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MEASURING THE SIZE OF PORES BY THE SEGMENTATION OF IMAGES FROM SCANNING ELECTRON MICROSCOPY

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Abstract: Segmentation is an image processing method used for partitioning an image into multiple sets of pixels, which are defined as its "super-pixels". Here, we are proposing a method based on segmentation, for determining the super-pixels corresponding to the pores resulting from freeze-drying of a pharmaceutical solution. The size of these pores, evidenced by the Scanning Electron Microscopy (SEM), are estimated through the areas of the super-pixels of the segmented image, and can then be used to estimate the resistance to mass transfer and hence optimise the cycle.

Keywords: freeze-drying, pore sizing, image segmentation, image processing, pharmaceutical solutions

Introduction

The freeze-drying process is currently used as a common industrial processing and preservation technique, applied to a wide variety of products, the large part of them being food, bio-products and pharmaceuticals. Consequently, freeze-drying is generating technological processes which have a high impact on industry and society.

Because of this fundamental role, the researches on freeze-drying process are attracting the efforts of several scientific disciplines, rendering it the subject of a highly interdisciplinary area of studies. Let us consider, for instance, the scaffolding of a dried solution created by such processes. This structure is requiring models which can help us to understand the reasons of its formation as a consequence of concentrations and heat fluxes applied to the sample. However, models are based on observations from microscopy and tomography and were demonstrated to be useful for the determination of the resistance to vapor flow and hence for cycle optimisation. For this reason, the modelling method can receive a large benefit from the processing of the data coming from such analyses, and therefore from the discipline of the image processing.

Here an example is proposed. We will show that we can use one of the fundamental methods of such a discipline, the image segmentation, to obtain data from SEM imaging, concerning the size of the pores resulting from the freeze-drying process.

Materials and Methods

To illustrate the approach, let us use a lyophilized sample produced from an aqueous solution of sucrose, having 5% w/w as solid content. The solution was freeze-dried as discussed in [1]. In the Fig.1 (left), we can see a SEM image of the sample, which is showing the presence of the typical pores created by the process. To estimate the size of pores, the approach we follow is that of segmenting the image. To obtain the segmentation, the image of the left panel is pre-processed to enhance brightness and contrast, and then smoothed by a Gaussian filter. After, the image is mapped into a binary map (Fig. 1, middle), which is segmented through a thresholding method [2]. The result of the segmentation is the partitioning of the SEM image into sets of super-pixels, which are represented in the Fig.1 (right), by the colored domains. Each super-pixel is characterized by a label, by means of which we can easily evaluate the area (in pixels) covered by the domain.

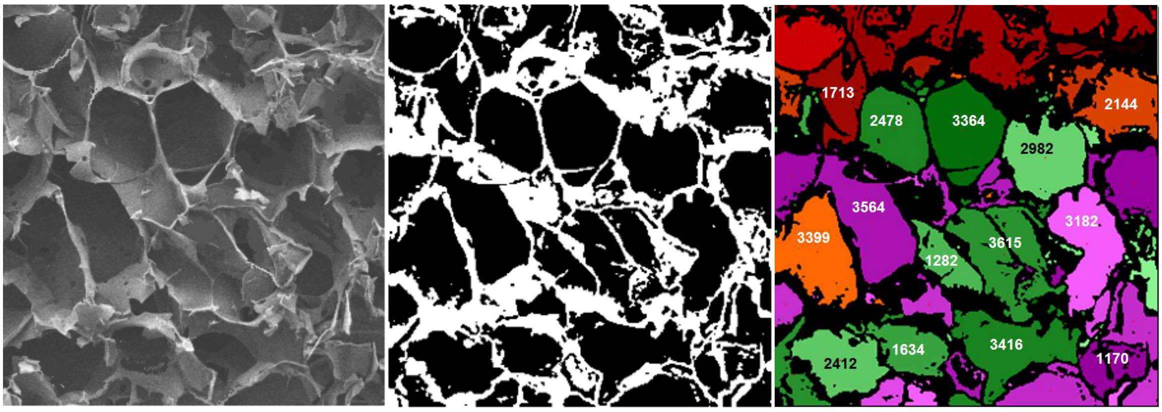

Fig. 1. A SEM image (left) is pre-processed to have the binary image (middle) used for segmentation. The segmented image (right) shows super-pixels (here rendered by different colour tones) and their area (in pixels). The image is 300 x 315 pixels (540 x 568 μm).

Results and discussion

The area of the super-pixel is a measure of the cross-section of the considered pore. Then, the segmentation gives us a set of data reporting the areas of the several cross-sections of the pores given by the image. As an example, Fig. 1 (right-graph) gives some values (the super-pixels crossed by the image frame are not considered in the calculation). The cross-section of pores is 2600 ± 700 (in pixels), that is, (8000 ± 2150) μm^2. It corresponds to a radius of about 50 μm.

Conclusions

As we have illustrated, the study of the pores of lyophilized samples can be improved by methods of image processing. Here, for instance, we have determined the segmentation of the super-pixels which are containing the pores, in order to estimate their size. Let us add that the approach we have used, the segmentation, is the first step for any further 3D reconstruction of the sample from images given by tomography. Actually, a 3D analysis is requiring the registration of such images, an operation usually performed through the alignment of super-pixels. Knowledge of the average pore size and its distribution is essential for the determination of the resistance to mass transfer and hence for the use of the most recent model-based tools for the development and scale-up of freeze-drying cycles.

References

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