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Method for determining homogeneity of fine dispersed mixtures based on the software analysis of photo cross-cut of the sample

Abstract. *The paper analyzes the main methods for determining the homogeneity of fine dispersed mixtures, as a result of which it has been found that all of them are characterized by significant complexity and low implementation rate. There has been developed a highly effective photoanalytical method for assessing homogeneity, which is based on software analysis of digital photo cross-cut of the mixture and determining the degree of its homogeneity based on comparison of the obtained color identifiers with the values of the reference database.*

Streszczenie. *W pracy przeanalizowano główne metody określania jednorodności drobno zdyspergowanych mieszanin, w wyniku czego stwierdzono, że wszystkie charakteryzują się dużą złożonością i niskim stopniem wdrożenia. Opracowano wysoce efektywną fotoanalityczną metodę oceny jednorodności, która opiera się na programowej analizie cyfrowego fotoprzekroju mieszaniny i określeniu stopnia jej jednorodności na podstawie porównania otrzymanych identyfikatorów barw z wartościami bazy referencyjnej .* (***Metoda wyznaczania jednorodności drobno zdyspergowanych mieszanin na podstawie analizy programowej foto-przecięcia próbki***)

Keywords: index coding of color, generalized identifier, photoanalytical method, algorithm, pixel.
Słowa kluczowe: indeksowanie koloru, identyfikacja, fotoanaliza

Introduction

Preparation of bulk mixtures is an integral part of many modern technological processes in the agro-industrial complex of the country [1, 2]. Homogeneity of the initial mixtures significantly affects the formation of various physical and mechanical properties and qualitative characteristics of the final product [3, 4].

The main problem of this technological operation is a considerable complexity and low speed of determining the homogeneity of fine dispersed mixtures with the particle size less than 1 mm [5], which causes the need to search and develop new methods for assessing the quality of mixing the components.

Analysis of recent research and publications

All existing methods of quantitative analysis of samples of a mixture of bulk materials regarding the content of a key component are divided into two groups [6, 7]. The methods of the first group are designed for direct quantitative analysis of the components of the mixture sample without prior dissolution [8, 9]. They include mostly gravimetric, radiometric and photographic methods. The second group includes methods that require pre-dissolution of a mixture sample in a suitable liquid [10]. This group includes chemical, conductometric, potentiometric, optical and some other methods of analysis of solutions [11].

Gravimetric methods of the first group involve the division of a mixture sample of loose materials into constituent components and their subsequent weighing. These particles of the mixture have a relatively large size (more than 2 mm), and their affiliation to a component can be determined by appearance (color, shape) and divided manually [12].

It is much more difficult to divide a sample of a mixture of fine dispersed materials into components [6, 13]. Well-known methods of the division of bulk materials involve sieving on sieves, separation in a magnetic field or in air separators [9, 14]. Sieving is used when the particles of the key component are a fraction that does not include particles of other components. This method of component separation is practically not suitable for the analysis of light weight samples [7, 15, 16]. The second method of separation is used when one of the components can be separated due to its magnetic properties [10, 17].

Gravimetric methods of the second group involve dissolving a sample of the mixture in a suitable liquid and further determining the weight of the component that went into solution and the residue [18, 19, 20]. These methods are time-consuming because they include operations of dissolution, filtration, drying, weighing and are characterized by low accuracy [4].

Among the chemical methods of analysis, the method of titration has become the most widespread. According to this method, a specially selected reagent (titrant) selectively reacts with a key component in solution [9, 15]. The disadvantages of this method are that the potential is not always rapidly established after the addition of the titrant as well as the need to conduct a large number of samples in many cases.

The conductometric method for determining concentration of the substance (electrolyte) dissolved in water is based on the ability of such solutions to conduct electric current. This ability is characterized by the value of electrical conductivity and depends on the concentration and nature of the solute. However, this method is not suitable if during the dissolution of the investigated portion of the mixture of bulk materials in water, substances with approximately the same mobility of ions are detected in solution [11, 12].

