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Spis treści

01	Mohamed M. EL-Shafhy, Alaa M. Abdel-hamed, Ebrahim A. Badran - Ferorezonans w sieciach rozdzielczych – stan wiedzy	1
02	Riad Bouzidi, Ismail Ghadbane, Mohammed Boukhari - Badanie eksperymentalne silnika indukcyjnego z wykorzystaniem metody sterowania skalarnego stosowanego w pojeździe elektrycznym	16
03	Abdelghafour HERIZI, Riyadh ROUABHI, Abderrahim ZEMMIT - Badanie porównawcze działania przesuwne, ślizgowo-rozmytego typu 1 i ślizgowo-rozmytego typu 2 maszyny synchronicznej z magnesami trwałymi	21
04	Aleksander OSTROWSKI, Dariusz KANIA - Uniwersalny samochodowy miernik spalania LPG/CNG	30
05	Rachid Dehini, Ryma Berbaoui, Othmane Abdelkhalek - Analizy rozpyłu mocy i stabilności systemu UPQC zintegrowanego z siecią dystrybucyjną.)	34
06	Nur HAMZAH, Firman FIRMAN, Muhammad Ruswandi DJALAL - Analiza charakterystyki paneli słonecznych na dachówkach ceramicznych i ceramicznych	39
07	Muhammed A Ibrahim, Ahmed Nasser B. Alsammak - Sterowanie prędkością silników z przełączaną reluktancją za pomocą zoptymalizowanego regulatora PID	46
08	Oleh TSURKAN, Ihor KUPCHUK, Yurii POLIEVODA, Oleksandr WOZNIAC, Yaroslav HONTARUK, Yrii PRYSIAZHNIUK - Cyfrowe przetwarzanie sygnałów jednowymiarowych w oparciu o algorytm filtrowania medianowego	51
09	Kamal BENABDALLAH, Abdallah Mansouri, Abdelhafid OMARI, DAAOU Bachir, Ramzi SALIM, Mourad BOUFADENE - Tryb przesuwany steruje napędem silnika indukcyjnego pod nieznanym momentem obciążenia za pomocą algorytmu super skręcania	57
10	Mokhtar BOUANANE, Abdelmadjid MENAD - Projekt i wdrożenie sterowania prędkością silnika BLDC w oparciu o regulator PI z wykorzystaniem dspic30f4011 i falownika IPM	62
11	Ahmad F. Al-allaf, Bashar H. Asker - Wykrywanie użycia telefonu komórkowego podczas testu online z wykorzystaniem technologii AI	67
12	Larbi MOURAD, Khelifi MUSTAPHA, Lahcene MOHAMED RIDA – Efektywne podejście do optymalizacji parametrów w systemie przesyłowym stosowane w DWT+SPIHT z wykorzystaniem algorytmu wielorybów (WA)	71
13	Chandrasekha Mohanachandran, Srinivasan Sumathi - Ocena wydajności kodów korekcji błędów przewodzenia do projektowania modemu komunikacyjnego linii elektroenergetycznej	79
14	Layth S Salman, Harith Al-Badrani - Modelowanie matematyczne i projektowanie inżynierskie wielopoziomowego falownika w oparciu o selektywną eliminację harmonicznych	83
15	Seyf Allah ZAANOUNE, Ahmed Wahid BELARBI, Hocine MOULAI, Abderrahmane ZIANI - Badanie mieszanin sześciopierścieni siarki i azotu skażonych oparami miedzi w wyłącznikach wysokiego napięcia	87
16	Riny Sulistyowati, Hari Agus Sujono, Yudha Cipta - Optymalizacja rozmieszczenia generacji rozproszonej za pomocą metody optymalizacji roju cząstek w podajniku Sampang	92
17	Valerii SEMENETS, Iryna SVYD, Ivan OBOD, Oleksandr MALTSEV, Waldemar WÓJCIK, Mariia TKACH, Sviatoslav STAROKOZHEV - Metoda zwiększania względnej przepustowości żądających systemów radarowych	97
