percentage of the tracer using method 1 (optical fluorescence analysis method) and method 2 (weight, control method) allowed to compare these results and to determine the difference between the values – in the article these differences

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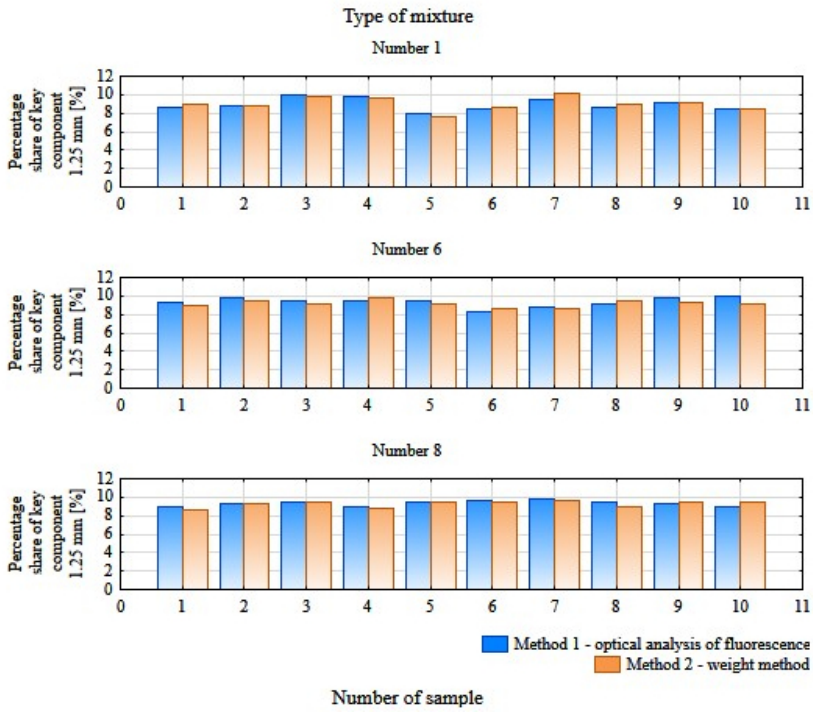

Fig. 2. Average percentage of the tracer $d_1 = 1.25$ mm obtained by two methods for selected mixtures.

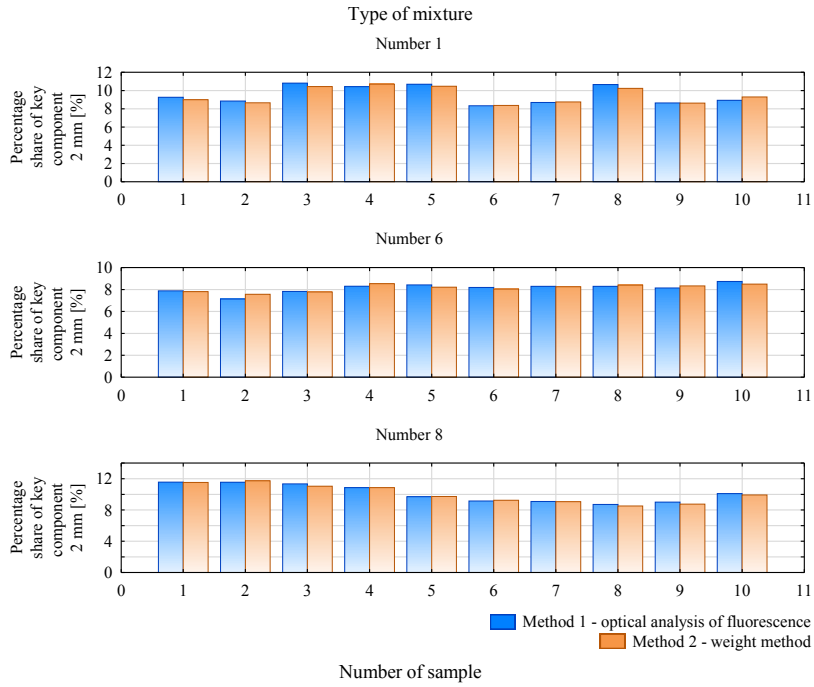

Fig. 3. Average percentage of the tracer $d_2 = 2.00$ mm obtained by two methods for selected mixtures.

were called the differential modulus. For the mixtures with the 1.25 mm diameter tracer these differences were in the range from 0.00 to 2.56 (respectively for mixtures 1–9: mixture 1 (0.01–0.37), mixture 2 (0.02–1.30), mixture 3 (0.07–1.06), mixture 4 (0.19–2.56), mixture 5 (0.03–1.09), mixture 6 (0.14–0.88), mixture 7 (0.02–0.64), mixture 8 (0.00–0.60), mixture 9 (0.04–0.91)). And for the mixtures with the 2.00 mm diameter tracer the differences were in the range from 0.00 to 1.27 (respectively for mixtures 1–9: mixture 1 (0.03–0.42), mixture 2 (0.06–1.27), mixture 3 (0.06–0.25), mixture 4 (0.01–0.27), mixture 5 (0.00–0.36), mixture 6 (0.03–0.43), mixture 7 (0.02–0.48), mixture 8 (0.00–0.29), mixture 9 (0.07–0.38)).

