

MICROSTRUCTURAL ANALYSIS OF MULTICOMPONENT HETERO-AGGLOMERATES FORMED IN A TURBULENT PIPE FLOW

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ABSTRACT

Solid-state batteries (SSB) differ fundamentally from conventional lithium-ion batteries in that they use solid electrolytes instead of liquid components. In order to efficiently combine the different solids and thereby achieve high electrochemical performance, hetero-agglomeration represents a suitable approach. In particular, the homogeneity and the number of hetero-contacts between different particles have a significant influence on the resulting electrochemical properties, which underlines the central importance of efficient mixing and agglomeration processes.

A jet-based mixing process in a turbulent pipe flow is used to produce cathode materials for SSB with the aim of improving the homogeneity and the electrochemical properties of the battery materials. The starting materials, consisting of the active material lithium iron phosphate (LFP), conductive additive carbon black (CB), and magnesium oxide (MgO), which is used here as a model material for the solid electrolyte lithium indium chloride (LIC), are first broken up and dispersed in the gas phase. The different particles then collide in a turbulent pipe flow, where interactions between the primary particles result in the formation of hetero-agglomerates.

This work focuses on the structural analysis of the hetero-agglomerates produced in the mixing process. The morphological characterization of the particle microstructure was performed using scanning electron microscope (SEM) images, which were quantitatively evaluated to determine the particle size distribution (PSD). Chemical characterization was performed using EDX, whereby the elemental composition was determined statistically from several spectra based on mean values, coefficients of variation, and frequencies of occurrence. In addition, the local degree of mixing was analyzed using EDX mapping by quantifying lateral hetero-contacts between the phases via binary masks. In an ongoing parameter study, various process parameters are being varied in order to investigate their influence on mixing and agglomeration.

Quantitative evaluation of the SEM images shows a shift in PSD along the pipe flow, indicating increasing agglomeration. At the same time, EDX analyses confirm progressive mixing of the particles, which is reflected both in homogenized elemental proportions and in increased formation of lateral hetero-contacts between the phases.

KEYWORDS

Mixing, Agglomeration, Hetero-agglomerates, SEM-EDX, Solid state battery

1 Introduction

The targeted mixing of particles is of central importance for their functional properties, especially in solid-state battery technology (SSB), as the electrochemical performance here depends significantly on homogeneous distribution and intensive interface contact between the starting materials. Unlike conventional lithium-ion batteries with liquid electrolytes, which wet pores and partially form interfaces independently, ion transport in SSB occurs exclusively via solid phases, meaning that insufficient mixing directly leads to increased interface resistance and limited performance [1,2]. A fine spatial mixture of different particle types therefore promotes efficient transport processes, improves ionic and electronic conduction paths, and can thus significantly increase electrochemical performance. In addition to the material composition, the microstructure of the resulting particle collectives is therefore also a decisive factor that significantly influences the overall performance of the material system.

Hetero-agglomeration, the process of combining different particulate materials in a targeted manner, represents a promising approach to improving structural and electrochemical properties. These properties are significantly influenced both by the composition of the mixture, such as the number of components, their concentrations, and the total particle quantity, as well as by secondary process parameters such as agglomeration or coating conditions. The process of hetero-agglomeration is shown schematically in Fig. 1. Combining three different particle components creates a new mixture. The degree of mixing describes the spatial distribution of the starting materials within the mixture and serves as a measure of its homogeneity [3,4]. A high degree of mixing leads to an increased number of contacts between the different particles, known as hetero-contacts. These can give the resulting mixture novel functional properties, such as a targeted improvement in electrochemical performance in the case of SSB.

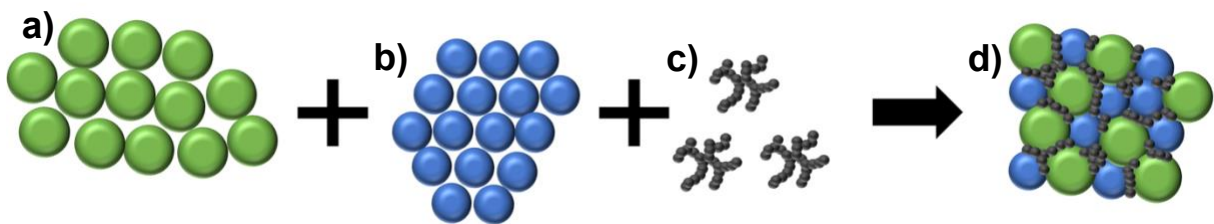


Fig. 1. LFP a) and MgO primary particles b), and CB Aggregates c), forming a hetero-agglomerate d).