18	Mykhaylo ZAGIRNYAK, Viktor LYASHENKO, Elena KOBILSKAYA - Modelowanie pola elektromagnetycznego anteny	102
19	Tetyana KORENKOVA, Viktoriya KOVALCHUK, Oleksandr SERDIUK, Mohamed Zaidan Qawaqzeh - System ochrony hydrodynamicznej pompowni w przypadku awaryjnego wyłączenia zasilania	105
20	Yurii POLIEVODA, Ihor KUPCHUK, Yaroslav HONTARUK, Iryna FURMAN, Mykola MYTKO - Metoda wyznaczania jednorodności drobno zdyspergowanych mieszanin na podstawie analizy programowej foto-przecięcia próbki	109
21	Mariusz KRAJEWSKI, Sergiusz SIENKOWSKI - Analiza właściwości iteracyjnego algorytmu najmniejszych kwadratów w pomiarze częstotliwości sieci energetycznej	114
22	Piotr POWROŹNIK, Miłosz CWIETNIEWIC - Wpływ zastosowania technologii VPN na czas przesyłania danych pomiędzy urządzeniami zgodnymi z koncepcją Internetu Rzeczy	118
23	Maciej MALINOWSKI, Krzysztof KUBICZEK, Marian KAMPIK - Precyzyjne szerokopasmowe współosiowe boczniki prądowe	123
24	Kordian DUDZIK, Krzysztof MUSIOŁ, Marian KAMPIK - System do badania liniowości precyzyjnej aparatury pomiarowej	127
25	Łukasz SOBOLEWSKI - Jakość prognozowania skali czasu nadzorowanej przez pierwotny wzorzec częstotliwości z zastosowaniem sieci neuronowej typu GMDH na przykładzie skali czasu UTC(NPL)	131
26	Leszek FURMANKIEWICZ, Mirosław KOZIOŁ, Ryszard RYBSKI, Robert SZULIM - System pomiarowy do badania czujników prądu i przemieszczenia liniowego w trybie zdalnym	135
27	Sergiusz SIENKOWSKI, Mariusz KRAJEWSKI - O estymacji wartości średniej napięcia sinusoidalnego	139
28	Wiesław MICZULSKI, Oliwia PERS - Optymalizacja wykorzystania energii elektrycznej z turbiny wiatrowej w gospodarstwie domowym	143
29	Łukasz MACIOSZEK, Sylwia ANDRZEJCZAK-GRZĄDKO, Joanna URBANIAK, Katarzyna DEMBIŃSKA - Zmodyfikowana metoda impedymetryczna w ocenie zanieczyszczeń mikrobiologicznych piwa niepasteryzowanego	147
30	Tomasz GARSTKA, Marlena KRAKOWIAK, Marcin KWAPISZ - Zintegrowana głowica pomiarowa do badań metodą Barkhausen	151
31	Jerzy ROJ, Łukasz DRÓZDZ - Metoda szacowania niepewności wielkości wyjściowych torów pomiarowych wykorzystujących algorytmy dyskretnej transformacji falkowej	155
32	Ryszard BOGACZ, Beata KRUPANEK - Wprowadzenie do 6G. Specyfikacja, technologie, wyzwania	160
33	Beata KRUPANEK, Ryszard BOGACZ - Wymagania Internetu Rzeczy a możliwości systemu 6G	165
34	Łukasz MACIOSZEK, Norbert ŁUKANISZYN, Jakub KOSTECKI, Ryszard RYBSKI, Urszula KOŁODZIEJCZYK - Koncepcja i implementacja systemu pomiarowego do zdalnego monitorowania stanu wału przeciwpowodziowego nad Odrą	169
35	Krzysztof DOWALLA, Piotr BILSKI, ROBERT ŁUKASZEWSKI, Augustyn WÓJCIK, Ryszard KOWALIK - Wpływ częstotliwości próbkowania na skuteczność systemu NILM z analizą sygnałów w dziedzinie czasu	173
36	Małgorzata AUGUSTYN - Rozpoznawanie cyfr arabskich pisanych ręcznie na podstawie sygnałów z 3-osiowego akcelerometru za pomocą metody DTW z uwzględnieniem różnych kryteriów detekcji	177
37	Magdalena NIZIOŁ - Opracowanie oprogramowania do obsługi systemu pomiarowego zgodnego ze standardem IEEE 1451 w środowisku LabVIEW	181

