HOMOGENEITY CHARACTERIZATION OF FEED POWDER USING HYPERSPECTRAL IMAGING AND CHEMOMETRICS

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Introduction

In many industrial fields such as pharmaceuticals, process engineering and food processing, handling and mixing of powders is an important step where many problems can happen, especially when the objective is to choose the right working parameters to perform a correct homogenization.

Homogeneity is the state of a batch of material where all the constituents are of the same nature; consisting of similar parts, or of elements identically distributed. Homogeneity can be afected by several factors as the physical and chemical characteristics of the particles constituting the powders, the humidity, the distribution of the constituents and the instrument used to perform the homogeneization among others. In the case of the food and feed industries, **the characterization of the homogeneity of mixtures of powders by the use of a rapid and sensitive method is a priority**.

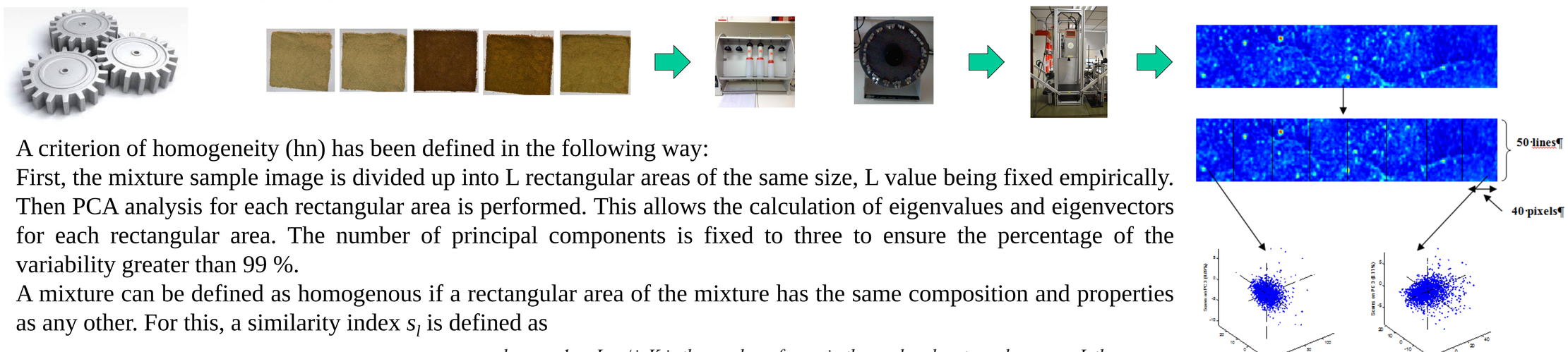
In this work, the aim is to propose a fast and easy methodology to define and characterize the homogeneity of feed mixture powders. This is an important requirement of the European Reference Laboratory for Animal Proteins (EURL-AP) when preparing inter-laboratory studies where homogeneity of samples should be assured. Here to characterize such homogeneity, the proposed procedure includes the use of a hyperspectral NIR imaging system combined with an image treatment PCA-based protocol [1] as an alternative to well-known statistical methods, such as auto-correlation functions or variances.

Material

- A) 8 mixtures of animal meal for poultry consumption with different concentrations of sand . These samples are used to develop and optimize the method proposed by Berthiaux et al. (2005) for the characterization of homogeneity of mixtures of food powders [1].
- B) 5 types of animal meal, which are intended for an interlaboratory study (ring test) organized by the EURL-AP . These real samples will be used to validate the method previously developed.

Procedure

All the samples have been homogenized using two different mixers (FALC F205 rotating disc at a speed of 6 rpm for 30 and 60 minutes for the first set of samples and a Heidolph Reax 20 at a speed of 6 rpm for 30 min for the second set of samples). Then all the samples have been measured using a NIR hyperspectral line scan or push-broom imaging system, combined with a conveyor belt. Each image consists of 500 lines of 320 pixels each acquired at the 1,100-2,400 nm wavelength range.



A criterion of homogeneity (hn) has been defined in the following way:

First, the mixture sample image is divided up into L rectangular areas of the same size, L value being fixed empirically. Then PCA analysis for each rectangular area is performed. This allows the calculation of eigenvalues and eigenvectors for each rectangular area. The number of principal components is fixed to three to ensure the percentage of the variability greater than 99 %.

A mixture can be defined as homogenous if a rectangular area of the mixture has the same composition and properties as any other. For this, a similarity index s_l is defined as

$$s_l = \frac{1}{L.K}\sum_{pj=1}^{L}\sum_{i=1}^{K}\times\sqrt{(PC_1^{pi}-PC_1^{ji})^2+(PC_2^{pi}-PC_2^{ji})^2+(PC_3^{pi}-PC_3^{ji})^2}$$

where p=1,…,L, p≠j, K is the number of rows in the analysed rectangular areas, L the number of areas of the mixture sample and PC_r^{ji} is the rth principal component calculated for ith row in jth rectangular area.