A novel jet-based direct mixing process was developed for the production of these composites, in which mixing takes place in the gas phase within a turbulent pipe flow. The individual particle systems are first dispersed by two brush dispersers and then brought together as particle laden aerosol jets in a turbulent mixing zone, where

particle interactions occur and the formation of hetero-agglomerates is specifically promoted. The focus is on a cathode material for SSB consisting of lithium iron phosphate (LFP), carbon black (CB), and lithium indium chloride (LIC), whereby a model material for LIC in the form of magnesium oxide (MgO) is used in this work.

The aim of this work is to develop and present a method for the structural characterization of the mixing state of hetero-agglomerates formed in the gas phase during the jet-based process. To this end, structural and elemental analyses using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) are employed to reveal fundamental relationships between process control, agglomeration behavior, spatial mixing, and the formation of hetero-contacts. The developed analytical concept serves as the basis for future parameter studies aimed at the targeted optimization of the process and the resulting material properties.

2 Materials and methods

2.1 Materials

Three components were used to produce the cathode material. Fig. 2 shows SEM images of these components at two different magnifications (10kx and 50kx). LFP acts as the active material in the cathode of an SSB and enables the reversible storage and release of lithium ions during the charging and discharging processes. It is characterized by high thermal stability, good cycle stability, and low cost [5]. Submicron LFP with a mass-based average primary particle diameter of 530 nm was used to ensure a sufficiently large contact area for ion transfer. CB was used as a conductive additive to increase the cathode's electrical conductivity. With a primary particle size of < 50 nm, it offers a high specific surface area, improves electrical contact between the LFP particles, and thus helps reduce internal resistance [6]. In its initial state, CB is already predominantly present as aggregates in the range of a few hundred nanometers. The third material used was submicron magnesium oxide (MgO) with an average primary particle diameter lower than 90 nm. It served as a model material for the actual solid electrolyte lithium indium chloride (Li_3InCl_6 , LIC), as the latter can only be processed under controlled dry room conditions due to its pronounced sensitivity to moisture. The particle size distribution (PSD) of MgO is similar to that of suitable lithium halide solid electrolytes (LIC), so that the results obtained can be transferred to the final battery system.