Considering the shortcomings of the above-described methods, there is a need to develop highly effective express methods for assessing the quality of mixing fine dispersed components with particle sizes ranging within 0.8-0.05 mm, which will be accurate enough for production needs, characterized by high speed and ease of homogeneity analysis, have no need to use expensive specialized equipment and materials and be safe for the environment and human life and health.

Purpose and tasks of research

The study is aimed to create a tool to improve the efficiency of product quality management due to the development of the method for express assessment of the homogeneity of a mixture of fine dispersed materials using

a device for computer digital imaging and software MathCad 10.0.

Research results

To achieve this goal, the staff of the Laboratory of Mechatronics and Robotics of Vinnytsia National Agrarian University proposed a photoanalytical method for assessing homogeneity, based on digital analysis of photo cross-cuts of mixtures in MathCad 10.0 with sequential identification of color of each pixel of the image.

Identification of color is carried out using the method of index coding in which each color shade is represented by one number, and this number expresses not the color of the pixel, but the index of color (its number) from the accompanying palette. This index palette specifies identifiers of color for the components of a particular fine dispersed mixture to be included in the reference database of the software algorithm. Further software analysis of photo cross-cut of mixtures in MathCad 10.0 involves determination of the generalized identifier of color and calculation of the homogeneity of the mixture using a polynomial regression equation. The regression equations for each type of mixture are obtained by regression-interpolation analysis of data on calibration studies and establishing relationships between pre-known reference values of the degree of mixing and numerical identifiers of color of the series (generalized identifier), which are also included in the reference database.

In order to validate the proposed express method for determining homogeneity, laboratory tests were conducted to determine the homogeneity of the simulation mixture containing two food components: table salt (NaCl) of extra fraction and coriander powder (Coriandrum sativum) in equal mass proportions.

Samples taken for analysis should be characteristic, that is, have such a weight that random deviations in them, the ratio of components, do not obscure the overall picture of the distribution of the substance over the volume of the controlled mixture. The minimum allowable sample weight Gm must ensure the reliability of the assessment of the quality of the mixture and is determined as follows [21, 22]:

(1) $$G_m = \frac{n \cdot 100}{c_0} \cdot \frac{\pi}{6} d^3 \rho,$$

n - the number of particles of the key component in the minimum weight sample; d—particle diameter, cm; ρ - the density of the particle material, g/cm.

The smallest deviation of the number of particles of the key component in samples from their theoretical number corresponding to the value co is ±1 pc. To ensure that such deviations do not change the co value by more than 6%, the minimum allowable sample weight is determined by the following formula [21]:

(2) $$G_m = \frac{100}{\delta c_0} \cdot \frac{\pi}{6} d^3 \rho l.$$

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(3) $$\frac{n-(n_ч-1)}{n} \cdot 100 = \delta\% \text{ , } n = \frac{100}{\delta}$$

For a real mixture with a normal law of concentration distribution in the samples of the key component, with a probability of 0.997, we consider that the maximum and minimum concentrations differ by no more than ±35%. In view of the fact that we are interested in the minimum allowable weight of the sample, and the concentration of the key component is included in the denominator of formula (3), for a real mixture, instead of c_o, we substitute the upper limit of the values of the average concentration of the key component in the samples, that is $c_o + 35$ [23]:

(4) $$G_m = \frac{10^4 \cdot \pi}{\delta(c_0+35)} \cdot d^3 \rho$$

In a real mixture, the particle size is not always the same, therefore, instead of d, we substitute the arithmetic mean particle diameter of the key component. If the bulk material is prone to lumping, then instead of d one should substitute the diameter of the lump dk, and instead of d for ρ – arithmetic mean bulk density of lumps $\rho_н$ [24]. Importance δ should be chosen so that the change in the number of particles in the sample by 1 pc does not affect the values determined by the analyzes c_i, can be accepted δ = 1,0%. The value of S may be unknown for a given controlled mixture; it can be set, given that most mixers achieve a mixture quality of at least V_c = 50% [15, 25]. Then $S = 0.5 \cdot c_o$.