Spis treści

38	Piotr OCHAŁ, Robert HANUS, Marcin ZYCH - Zastosowanie konwolucyjnej sieci neuronowej do identyfikacji struktury przepływu ciecz-gaz w rurociągu z wykorzystaniem histogramów sygnałów z detektorów scyntylacyjnych	185
39	Barbara WILK - Wyznaczanie wybranych parametrów sygnału PPG i ocena ich zmienności podczas testu zaciskania pięści	190
40	Damian HALLMANN, Piotr Jankowski - Badania symulacyjne wpływu wybranych zakłóceń napięcia zasilania na prądy silnika indukcyjnego małej mocy	195
41	Adam PILSNIAK, Szymon PASKO, Przemysław ZACKIEWICZ, Aleksandra KOLANO-BURIAN1 - Zmodyfikowana metoda balistyczna do wyznaczania charakterystyki magnesowania	201
42	Adam CICHY - Pomiar współczynnika strat dielektrycznych metodą dwóch woltomierzy	205
43	Tadeusz BIAŁOŃ, Roman NIESTRÓJ, Tomasz ROGALA, Wojciech SKARKA - Innowacyjne zasilanie autonomicznego robota mobilnego ogniwem paliwowym	209
44	Artur PASIERBEK - Straty mocy w liniach kablowych wysokiego napięcia	215
45	Emil KRÓL, Marcin MACIAŻEK - Metody analizy wibroakustycznej silników z magnesami trwałymi	218
46	MICHAŁ GOCKI, AGNIESZKA JAKUBOWSKA-CISZEK, PIOTR PRUSKI - Pomiarowa estymacja parametrów modeli ułamkowego rzędu nowej klasy symetrycznych superkondensatorów polimerowych	224
47	Sebastian RÓŻOWICZ, Kamil P. PADUSZYŃSKI, Henryk WACHTA - Analiza kontrastu ujemnego na przejściach dla pieszych	229
48	Paweł PIĄTEK, Paweł KOCWA, Łukasz ZAWADA - Ocena jakości sprężania połączeń śrubowych	234
49	Edyta KUCHARSKA, Katarzyna GROBLER-DĘBSKA, Jerzy BARANOWSKI, Waldemar BAUER, Natalia KASHPRUK - Systemy ERP w energetyce - możliwości i wyzwania	239
50	Milena KURZAWA - Zastosowanie algorytmu Lanczos via Pade do wyznaczania wartości parametrów schematu zastępczego dławika	243
51	Bartosz HAŁUSZCZAK, Łukasz PUTZ, Arkadiusz DOBRZYCKI, Stanisław MIKULSKI - Hybrydowa instalacja OFF-GRID jako źródło zasilania w budynku lotniskowym	248
52	Michał FILIPIAK, Ryszard NAWROWSKI, Dariusz KURZ, Remigiusz PIERZCHAŁA - Analiza Opłacalności Stosowania Instalacji Fotowoltaicznej We Współpracy Z Magazynem Energii Dla Domu Jednorodzinnej	253
53	Dariusz KURZ, Ryszard NAWROWSKI, Michał FILIPIAK, Wojciech WĘGRZYN - Analiza możliwości zarządzania i rozdziału energii elektrycznej, wyprodukowanej w prosumenckiej instalacji fotowoltaicznej, w budynku z automatyką budynkową	259
54	Jacek BARTMAN, Tadeusz KWATER, Bogdan KWIATKOWSKI, Damian MAZUR - Analiza zborności parametrów odbiorników energii elektrycznej w kontekście bezinwazyjnej identyfikacji urządzeń	265
55	Tomasz KRASZEWSKI, Anna PIWOWAR, Renata FRĄCZEK, Piotr ŚWISZCZ, Łukasz BIS - Przegląd metod wizyjnej detekcji	269
56	Tomasz KRASZEWSKI, Radosław SOKÓŁ, Artur PASIERBEK, Marcin POŁOMSKI, Anna PIWOWAR - Ocena metod fotogrametrycznych w szybkiej identyfikacji wad	273
57	Tadeusz BIAŁOŃ - Optymalizacja wzmocnień obserwatora Luenbergera silnika indukcyjnego metodą roju cząstek	277
58	Kazimierz Kawa, Edyta Kucharska, Rafał Mularczyk - Wpływ sterowania węzłami cieplnymi na koszty eksploatacji zespołów nieruchomości	284
59	Barbara KULESZ, Andrzej SIKORA, Damian SŁOTA - Narzędzie identyfikacji parametrów układu izolacyjnego maszyny elektrycznej	292
60	Damian GŁUCHY, Grzegorz TRZMIEL - Analiza wpływu charakterystyk mocy turbin wiatrowych na ilość wytwarzanej energii	296
61	Piotr LEGUTKO - Zastosowanie i wpływ koncentratora pola magnetycznego w procesie hartowania indukcyjnego kół zębatych	301
62	Sebastian ŁACHECIŃSKI - Przetwarzanie danych temporalnych dla wymiaru czasu transakcyjnego na platformie MariaDB	307
63	Mohammad KAZEMINEJAD, Mozhdeh KARAMIFARD - Optymalny i ekonomiczny projekt samodzielnego hybrydowego systemu energii odnawialnej zintegrowanego z magazynowaniem baterii przy użyciu algorytmu sztucznego pola elektrycznego	313
64	Ryszard SROKA, Antoni CIEŚLA, Mikołaj SKOWRON - „Nauki elektryczne” w Akademii Górniczo-Hutniczej w Krakowie – wczoraj i dziś	320

