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### Multifractal breakage patterns of thick maltodextrin agglomerates



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#### ABSTRACT

Breakage of food materials affects operations in industry such as storage, transport and quality control, and has an influence on sensorial attributes. However, the geometry of the breakage pattern is a complex and rarely studied subject. Maltodextrin has been used as an ingredient in food related systems. The aim of this work is to characterise the geometry of the breakage patterns in thick maltodextrin agglomerates (MDAs), by digital image analysis (DIA) and multifractal spectra. Results showed that morphology of the breakage pattern was independent of the powder particle size, agglomerate thickness and compaction force. On the other hand, significant differences in pattern's lacunarity were found between the different loads applied whereas different distributions of the irregularity of the breaking pattern measured from the centre to the border of the MDA were obtained. Multifractal spectra reported different generalised fractal dimensions and shapes. Therefore, the breakage profile has a heterogeneous shape with microstructural differences along the breakage line. To the best of our knowledge, this is the first time that multifractal analysis has been used to characterise the breakage pattern geometry in thick MDAs. Size distributions of residual particles (RP) generated during the fracturing process were correlated with the breakage pattern of the breakage pattern and provides valuable information for further research on brittle food products.

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#### 1. Introduction

The phenomenon of breakage is common in operations that involve the handling of brittle foods. The determination of the mechanical fracture characteristics during processing, transportation and storage is important in food processing [1–3].

The process of breakage has been defined as a division of an object by external forces into a number of fragments [4]. The fracture point of a material is considered to occur at the maximum of the stress–strain curve, and it is the result of a plastic deformation after exceeding the elastic deformation point [4]. According to Griffith [5], breakage may be expected if the maximum stress and the maximum extension exceed a critical stress. However, breakage may initiate from an existing crack when the critical stress field around it exceeds a stress intensity factor [6]. Additionally, the mechanisms of fracture require increasing amounts of energy, and it is the geometry or topography of the surfaces of particles contacting each other that determines the fracture mechanism [7]. In a recent study [8], a method was presented to control crack propagation in the low-pressure chemical vapour deposition of silicon nitride on a silicon wafer. The cracking onset can be reached by using a micro-notch structure with an optimal tip angle in order to concentrate the stress on a specific zone [8].

The geometry of the breakage pattern in food products represents a complex and rarely studied subject, which is important in brittle foods. For this purpose, digital image analysis (DIA) has been used to describe the material structure by means of relationships between structures, processing and functionality [9]. Dan et al. [10] represented the spatial patterns of the stress intensities of crunchy and fractured food products by using a distribution of pixels generated by image texture analysis. This study showed the importance between the morphology and the distribution of spatiotemporal stress in the dynamic breakage process of brittle foods. Additionally, Dan et al. [11] analysed the post-failure fracture process of different cucumber cultivars to explore the mechanical properties that determine cultivar-specific crispiness and crunchiness. They found a two-dimensional stress distribution map that showed the brittle points that generated fractures in the interior of the cucumber.

Fractured surfaces in brittle solids are typically irregular and rough. This roughness is characteristic of many natural phenomena, and contrary to their apparent randomness, fractures may present

Abbreviations: DIA, digital image analysis; MDA, maltodextrin agglomerate; RP, residual particle.

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characteristic, predictable patterns. A fractal is a particular case of an irregular object that has specific properties under a number of different scaling processes [12]. DIA and the fractal dimension are widely used to describe the irregularity of materials [13]. However, a single fractal analysis has some limitations. First, fractal dimension alone does not indicate how the space that corresponds to the breakage pattern correlates with the entire image. Second, fractal dimension describes the breakage pattern with only one measuring set [14]. It is possible to assume that the breakage pattern is a complex set formed from different subsets or regions that have different scaling properties [15]. Multifractal analysis may be used to describe a complex breakage pattern because these objects are an extension of fractals and differ from simple fractals by two significant features: 1) they are based on measures of sets and not on the sets themselves; and 2) a set of data analysed by means of multifractal techniques offers an entire spectrum of fractal dimensions [15]. A multifractal object is always invariant by translation, and depending on the observation scale, it is possible to distinguish details that cannot be detected in the whole object [16]. Multifractals involve the decomposition of self-similar measures into entangled fractal sets, which may be characterised by their fractal dimension and singularity strength or the probability of having a certain fractal dimension in a local section of the object [14,17]. Multifractal characterisation does not involve a single dimension but instead a sequence of generalised fractal dimensions. A combination of all fractal sets produces a multifractal spectrum that characterises the variability and heterogeneity of the studied variables. The advantage of the multifractal approach is that multifractal parameters can be independent of the size of the studied objects [17]. In food research, multifractal analysis has been successfully applied as a descriptor of food structure [18], for example, the evolution of the epidermis netting of muskmelon [14], the spatial pattern of fatty infiltration in pork meat [19] and the pore size distribution of apple tissue [20].