For evaluating homogeneity of mixtures (CV coefficient), differences in the results obtained by the two methods were observed in the range of 0.12–2.35 for mixtures with the 2.00 mm diameter tracer (respectively: mixture 1 (0.27–2.35), mixture 2 (0.21–1.65), mixture 3 (0.68–1.12), mixture 4 (0.48–0.85), mixture 5 (0.31–0.50), mixture 6 (0.19–0).56), mixture 7 (0.54–1.20), mixture 8 (0.12–0.87), mixture 9 (0.98–1.17)). And 0.17–6.82 for mixtures with a 1.25 mm tracer (respectively: mixture 1 (0.40–2.27), mixture 2 (0.98–2.96), mixture 3 (0.88–2.16), mixture 4 (3.05–6.82), mixture 5 (0.17–4.34), mixture 6 (0.65–1.00), mixture 7 (0.38–1.26), mixture 8 (0.58–0.91), mixture 9 (0.82–1.61)).

The magnitude of the differential modulus of the fraction of the tracer obtained by optical fluorescence analysis (method 1) in relation to the control method (method 2) for a tracer with two different average particle sizes for mixtures 1, 6 and 8 is shown in Fig. 4.

In order to verify whether the difference in the results obtained for the nine tested mixtures by the fluorescent method (Method 1) using tracers of different size (d_1 = 1.25 mm, d_2 = 2.00 mm)

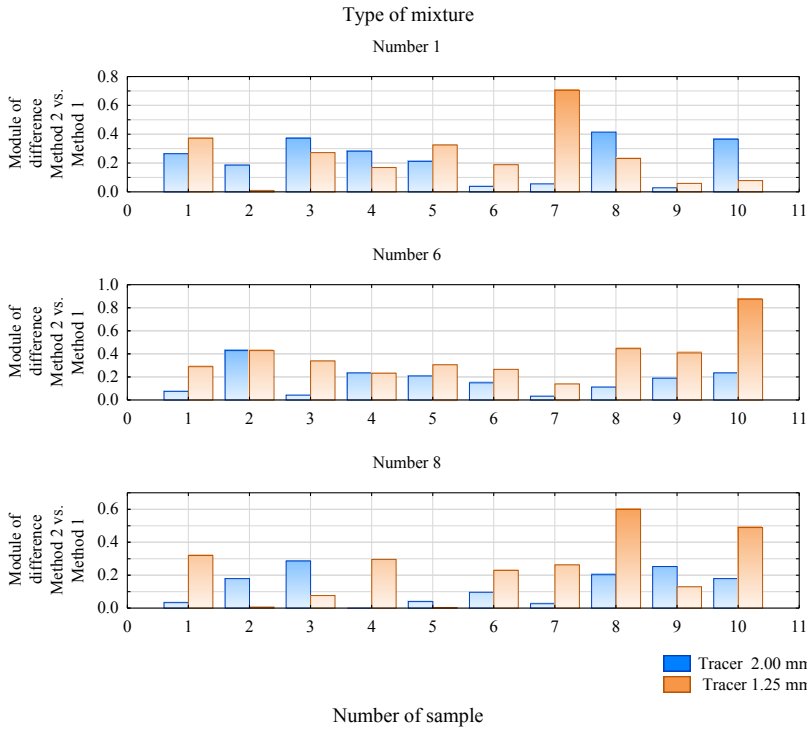

Fig. 4. Differential modulus between the results obtained by the two methods for a 1.25 mm and 2.00 mm tracer for selected mixtures.

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with respect to the control method (Method 2) is statistically significant, calculations were made using the Student's t-test. The use of a given test was preceded by verification of distribution normality (the Shapiro–Wilk test) and the equality of variance (Leven test). A null hypothesis was adopted about the equality of mean results of the differential modulus obtained with the use of a tracer with the mean particle size d_1 and d_2 against an alternative hypothesis about inequality of results. The significance level $\alpha = 0.05$ was assumed. The graphical interpretation of the statistical comparative analysis is presented in Fig. 5.

