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Image analysis: Statistical study of particle size distribution and shape characterization

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A B S T R A C T

Image analysis can serve as a fast and convenient approach for the analysis of particle size and shape. However, there is no consensus as to the minimum number of particles required for such analysis and the statistical methodology to be used in its evaluation. Four methodologies for determination of this minimum number for particle size distribution analysis and two for that of particle shape were tested using particles of guava juice powder and guava juice powder granulated in a fluidized bed. The Chi-Square test proved to be a robust and efficient mean for determination of particle size distribution and particle shape characterization. 550 particles was found to be the minimum number of particles necessary for the determination of the particle size distributions, with 100 particles required for determination of the shape descriptors for this specific material.

1. Introduction

Due to the wide availability of computers and software for images capturing such digital pictures can be utilized for a rapid and convenient analysis of particle size and shape [1]. This standard method is advantageous because there is no sample consumption, and only small quantities of the specimen are required.

When particle size and shape distributions are investigated by image analysis, however, it is important to consider the determination of the minimum number of particles that should be analyzed [2,3]. Currently, there is no consensus as to this minimum. Indeed, values ranging from 100 [4], and 150 [5], up to 14,000 particles [6] have been suggested for determination of average particle size. Generally, this minimum number of particles is considered to be that number which ensures stabilization of the average value and standard deviation of all descriptors [4,5,7]. Turchiuli et al. [5], analyzed 150 particles of zein agglomerated with maltrodextrin to establish constant values of the average particle diameter and standard deviation as well as the shape descriptors. The same stabilization methodology was used by Rondet et al. [7]. A minimum number of particles of 200 of Kaolin agglomerated with water were used to establish D_{50} and shape descriptor. Other authors have simply set the number of particles to be analyzed without considering the variation in standard deviations [1,8,9].

The aim of the present paper is to compare the results obtained by four methodologies for the determination of the minimum number of particles that should be analyzed for the calculation of particle size distribution as well as for the calculation of particle shape distribution. The following section presents the theoretical basis for the statistical methods.

2. Theoretical approach

2.1. Kolmogorov–Smirnov test

The utilization of the statistical test of Kolmogorov–Smirnov was proposed by Vigneau et al. [10], to verify whether two cumulative distributions obtained by image analysis are similar. One-sample Kolmogorov–Smirnov test assesses the degree to which an observed pattern of categorical frequencies differs from the pattern that would be expected. It is a non-parametric method that does not depend on the type of distribution, so it can be used for any kind of distribution. According to this methodology, it is possible to calculate a confidence interval for a distribution (UL and LL) on the basis of the number of particles analyzed (N_{an}):

$$\begin{aligned} UL(v) &= \min\left(1, F(v) + \Delta_{\alpha,N_{an}}\right) \\ LL(v) &= \max\left(0, F(v) - \Delta_{\alpha,N_{an}}\right) \end{aligned} \tag{1}$$

where UL(v) is the upper limit, LL(v) is the lower limit, F(v) is the value of the function for the size v, N_{an} the number of particles

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analyzed and $\Delta_{\alpha,N_{an}}$ is the width of the confidence interval, as defined below:

$$\Delta_{\alpha,N_{an}} = \sqrt{-\frac{2}{2N_{an}}\ln\left(\frac{\alpha}{2}\right)} \quad (2)$$

where α is the significance level of the test.

With this methodology, Vigneau et al. [10] established *a priori* that the cumulative distribution obtained by the analysis of 500 particles must be within the region defined by the upper and lower limits.

2.2. Chi-Square test

The Chi-Square test is one of the most used for the comparison of observed data with what would be expected according to a specific hypothesis [11]. The Chi-Square is calculated by:

$$\chi^2 = \sum_{i=1}^{k}\frac{(O_i-E_i)^2}{E_i} \quad (3)$$

where χ^2 is the test statistic that must be compared with the Chi-Square distribution with k − 1 degrees of freedom; O_i is the observed data value in the size interval i, E_i is the expected value for the size interval i, and k is the number of intervals of the particle size distribution. The decision rule is based on the p-value, which is the smallest level of significance for which there is evidence of a significant difference between the two distributions.