The objectives of the present study are to characterise the geometry of the breakage patterns in maltodextrin agglomerates (MDAs) by DIA and to calculate their multifractal spectrum to determine possible relationships between the breakage patterns and residual particles (RP) or debris obtained after the fracture.

#### 2. Experimental section

#### 2.1. Preparation of MDAs

Thick MDAs [21] in the form of round tablets (13 mm diameter) were prepared using a Carver hydraulic laboratory press (model C S/N 32000-224, USA) and a 13 ton die (WQ09D PerkinElmer, UK) at three compaction forces as reported by Meraz-Torres et al. (Table 1) [21]. Maltodextrin powder (200 g) was spread on the top of three stainless steel sieves (US standard sieve series, W.S. Tyler Co., USA) with mesh numbers 120, 170 and 270, to obtain samples having a number of defined particle sizes, namely 125, 89, and 53 µm pore sizes [21]. Maltodextrin powder and MDAs with 7% (wb) moisture content were used in all experiments.

#### 2.2. Experimental design

A Box–Behnken [22,23] response surface design was used to analyse the effect of three factors of the MDAs on some properties of the fracture

 Table 1

 Factors and levels for experimental design using Box–Behnken method.

Variables	-1	0	+1
Powder particle size (µm)	53	89	125
Thickness of MDA (mm)	1	2	3
Compaction force (ton)	0.5	1.0	1.5

pattern. The design had a total of 17 experimental runs. The levels and the factors are shown in Table 1. Three response variables were evaluated through this method: lacunarity, length of the fracture, and mass loss. Analyses of the RP sizes and multifractal spectrum distribution characteristics (i.e., kurtosis and skewness) were carried out using MINITAB 16 software (Minitab Inc., USA).

#### 2.3. Breakage test for MDAs

The initial weight of the MDAs was registered before the breakage test using an analytical balance (Ohaus Analytical Plus, USA). MDAs were subjected to bending–breakage using a compression and tensile testing machine (Instron 5565, USA) fitted with 50 and 500 N load cells, which were coupled to a probe (3.5 mm diameter, 0.096 cm<sup>2</sup> contact area, 15 cm height) operating at a 50 mm/min vertical descending velocity. When the breakage test finished, the final weight of the fractured MDAs was evaluated and RP from the breakage test were collected for further analysis.

#### 2.4. Digital image analysis (DIA)

Images of the broken MDAs were captured through a Stereo Microscope (Nikon SMZ 1500, Japan) with a scale of 0.02 mm/pixel and a resolution of  $1600 \times 1200$  pixels. Top illumination was from a daylight lamp (Model 13 Plus, 3" Deluxe Daylight 5100 K, Stocker & Yale, USA). In the case of the images of the RP from the breakage, the scale was 0.02 mm/pixel with a resolution of  $640 \times 480$  pixels, and the same illumination system described above was used.

Images of the fractured MDAs were analysed with the software ImageJ v.1.45 (National Institutes of Health, Bethesda, USA) to determine the fracture length and lacunarity and to perform a multifractal analysis, using the Frac-Lac 2.5 tool of the ImageJ software. The size distribution of the RP from the breakage was analysed with Sigma Scan Pro software (V5.0, SPSS, USA) with the size of the RP measured as the Feret diameter.

Image processing consisted of converting the image to an 8 bit format (i.e., grey scale image). A  $3.5 \times 13.63 \text{ mm}^2$  section was cropped from the original image including the breakage pattern of one fragment of the broken MDA. Then, the threshold was adjusted to 95/255 RGB to obtain a binary image [21], and finally, the contour of the binary image was selected and manually cropped.

#### 3. Multifractal analysis theory

Multifractal analysis was applied to the breakage patterns through the box-counting method. Once the information about the number of pixels of a pattern's binary image covered by individual boxes was obtained, it was possible to determine the probability of finding those pixels in a given gliding box as described by [24]:

$$P_i(L) \sim L^{\alpha}_{i}, \tag{1}$$

$$P_i(r) = L_i(r)/L_T(r), \tag{2}$$

where  $L_i$  is the number of pixels in one box for the given scale,  $L_T$  is the total number of pixels in all boxes for the chosen scale, and r is the box size.

The singularity ( $\alpha_i$ ) is defined as rapid changes in the mass probability values for a change in each local position of the system. Similar  $\alpha_i$  values can be found at different positions *i* [20].

Multifractals may be expressed by a generalised fractal dimension  $(D_q)$ , where q is a parameter for exploring different regions of the singular measurement and is a number within  $-\infty$  to  $+\infty$  [15]. For q > 1,  $D_q$  represents the more singular regions; for q < 1,  $D_q$  stresses the regions with the lowest singularity; and for q = 1,  $D_q$  represents the information

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