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 zdyspersgowanych 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 zdyspersgowanych 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 G_m must ensure the reliability of the assessment of the quality of the mixture and is determined as follows [21, 22]:

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

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 c_0 is ± 1 pc. To ensure that such deviations do not change the c_0 value by more than 6%, the minimum allowable sample weight is determined by the following formula [21]:

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

3 with considering

$$(3) \quad \frac{n - (n_0 - 1)}{n} \cdot 100 = \delta\%, \quad n = \frac{100}{\delta}$$

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 $\pm 35\%$. 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_0 , we substitute the upper limit of the values of the average concentration of the key component in the samples, that is $c_0 + 35$ [23]:

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

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 ρ_H [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 $\delta = 1,0\%$. 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 $S = 0.5 \cdot c_0$.

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:

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

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).

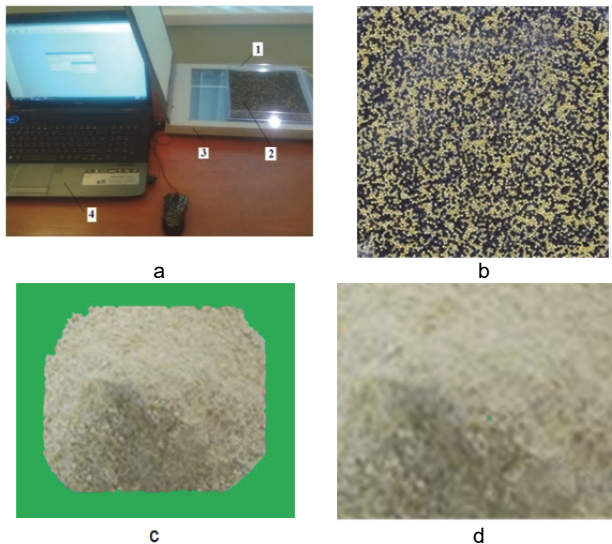


Fig.1. Options for obtaining a computer digital image: a - scanning; b, d - converted photo section image after scanning and photography, respectively; c - digital photography on a green background; 1 - transparent baking sheet; 2 - initial mixture; 3 - scanner; 4 - computer.