The procedure of express homogeneity assessment includes the following steps:

1. Sampling of a mixture weighing 50 grams.

2. Even distribution of the selected volume of the individual sample in a rectangular transparent container or in a monochrome green container.

3. Obtaining a computer digital image in one of the ways:

- placing a transparent container with a breakdown of the mixture on the canvas of the tablet scanner (Fig. 1, a), scanning the bottom of the container and saving the image to a computer;

- when using the green container, it is necessary to take a photo (in a horizontal plane) of the leveled surface of the mixture on a digital camera or smartphone camera (Fig. 1, c) and save the image on a computer;

A green container and a smartphone camera were used in the laboratory tests.

4. Forming a digital profile of the mixture by converting the image to the desired pixel ratio. (Fig. 1, b, Fig. 1, d).

During laboratory tests, the image downloaded to the computer was converted to a pixel ratio of 600x600 (Fig. 2, a). Then, using MathCad 10.0 software, the obtained image was decomposed into a two-dimensional matrix containing 360,000 cells, assigning a color identification to each pixel from 0 – “black” to 255 – “white” color (Fig. 1, c). So, each cell contains the identification value of color of the corresponding pixel on the photo cross-cut of the resulting mixture.

5. In order to form a reference database for a mixture of salt and coriander powder, a number of calibration experimental studies were performed, which allowed to determine the degree of homogeneity of each sample. Subsequently, statistical analysis of the data of two-dimensional matrices obtained by processing photo cross-cuts of each of the selected samples, for which the degree of homogeneity was already known, was performed. As a result of processing such data with numerical identifiers of color, the distribution of average values of generalized identification values of color was determined according to the values of the degree of homogeneity for the mixture of salt and coriander powder (Fig. 3).

6. Based on the obtained identification values of color of a series of the mixture cross-cut under pre-known degree of mixing (Fig. 3), regression-interpolation analysis of data was carried out and polynomial equation of the 4th degree (5), which characterizes the homogeneity distribution depending on the numerical value of the identifier, was constructed:

(5) $$M = 704.46 \cdot x^4 - 1.72 \cdot 10^3 \cdot x^3 + 1.5 \cdot 10^3 \cdot x^2 - 598.7 \cdot x + 282.6$$

x - a global identifier.

7. Introduction of regression equation (5) in MathCad 10.0 (Fig. 4, b) is the final stage in the development of the software part of the photoanalytical method of express assessment of the

homogeneity of bulk mixtures and filling the reference database for the simulation mixture. The equation can significantly speed up the use of the proposed method in the future and can be used to determine the degree of homogeneity of the two-component mixture of salt and coriander powder.

To verify the validity of the proposed method, a control experiment was performed. According to its program, the degree of homogeneity for 5 selected samples of two-component mixture of salt and coriander powder was determined by the chemical method on the certified high-precision equipment and the suggested photoanalytical method. To compare the obtained results, a diagram was constructed (Fig. 5).