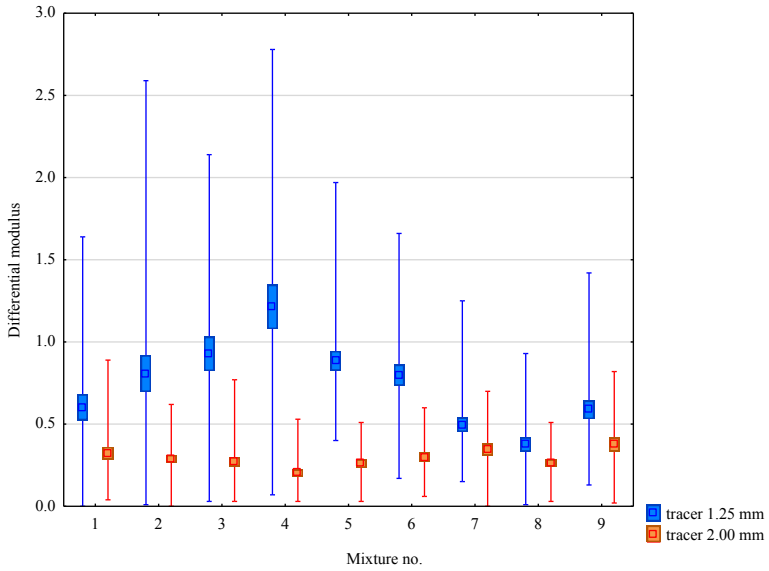

Fig. 5. Graphical interpretation of statistical comparative analysis (point: mean, box: standard error, whiskers: min-max).

The results of the Student's t-test indicated that there were no grounds for assuming the null hypothesis at the level of significance $\alpha = 0.05$. Therefore, in each case (9 mixtures) it was confirmed statistically that the results obtained with the use of the tracer with the average particle size $d_1 = 1.25$ mm are different from those obtained with the use of the tracer with the average particle size $d_2 = 2.00$ mm. The direction of this differentiation is shown in Fig. 5. In the case of mixtures 1–9, the use of the tracer with a larger size ($d_2 = 2.00$ mm) allowed to obtain a lower value of the difference of results in relation to the control method (weight method). Therefore, the tracer of this dimension is better for assessing the quality of loose mixtures by optical fluorescence analysis than the smaller tracer (1.25 mm). From a practical point of view, it is worth assuming that the use of the 2.00 mm diameter tracer allows to obtain results with a smaller error. Therefore, if possible, *i.e.* if the granulometric composition of the mixture allows the use of a tracer with this dimension, it is recommended. On the other hand, in the case of mixtures with a significant degree of fineness (below the fineness of mixtures under test), the 1.25 mm diameter tracer can be used. However, it should be remembered that the results in this case will be subject to greater error. This results from the fact that the assumption of the method is to assess the homogeneity of granular mixtures based on a tracer which has the features of a mixed system, also in terms of particle dimensions.

The basic problem in metrology is the determination of precision, measurement accuracy and the associated true value. According to [35] the accuracy of measurement is "closeness of the agreement between the result of a measurement and a true value", which is related to the

occurrence of random and systematic errors [36, 37]. It should be noted that in many aspects of measurement of granular materials, we do not know the true value which we can refer measurement results to. Therefore, we cannot talk about the accuracy of measurement [38] in the exact sense of this sentence. That is why measurements of this type of materials give rise to numerous metrological and mathematical problems [15].

In the case of the presented solution, the results obtained by optical fluorescence analysis (validated method) were compared with those obtained by a reference method (weight method). The calculated recovery values ranged from 96.9 to 101.9% for 1.25 mm tracer and 98.4 to 100.9% for 2.00 mm tracer. Therefore, the high accuracy of the proposed method can be accepted (assumed acceptance for recovery 95–105%).

Precision can be determined on the basis of an analysis of the difference between the results obtained by the two methods, presented in Table 2. The differences (modulus of difference) are in the range (average values) between 0.38–1.34 for the 1.25 mm tracer and 0.20–0.38 for the 2.00 mm tracer. It can therefore be assumed that the method is highly precise.

The repeatability of the method is also high. This is due, among others, to the fact that each series of tests (the whole process of mixing, determination of the tracer content) was performed in three repetitions and each time the tracer content was determined by optical fluorescence analysis and the control method. Analysing the results presented in the publication (Table 2, Figs. 2–5), the thesis made is highly reliable. However, it is worth remembering about the need to maintain specific parameters of the method, such as type and concentration of the fluorescent substance (0.01% of Rhodamine B), type of the tracer (maize grains), size of individual samples, conditions of image acquisition (chamber, digital camera, type of lighting), image analysis (special software). By observing the guidelines presented in the methodology, it is possible to selectively determine the content of the tracer in the presence of the remaining components of the mixture, regardless of their quantity or degree of fineness.