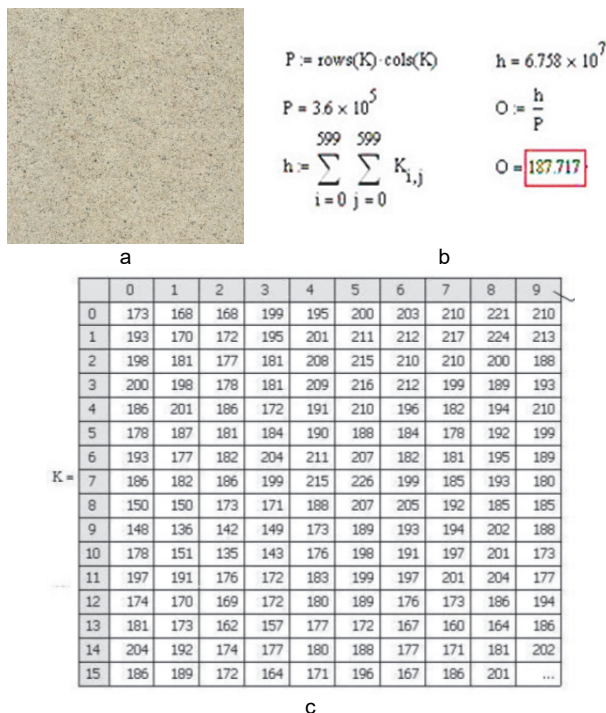


Fig. 2. Fragments of the listing for software analysis of the mixture homogeneity by photo cross-cut in MathCad 10.0: a - photo-section of the sample simulation mixture; b - determination of the average value of the generalized identifier of color; c - identification

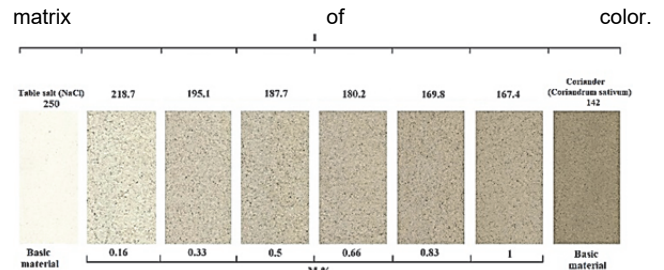
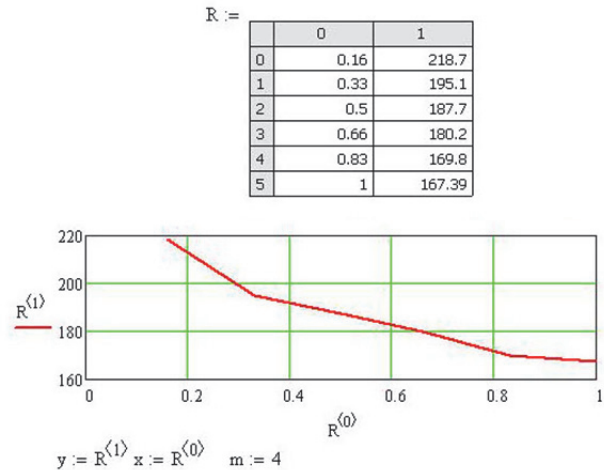


Fig. 3. Map of the mixture homogeneity distribution according to color: I - identification value of color; M - degree of the mixture homogeneity according to color, %



$$S = \begin{pmatrix} 3 \\ 3 \\ 4 \\ 282.596 \\ -598.737 \\ 1.504 \times 10^3 \\ -1.725 \times 10^3 \\ 704.46 \end{pmatrix}$$

$A(t) := \text{interp}(S, x, y, t)$
 $t := 0..1$
 $P := S_7 \cdot x^4 + S_6 \cdot x^3 + S_5 \cdot x^2 + S_4 \cdot x + S_3$

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

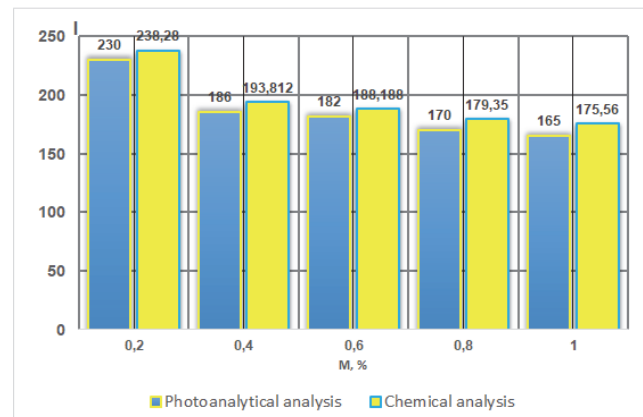


Fig. 5. Results of a control experiment to determine the degree of homogeneity for 5 selected samples of a two-component mixture of salt and coriander powder

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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