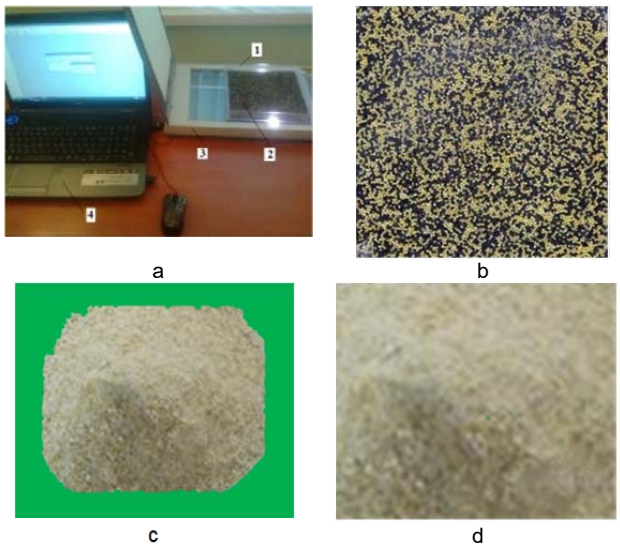
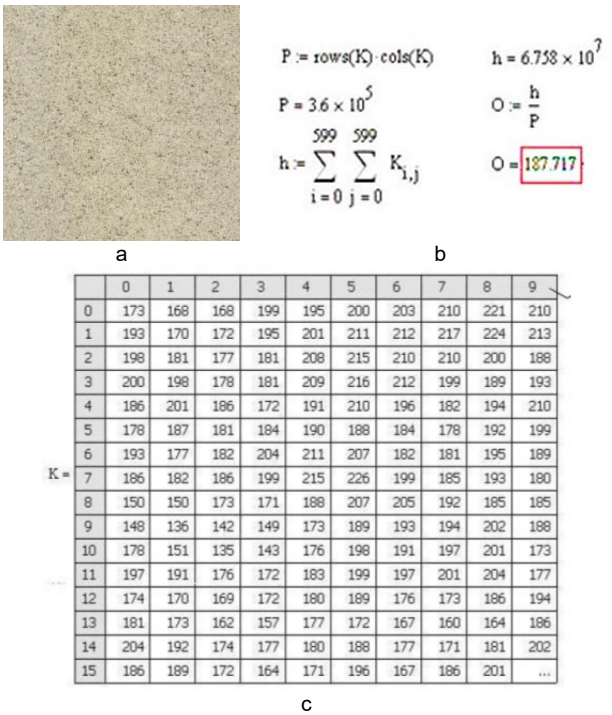


Fig.1. Options for obtaining a computer digital image: a - scanning; b, d – converted photosection image after scanning and photography, respectively; c - digital photography on a green background; 1 - transparent baking sheet; 2 - initial mixture; 3 – scanner; 4 - computer.



	0	1	2	3	4	5	6	7	8	9
0	173	168	168	199	195	200	203	210	221	210
1	193	170	172	195	201	211	212	217	224	213
2	198	181	177	181	208	215	210	210	200	188
3	200	198	178	181	209	216	212	199	189	193
4	186	201	186	172	191	210	196	182	194	210
5	178	187	181	184	190	188	184	178	192	199
6	193	177	182	204	211	207	182	181	195	189
7	186	182	186	199	215	226	199	185	193	180
8	150	150	173	171	188	207	205	192	185	185
9	148	136	142	149	173	189	193	194	202	188
10	178	151	135	143	176	198	191	197	201	173
11	197	191	176	172	183	199	197	201	204	177
12	174	170	169	172	180	189	176	173	186	194
13	181	173	162	157	177	172	167	160	164	186
14	204	192	174	177	180	188	177	171	181	202
15	186	189	172	164	171	196	167	186	201	...

Fig. 2. Fragments of the listing for software analysis of the mixture homogeneity by photo cross-cut in MathCad 10.0: a – photo-section of the sample simulation mixture; b – determination of the average value of the generalized identifier of color; c – identification matrix of color.