The detection limit has been defined in numerous tests conducted by the authors. For this purpose, various types of substances with specific concentrations emitting light in response to ultraviolet radiation such as Tinopal, Uranine, Eosine, Rhodamine B were analysed. Studies in this area were carried out for ternary systems of green peas, sorghum, maize [39] wheat, safflower, barley [40] and multicomponent systems (from eight to twenty components) where whole grains [34] and ground components were mixed [41]. As a result of these experiments and the comparison of the results obtained each time with the same reference method (weight method), it was indicated that it is Rhodamine B with a concentration of 0.01% that meets the requirements of other validation parameters such as accuracy and precision.

The introduction section presents the most common methods for evaluation of powder mixing. Special attention is paid to methods using computer image analysis and fluorescence phenomenon. In relation to this information, two basic questions are answered below:

– What makes the method of optical fluorescence analysis innovative?
– What are the advantages of this method and its limitations?

The innovation of the method of optical fluorescence analysis results from the combination of fluorescence in response to ultraviolet radiation with computer image analysis. This solution leads to elimination the interference caused by the tracer being covered by the mixture components, which led to underestimation of its actual content [27]. The excitation of Rhodamine B to the emission of light radiation made it possible to precisely determine its fraction among a great number of different ingredients. Moreover, the tracer introduced is one of the components of the mixture, *i.e.* it has features of a mixed material. The use of an optical method such as Particle Image Velocimetry PIV using fluorescent particles for non-invasive determination of the two-phase system flow field was presented by Yang *et al.* [28]. This solution has reduced the

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error resulting from grayscale image analysis and has enabled to study the interaction between two phases of sand and wind. The utilized UV-induced fluorescence phenomenon eliminates the drawback of the fluorescence spectroscopy method, namely the possibility of incorrect estimation of a given component in the presence of other fluorescent components [32]. Moreover, it allows to retain the advantages of the method based on image analysis, *i.e.* being simple to use and low cost [27]. Moreover, due to the evaluation of the blend uniformity based on fluorescent maize, the method can be applied to multicomponent mixtures that are of a similar colour.

The strengths can therefore be presented in several points: low cost, simple to use, fast in data acquisition, can be used for multicomponent mixtures with different particle sizes, the tracer has the properties of a mixed material. However, this method has certain limitations, namely: requires a special chamber for image acquisition, cannot be used in the industry because Rhodamine B is an irritant chemical, requires sample taking. However, in the authors' opinion proper preparation of the test stand will allow for taking samples (photos) on line (non-invasively), wet method. It also requires the use of a solution of fluorescent substance and, as a consequence, liquid waste production.

4. Conclusions

The innovation of the optical fluorescence analysis method consists in combining the fluorescence phenomenon induced by ultraviolet radiation with computer image analysis. This solution leads to elimination the interference caused by the tracer being covered by the components of the mixture which leads to underestimation of its actual content and therefore measurement errors. The UV-induced fluorescence phenomenon used eliminates the drawback to the fluorescence spectroscopy method, namely the possibility of incorrect estimation of a component in the presence of other fluorescent components. The fluorescence optical analysis method can be used to assess the homogeneity of granular mixtures. The method has many advantages, *i.e.* low cost, it is simple to use, data acquisition is fast, it can be used for multicomponent mixtures with different particle sizes, the tracer has properties of mixed material. However, this method has some limitations, namely: it requires a special chamber for image acquisition, it cannot be used in the industry because Rhodamine B is an irritant chemical, it requires sampling, it is a wet method, it requires a solution of fluorescent substance. The repeatability of the method is also high.

Detailed conclusion which might be useful in industrial condition are as follows:

1. Precision can be determined by analyzing the difference between the results obtained by the two methods. The differences (mean values) are in the range between 0.38–1.34 for the 1.25 mm tracer and 0.20–0.38 for the 2.00 mm tracer. It can therefore be assumed that the method is highly precise.
2. The new method of optical fluorescence analysis was compared with the weight (reference) method. On this basis the difference between the values – the differential modulus was calculated. The use of a larger size of tracer (d_2 = 2.00 mm) made it possible to obtain lower values of this difference, i.e. from 0.00 to 1.27, and for mixtures with the 1.25 mm diameter tracer the differences were in the range from 0.00 to 2.56.
3. Using a tracer with the diameter of 2.00 mm allows for results with a smaller error. Therefore, if this is possible, *i.e.* if the granulometric composition of the mixture allows the use of a tracer with this dimension then it is recommended. On the other hand, in the case of mixtures with a significant degree of fineness (below the fineness of mixtures under test), the 1.25 mm diameter tracer can be used. However, it should be remembered

that the results in this case will be subject to greater error. This results from the fact that the assumption of the method is to assess the homogeneity of granular mixtures based on a tracer which has the features of a mixed system, also in terms of particle dimensions.

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