The study of the different values of L allows us to monitor the homogeneity of mixtures. It is done by comparing the variability between the rectangles analyzed by reducing their surfaces. The value of s_l is smaller for similar rectangles, and higher when the differences between them are greater.

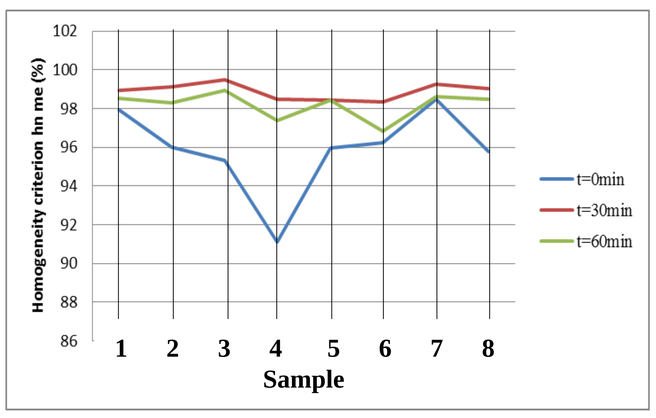
Then the h_n homogeneity criterion is calculated as follows :

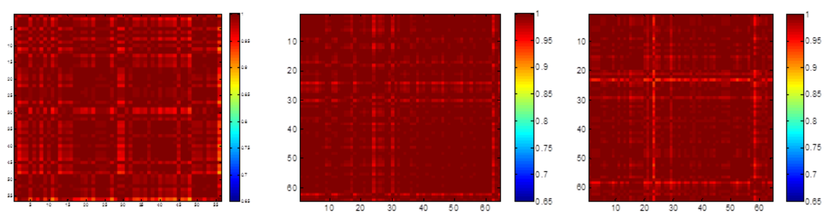
$$h_n\,(\%) = \left(1-\frac{s_{l\,mean}}{s_0}\right)\times 100$$

where s_0 is the sum of the values for a mixture s_l considered completely heterogeneous (or perfectly segregated), it is set to 0.5 in our study according to [1].

Results

Samples A) Summary of mixtures analysed and images showing the degree of variability between rectangles for a certain image

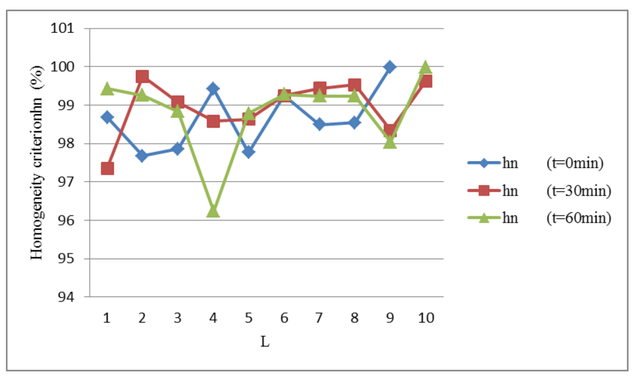




T=0min
h_n=97.94%

T=30min
h_n=98.94%

T=60min
h_n=98.54%



Samples B) Summary of mixtures analysed - the results are expressed in the terms defined by the working group for blend uniformity testing [2].

Mixtures	CV = std / mean (in %) *threshold: 4%*	Amplitude (in %) *threshold: 15%*	Mean (hn) - hn (in %) *threshold: ± 7.5%*	Homogeneity conclusion
Animal meal	0.43	0.96	[-0.64, 0.31]	✓
Animal meal + 0,1% terrestrial meal	0.63	1.66	[-1.02, 0.63]	✓
Fish meal + blood meal	0.43	0.85	[-0.46, 0.38]	✓
Fish meal + 0,1% pig meal	0.77	1.84	[-1.02, 0.83]	✓
Fish meal	1.22	2.85	[-1.13, 1.72]	✓

CV: Coefficient of variation or relative standard deviation (RSD)

Conclusions

This study has allowed us to characterize the homogeneity of binary mixtures of feed ingredients as well as check the influence of homogenization duration. The results obtained indicate that degree of homogeneity of a feed powder mixture is influenced by several factors namely the homogenization time, the physical and chemical characteristics of the particles and the proportions of components.

h_n varies from sample to sample, despite the fact that they have been submitted to the same protocol, the same working conditions and equal treatment of homogenization. It seems that 30 minutes of homogenization is enough to achieve a good homogeneity of the mixture. The homogenization for 60 minutes seems too long as it caused a slight decrease in h_n. This can be explained by the well known 'demixing' or 'segregation' phenomenon. The h_n criterion remains almost constant regardless of the number of rectangles (not shown)

The results obtained for the validation set (samples B) have been compared to other techniques to determine homogeneity as PCR, classical microscopy or NIR microscopy proving the great performance of this method to quantify homogeneity.

Acknowledgments

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Refs

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