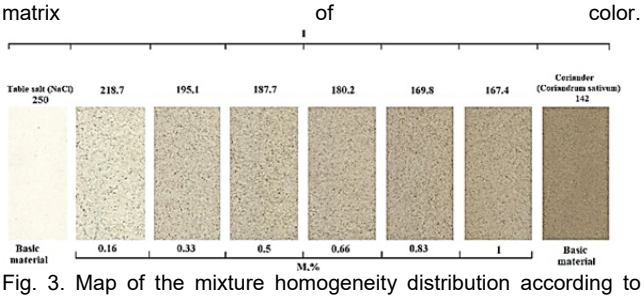
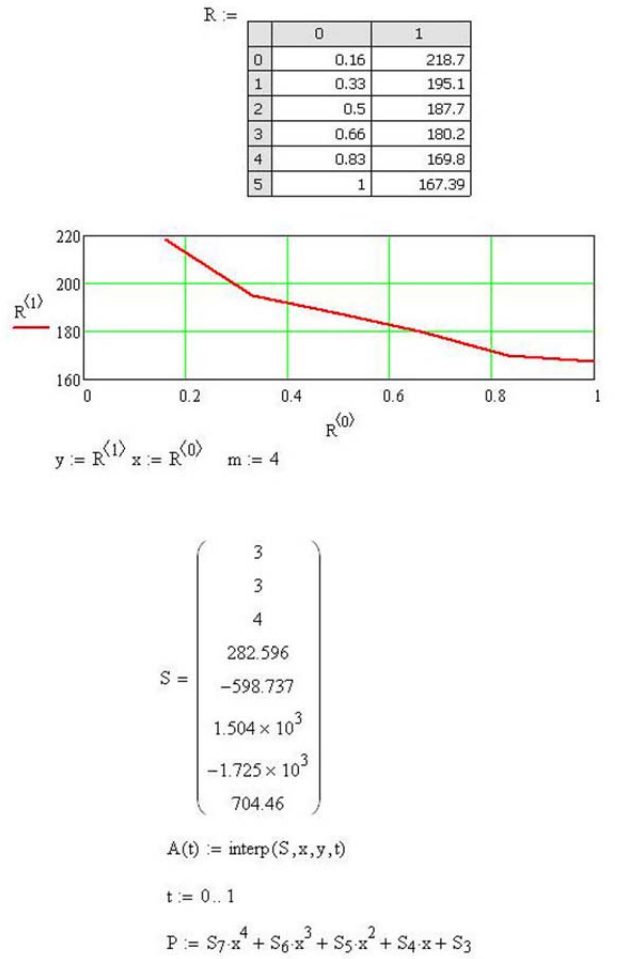


Fig. 3. Map of the mixture homogeneity distribution according to color: I – identification value of color; M – degree of the mixture homogeneity according to color,%



	0	1
0	0.16	218.7
1	0.33	195.1
2	0.5	187.7
3	0.66	180.2
4	0.83	169.8
5	1	167.39

Fig. 4. Fragment of the listing regression-interpolation analysis of the data of the distribution map of the homogeneity of the mixture

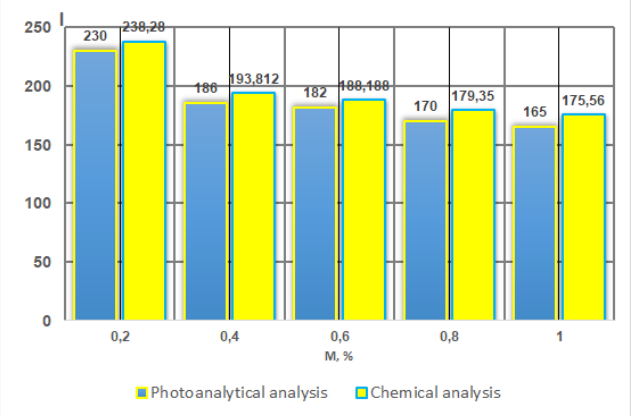


Fig. 5. Results of a control experiment to determine the degree of homogeneity for 5 selected samples of a two-component mixture of salt and coriander powder

According to the diagram (Fig. 5), a relative deviation in the results of chemical and photoanalytical analysis ranges within 4.6…7.4%, which indicates suitability for further application of photoanalytical method to assess the homogeneity of fine dispersed mixtures.

Conclusions

Thus, as a result of research, a highly efficient and inexpensive express method for assessing the quality of mixing fine dispersed components with the particle size ranging within 0.8-0.05 mm was offered, a software algorithm for its implementation in MathCad 10.0 was developed, and a step-by-step procedure for performing express assessment of homogeneity and formation of the reference database for the study of other types of multicomponent mixtures was described.

Applicability of the proposed method was confirmed by a sufficiently high level of accuracy of the results obtained using the photoanalytical method. Compared to the results obtained due to chemical analysis, which is one of the most modern high-precision methods, the discrepancy ranges within 3.6…6.4%, which is sufficient enough for the vast majority of production processes in Ukraine.

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