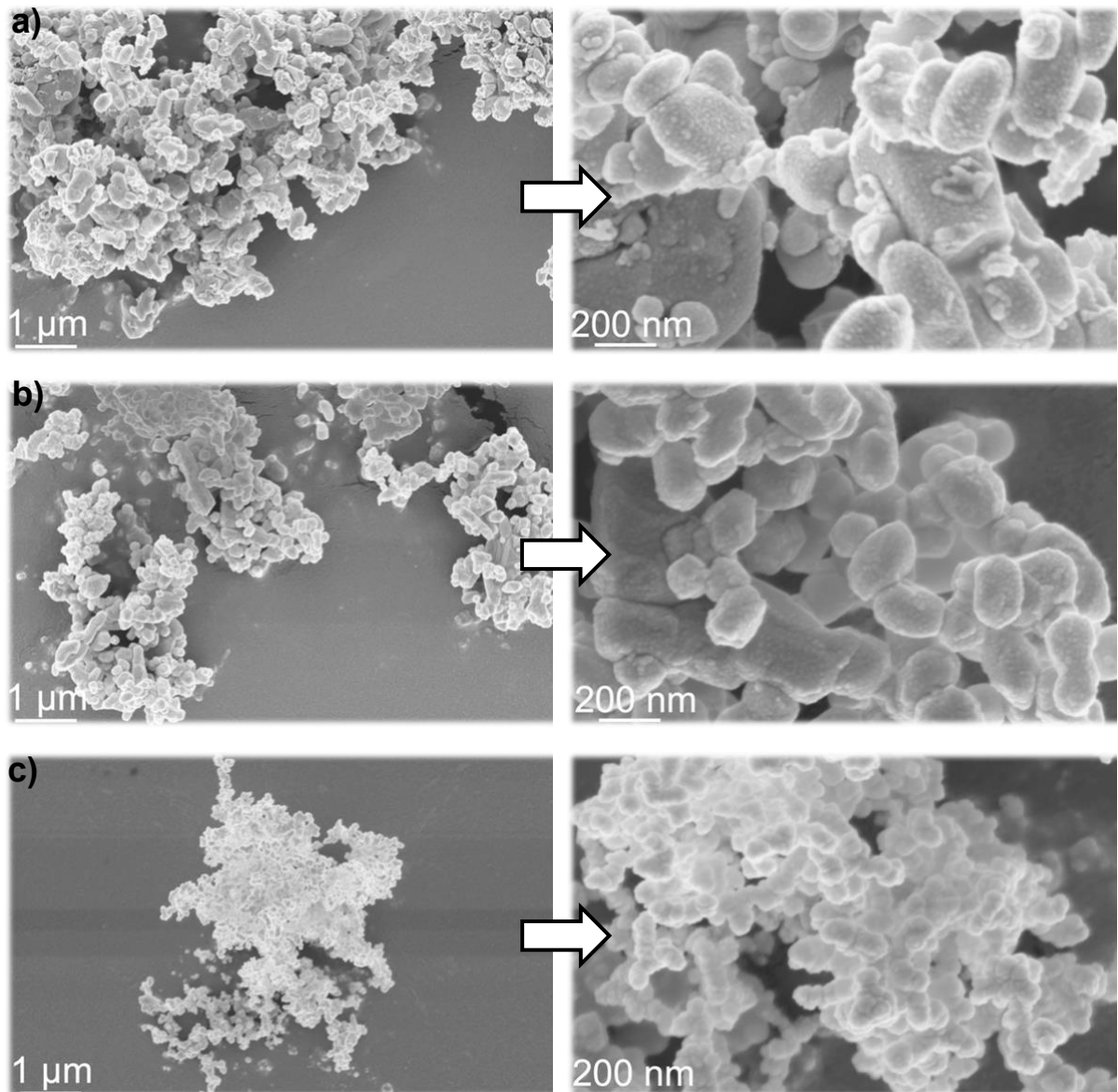


Fig. 2. Starting materials LFP a), MgO b) and CB c) with magnification of 10 kx and 50 kx.

2.2 Experimental methods

This paper investigates a jet-based mixing process in turbulent pipe flow (see schematic representation in Fig. 3 a)). In the proposed process the starting materials are first deagglomerated to the primary particle level using brush dispersers (RBG 1000 ISD from Palas, see Fig. 3 b)) and dispersed into a turbulent mixing zone. There, collisions between different particle types result in interactions that lead to the formation of hetero-agglomerates due to adhesive forces. The resulting particle collective then flows through a long turbulent pipe section, where progressive mixing and agglomeration into larger structures takes place. This process was investigated both experimentally (see [7,8]) and numerically (see [9,10]) and optimized specifically on this basis. In particular, geometric parameters and flow conditions were analyzed to improve particle interactions and mixing. To optimize particle flow early on, the

geometry was designed so that the aerosol jets converge at a defined angle rather than colliding head-on (see Fig. 3 a)).

Particles can be sampled at defined positions (P1...P6) along the mixing process using a specially developed pneumatic sampling unit for ex-situ SEM (see Fig. 3 c)). The unit pneumatically opens a flap in the pipe wall, whereupon an SEM sample holder (SEM stub) is briefly inserted into the aerosol stream and then retracted again. The stubs are aligned so that the aerosol stream hits their surfaces perpendicularly. This enables samples to be taken from the running, highly concentrated particle flow with precisely defined particle loading times. Short loading times (approx. 60 ms in this case) prevent the stubs from becoming overloaded, thus optimizing SEM analysis. Before examination, the SEM stubs are sputter-coated with a thin layer of gold (approx. 15 nm) to improve conductivity and minimize charging effects.

