Particle agglomeration analysis using PATVIS APA and deep learning

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INTRODUCTION

Pharmaceutical, chemical, cosmetic, food, and other industries utilize various processes to modify particle properties, where agglomeration of particles occurs either as an accurately controlled process or as an undesired phenomenon. In both cases, real-time estimation of the particle agglomeration provides means for process monitoring and process control.

In pellet coating, agglomeration negatively affects the process yield and the coat integrity. Variations in the properties of materials and operating conditions as well as sporadic events are major causes for unwanted agglomeration. Traditionally, the agglomerate fraction is assessed at the end of the coating process by sieve analysis, which is an approach with obvious drawbacks. Currently, the most promising in-line agglomerate fraction analysis methods applied to pellet coating processes are based on optical measurements [1].

In this paper, we present a novel image analysis approach for automatic, non-invasive recognition of agglomerated particles and estimation of the agglomerate fraction. Image sequences of mixtures of individual pellets and agglomerates with predefined ratio (reference agglomerate fraction) were acquired and the detected particles were classified. Furthermore, the agglomerate fraction of each mixture was assessed and compared to the reference values.

MATERIALS AND METHODS

Agglomerate fractions

Film-coated microcrystalline cellulose pellets with a size distribution 700 µm–1000 µm and agglomerates of such pellets were obtained. 100 g agglomerate fraction mixtures of 0.0, 0.5, 1.0, 2.5, 5.0, 10.0, 20.0, and 100.0 % w/w were prepared by weighing using an analytical balance.

Imaging setup

Images of pellets were acquired in a simulator mimicking pellet movement during the coating process (i.e., 2D fluid-bed system) using an in-line visual inspection system PATVIS APA (Sensum, Slovenia) (Figure 1). The mounting position enabled imaging of the pellets in free fall. Each agglomerate fraction mixture was imaged for 10 min. The system allowed an image acquisition rate of 100 images per second, which resulted in an average sample size of 300 000 particles.

Figure 1: Image acquisition of pellets in a 2D fluid-bed system (right) with visual inspection system PATVIS APA (left).

Image analysis

The image analysis method is comprised of two major stages: first, particle regions are detected by clustering-based image segmentation [2]. Second, the particle regions are classified as individual particles or agglomerates using a deep-learning model, namely a convolutional neural network (CNN) (Figure 2). A CNN is a machine-learning model that can automatically learn to differentiate between individual particles, groups of individual particles and agglomerates (Figure 4) by exposing it to images of known (labeled) particles, i.e. model training.

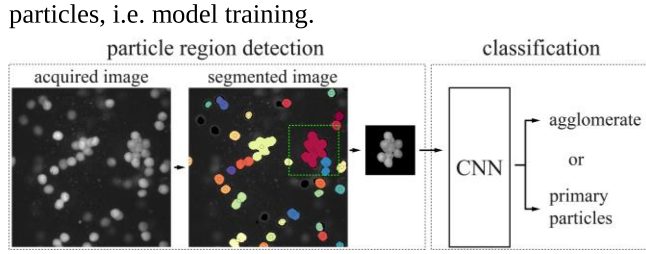

Figure 2: Overview of the two-stage image analysis method.

After classification, the agglomerate fraction AF can be expressed as the volumetric ratio between the agglomerated particles and all analyzed particles in a certain time interval. If there are N agglomerates and M individual particles, the AF can then be expressed as (Equation 1):

$$\mathrm{AF} = \frac{\sum_{i=1}^{N} \mathrm{ESV}_i^{\mathrm{agg}}}{\sum_{i=1}^{N} \mathrm{ESV}_i^{\mathrm{agg}} + \sum_{j=1}^{M} \mathrm{ESV}_j^{\mathrm{ipt}}}, \quad (1)$$

where $\mathrm{ESV}^{\mathrm{agg}}$ and $\mathrm{ESV}^{\mathrm{ipt}}$ are the equivalent spherical volumes of agglomerates and individual particles, respectively.

Figure 4: Sample images of an individual pellet (left), a group of individual pellets (middle) and an agglomerate (right).

RESULTS AND DISCUSSION

Figure 4 shows the ROC curves for classification based on estimated particle sizes by a previously proposed method [3] and based on the novel CNN on a test set of labeled images of individual pellets and agglomerates. The results show a significantly better performance of the CNN classifier (96.7 % classification accuracy at threshold 0.5 for the CNN classifier versus 85.1 % classification accuracy at threshold 1500 µm for the size-based method).

Table 1 shows the reference and measured agglomerate fractions, measured on agglomerate fraction mixtures. The results indicate that the size-based method is less accurate at detecting low agglomerate fractions because of more frequent false-positive agglomerate detections (Figure 4). In contrast, the proposed CNN method estimates low fractions of agglomerates more accurately due to the improved recognition of agglomerates (RMSE of 2.25 % and 4.73 % for the CNN and size-based classifier, respectively).

$\mathrm{AF}_{\mathrm{ref}}$ (%)	$\mathrm{AF}_{\mathrm{size}}$ (%)	$\mathrm{AF}_{\mathrm{CNN}}$ (%)
0.0	1.45	0.40
0.5	1.67	1.05
1.0	2.10	1.33
2.5	3.59	3.06
5.0	6.78	5.78
10.0	11.09	10.46
20.0	22.77	22.15
100.0	87.31	94.15
RMSE	4.73	2.25

Table 1: Results on mixtures with predefined agglomerate fractions ($\mathrm{AF}_{\mathrm{ref}}$): $\mathrm{AF}_{\mathrm{size}}$ – agglomerates recognized based on their size, $\mathrm{AF}_{\mathrm{CNN}}$ – agglomerates recognized with CNN.

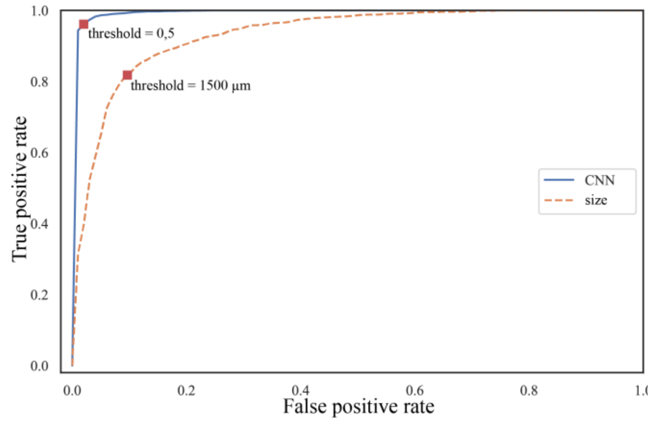

Figure 3: ROC curve for classification based on estimated particle sizes and based on the novel CNN approach.

CONCLUSION

An automated image analysis method was developed for in-line recognition of agglomerates and estimation of the agglomerate fraction of pellets during the coating process. The obtained results compared to the previously proposed method [3] indicated a considerably improved recognition of agglomerates, which consequently improves the accuracy of the agglomerate fraction estimation.

Capturing images in controlled conditions separately for individual pellets and agglomerates facilitates the acquisition of learning images and is a key step for the practical feasibility of using machine learning to identify agglomerates in actual pellet coating processes.

Eventually, the timely information about the agglomerate fraction can be used for process control by manual or automatic optimization of process parameters to retain the agglomerate fraction in an acceptable range.

Although we tested the proposed method only on pharmaceutical pellets, it can be used for agglomerate fraction estimation of particles in other processes as well.

REFERENCES

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