2.3. Paine's methodology

On the basis of a volume-based cumulative distribution, Paine [12] has proposed a methodology for the establishment of the minimum number of particles (N_{crit}) that should be analyzed in the determination of particle size distribution:

$$N_{crit} = \exp[1.71\cdot\exp[0.5r]\cdot(GSD_r-0.83)] \quad (4)$$

where r refers to the base distribution intervals, being equal to 1 for numeric average; 2 for equivalent diameters based on area; and 3 for equivalent diameters based on volume; GSD_r is the geometric standard deviation:

$$GSD_r = \left(\frac{d_{r,84}}{d_{r,16}}\right)^{1/2} \quad (5)$$

where $d_{r,84}$ is the diameter on r base where 84% of the particle distribution is found, and $d_{r,16}$ is the diameter where 16% of the particle distribution is detected.

The methodology proposed by Paine [12] only applies to a log-normal distribution. Both methodologies proposed by Vigneau et al. [10] and Paine [12] are used only with a cumulative distribution function, which is a continuous function of particle size. These tests may not necessarily, however, be suitable for the determination of the number based particle size distribution, widely employed.

2.4. Value stabilization methodology

For several authors [4,5,7], the minimum number of particles to be used for image analysis is considered to be the number necessary to ensure the stabilization of the average value and standard deviation of all descriptors. This methodology has the advantage of being independent of the distribution type of the descriptor.

3. Material and methods

3.1. Material and equipment

In this paper two forms of guava juice powder were used. One was commercially produced by spray-drying at a local factory and the second was obtained by granulation of the spray-dried juice powder in a fluidized bed (Fig. 1).

The equipment operation was similar to that described by Dacanal [13]. The fluidizing air temperature was kept at 80 °C by means of an on/off electrical heater. The fluidizing air flow was monitored by a rotameter, and the air velocity was fixed at 0.25 m/s. The elutriated particles were collected by means of a cyclone.

Water at 27 °C at a flow rate of 0.6 mL/min was employed as liquid binder. The liquid and the air, at a pressure of 1 bar, were pumped into a bi-fluid nozzle (model 1/8JBC-SS13A-SS,Spraying System do Brasil), resulting in atomization of the liquid. The height of the nozzle was fixed at 600 mm above the distribution plate. The amount of guava powder used in each batch was 0.250 kg, and the experiment was carried out in triplicate.

3.2. Image acquisition and editing

Image acquisition was accomplished for three samples (of approximately 10 mg each) placed in Petri dishes with 9.15 cm internal diameter. A Kodak digital camera, EasyShare DX4530, coupled to a Carl Zeiss Jena stereo microscope, model CITOVAL 2, was employed. A 24× magnification was used, which allowed for precise determination for particles measuring up to 7 μm in diameter (9 pixels as minimum sensibility of image acquisition). The pictures were taken in a spiral, from the outside to the inside as the plate was rotated clockwise. The calibration slide was also photograph. Approximately 30 micrographs of 1932 × 2580 pixels each were recorded for each slide, corresponding to an area of 6.3 cm^2. All the samples were analyzed in triplicate.

Each picture was edited using the Serif Photoshop program (Serif Inc., Nottingham, UK), with adjustments of 10% brightness and 80% contrast. The pictures were transferred to the grayscale, and the negative obtained, the threshold was then established, to make them binary.

3.3. Determination of particle size distribution

The edited photos were analyzed by the "Image Tool" program (UTHSCSA, San Antonio, Texas, USA), which calculates the area of each particle in the picture, but ignoring those particles touching the edges. The program was calibrated, in micrometers, in accordance with the picture of the calibration slide (2.1 μm/pixel).

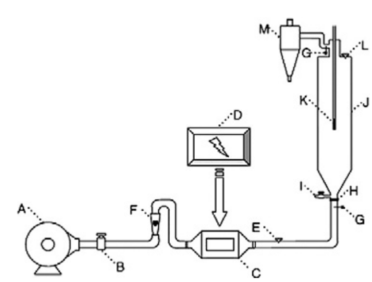

Fig. 1. Scheme of the equipment used in the agglomeration process: A—blower; B—valve; C—heater; D—voltage regulator; E—air deflector; F—rotameter; G—temperature sensor; H—air distributor; I—sample outlet; J—bed chamber; K—nozzle; L—product inlet; M—cyclone [13].