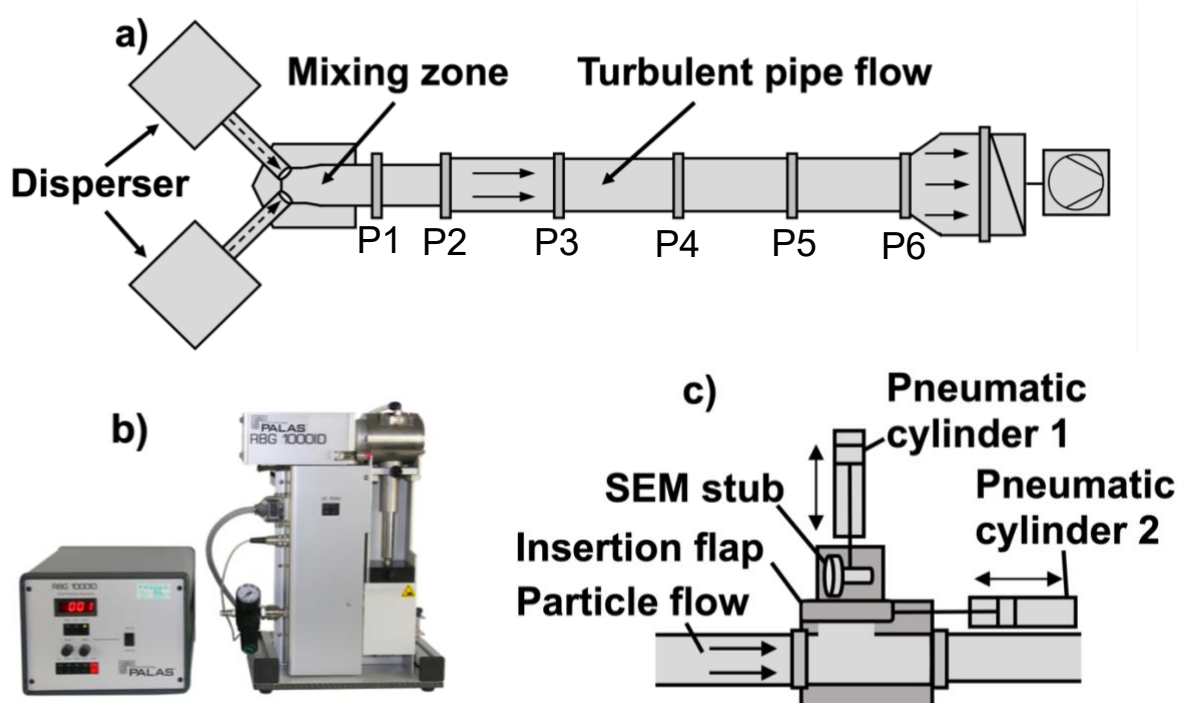


Fig. 3. Experimental setup of jet-based direct mixing process a), brush disperser used b) and pneumatic sampling unit c).

The production of high-performance, long-lasting SSB cathodes requires a precisely balanced mixing ratio of the components. In this study, a cathode composite with a mixing ratio of 58 wt% active material (LFP), 38 wt% solid electrolyte (MgO), and 4 wt% conductive additive (CB) was targeted (see [11]). The process parameters shown in Table 1 were determined, on the one hand, with regard to the target material ratio and, on the other hand, based on the findings of previous experimental studies. These studies identified an optimal compromise between effective deagglomeration of the starting materials and a sufficiently high mass concentration, which promotes the formation of hetero-agglomerates (see [8]).

Table 1. Process parameters used in this study.

LFP - CB	Mass flow / g/h		Volume flow / m ³ /h	Mass concentration / g/m ³
	MgO	Total		
164	100	264	7	37.7

2.3 Characterization methods

2.3.1 Scanning electron microscopy (SEM)

The morphological characterization of the samples was performed using SEM. To examine the PSD, overview images were taken at a magnification of 500x. This magnification was specifically chosen to ensure that agglomerates and larger particle clusters were completely within the image field, thereby enabling a representative and statistically reliable recording of the agglomerate structure. At the same time, this magnification still offers sufficient resolution to detect individual primary particles down to the submicron size range. Although these particles are not displayed with maximum sharpness at 500x, they can still be identified and taken into account in the image analysis.

Five images were taken for each sample (at the center, top left and right, and bottom left and right position of the SEM stub). The positions were saved in the SEM and specifically selected for each analysis to ensure identical measurement conditions. The images were taken at an acceleration voltage of 5 kV and a beam current of 200 pA. The comparatively low acceleration voltage was chosen to reduce charging effects and at the same time improve the resolution near the surface. The working distance was set to a constant 5 mm to ensure reproducible imaging conditions.

2.3.2 Energy dispersive X-ray spectroscopy (EDX)

Chemical characterization was performed using EDX in combination with SEM. First, an agglomerate to be analyzed was selected from the same image section as used for the PSD analysis (500x magnification; see section 2.3.1). This agglomerate was then examined at a higher magnification (2500x) using EDX mapping. This procedure ensures reproducible agglomerate selection and, at the same time, sufficiently high spatial resolution for analyzing local mixture structures and phase contacts.

The EDX measurements were performed at an acceleration voltage of 10 kV. Compared to the lower voltages (5 kV) used for morphological SEM analysis, the higher acceleration voltage enables more efficient excitation of characteristic X-ray lines, resulting in higher intensity and improved signal-to-noise ratio, especially for elements with higher excitation energies. At the same time, the voltage was deliberately chosen to be moderate in order to limit the electron interaction volume and thus ensure sufficient spatial resolution of the elemental analysis, so that phase contacts between neighboring particles can be reliably resolved. A beam current of 2 nA was used to achieve a sufficiently high X-ray intensity for recording element distribution maps. For mapping, a constant dwell time per pixel of 60 μ s at 4 frames

was used to ensure homogeneous signal acquisition and reproducible intensity distributions.

Data acquisition and evaluation were performed using AZtec software from Oxford Instruments. From the EDX maps, TruMaps were generated and used as input data for the subsequent analyses. TruMaps are spectrally unfolded, quantitatively corrected element distribution maps in which overlaps of characteristic lines and background contributions are separated mathematically. This results in improved phase separation of the elements and forms the basis for calculating local compositions in the context of Lacey mixture index analysis, as well as for generating binary phase masks for hetero-contact analysis (see section 2.4.2).

2.4 *Fiji-based image analysis*

2.4.1 *PSD analysis*

This work focuses on the quantitative structural analysis of the hetero-agglomerates formed in the gas phase mixing process. The SEM images were quantitatively evaluated in Fiji (ImageJ) to determine the PSD and characteristic agglomerate sizes. For this purpose, the images were scaled, segmented, and analyzed on a particle basis. The particles were segmented using automatic thresholding (Otsu method). As part of the particle-based analysis, the projection area A_p was determined for each identified object. To describe a characteristic particle size, the area-equivalent diameter d_A was calculated as follows:

$$d_A = \sqrt{\frac{4 \cdot A_p}{\pi}}. \quad (1)$$

This parameter was evaluated in terms of both number and mass. The number-based PSD is derived directly from the frequency distribution of the calculated diameters. The mass-based PSD was calculated from the number-based distribution using moment transformation. For this purpose, assuming spherical particle geometry and constant density, a conversion was performed using the corresponding distribution moments.

2.4.2 *Mixing quality analysis*

To quantitatively assess the mixing state, an image analysis of EDX mappings was performed. This analysis was based on element-resolved intensity distributions (net count rate), which were exported as TIFF files and further processed in Fiji. The mixing state was quantified using a cell-based Lacey mixing index (see [12]) derived from the EDX intensity maps.

Each element map was divided into a regular grid of N non-overlapping square cells of size $s \times s$ pixels using a Fiji macro. For each element k , there is an intensity distribution I_k that is approximately proportional to the local concentration of that element. Here, $I_{k,p}$ describes the X-ray intensity of the element measured at pixel p .

For each cell i the average intensity

$$I_{k,i} = \frac{1}{n^{\text{pix}}} \sum_{p \in i} I_{k,p} \quad (2)$$

was determined, where $n^{\text{pix}} = s^2$ is the number of pixels per cell. The cell size s was kept constant for all samples compared with each other in order to ensure direct comparability of the calculated mixture indices. The local composition per cell is determined based on the averaged intensities of the investigated elements (here: P and Mg):

$$c_i = \frac{I_{\text{P},i}}{I_{\text{P},i} + I_{\text{Mg},i}}. \quad (3)$$

From the local composition per cell c_i the global average composition

$$\bar{c} = \frac{1}{N} \sum_{i=1}^N c_i \quad (4)$$

and the measured variance of the cell compositions

$$\sigma^2 = \frac{1}{N-1} \sum_{i=1}^N (c_i - \bar{c})^2 \quad (5)$$

were first determined.

The reference variance for the completely segregated state is:

$$\sigma_0^2 = \bar{c} (1 - \bar{c}). \quad (6)$$

This corresponds to the theoretical maximum variance for a given global composition $\bar{c}$, i.e., the case in which each cell contains either only the reference component ($c_i = 1$) or none of it ($c_i = 0$) [12].

The variance of the random mixture is given by

$$\sigma_r^2 = \frac{\bar{c} (1 - \bar{c})}{\bar{n}}, \quad (7)$$

where $\bar{n}$ is the average number of statistically independent samples per cell. Since each pixel in this work represents an independent EDX intensity measurement $\bar{n} = n^{\text{pix}} = s^2$ is set. Since the cells typically contain $n^{\text{pix}} \gg 1$ pixels, the following applies:

$$\sigma_r^2 = \frac{\bar{c} (1 - \bar{c})}{n^{\text{pix}}} \approx 0. \quad (8)$$

The denominator of the complete Lacey formula is thus simplified to:

$$\sigma_0^2 - \sigma_r^2 \approx \sigma_0^2. \quad (9)$$

The cell-based Lacey mixture index was therefore calculated in the following simplified form:

$$M = 1 - \frac{\sigma^2}{\sigma_0^2} = 1 - \frac{\sigma^2}{\bar{c}(1 - \bar{c})}. \quad (10)$$

The Lacey index can range from 0 to 1. A value of 0 indicates a completely segregated state in which the cell compositions deviate maximally from the mean value $\bar{c}$. A value of 1, on the other hand, represents an ideally homogeneous mixed state, where all cell compositions align with the mean value $\bar{c}$, resulting in the disappearance of variance ($\sigma^2 = 0$). A higher value of M corresponds to a higher degree of mixing on the microscale defined by the cell size s . Since M is scale-dependent, all samples were evaluated with identical cell size s to ensure direct comparability of the mixing indices. For the analysis in this paper, a cell size of $s = 128 \times 128$ pixels ($n^{\text{pix}} = 16384$) was selected, corresponding to an edge length of $2.912 \mu\text{m}$. For a Map with an image size of 2048×1536 pixels, this results in a total number of $N = 192$ cells.

2.4.3 Hetero-contact analysis

The Lacey index quantifies the homogeneity of powder mixtures and allows for a comparison of the mixing quality under different process conditions. However, it captures only the spatial phase distribution, not the actual contacts at phase boundaries.

Therefore, a hetero-phase contact analysis was additionally performed to determine the proportion of LFP particle boundaries in direct contact with MgO. The intensity of these contacts was quantitatively measured using EDX mapping. This was based on element-selective distribution maps (EDX TruMaps) recorded using Aztec software from Oxford Instruments. Further processing was carried out using Fiji.

To identify phase boundaries, binary phase masks were generated from the element-selective intensity maps. A consistent thresholding method was used to uniquely assign a phase to each pixel. Since EDX measurements have a finite interaction volume, this was accounted for by applying a morphological dilation to the phase masks. To this end, one of the two phase masks (here: P) was expanded by n pixels. The dilation serves to represent the spatial resolution limit of the EDX signal by connecting adjacent contact pixels. The number of three dilation steps was determined based on a convergence analysis. After three iterations, the contact mask exhibited sufficient stability in terms of area and spatial extent, such that further dilations would not have provided any additional methodological benefit but would have increased the risk of oversegmentation.

The hetero-contacts were defined as the overlap of the dilated mask from the second phase:

$$M_{\text{Contact}} = M_{\text{P}}^{\text{dil}} \cap M_{\text{Mg}}. \quad (11)$$

This mask contains all pixels that are interpreted as hetero-contact regions. A hetero-contact pixel exists when a pixel of one phase is directly adjacent to at least one pixel of the complementary phase. The contact area A_K was calculated based on the number of contact pixels N_K and the pixel area A_{pix} :

$$A_K = N_K \cdot A_{\text{pix}}. \quad (12)$$

The calculated contact area A_K was normalized to the total area of the analyzed image A_{ges} . The resulting hetero-contact area fraction (HCAF) is given by

$$\text{HCAF} = \frac{A_K}{A_{\text{ges}}}. \quad (13)$$

The HCAF describes the relative proportion of the image area in which hetero-contacts exist between the phases. Higher values correspond to an increased proportion of interfaces between the components

The combination of morphological analysis (PSD), quantitative composition analysis (Lacey index), and structure-resolving hetero-contact analysis (HCAF) enabled the systematic characterization of the relationship between agglomeration behavior, spatial mixing, and phase contact.

3 Results

To demonstrate the applicability of the developed method, it was applied to selected samples at various stages of the mixing process. To characterize the hetero-agglomerates, the PSD, the mixing state using the cell-based Lacey index, and the HCAF were utilized to capture both structural and distribution- and contact-based properties.

Fig. 4 shows the averaged particle and agglomerate size distribution of samples taken at the beginning of the process immediately after the mixing zone (P1) and at the end of the process (P6), as determined using Fiji, presented in both number-based and mass-based representations. The number-based distribution shows only a slight shift of the PSD from P1 to P6 toward larger particles. The mass-based PSD reveals clear differences. At P1, the mass median is approx. 19 μm , which already indicates early agglomeration in the mixing zone. As the process progresses, the PSD shifts toward larger agglomerate sizes, with the mass median reaching approx. 39 μm at the end (P6). This indicates continuous agglomeration during the mixing process, which manifests itself in the formation of large cluster structures.

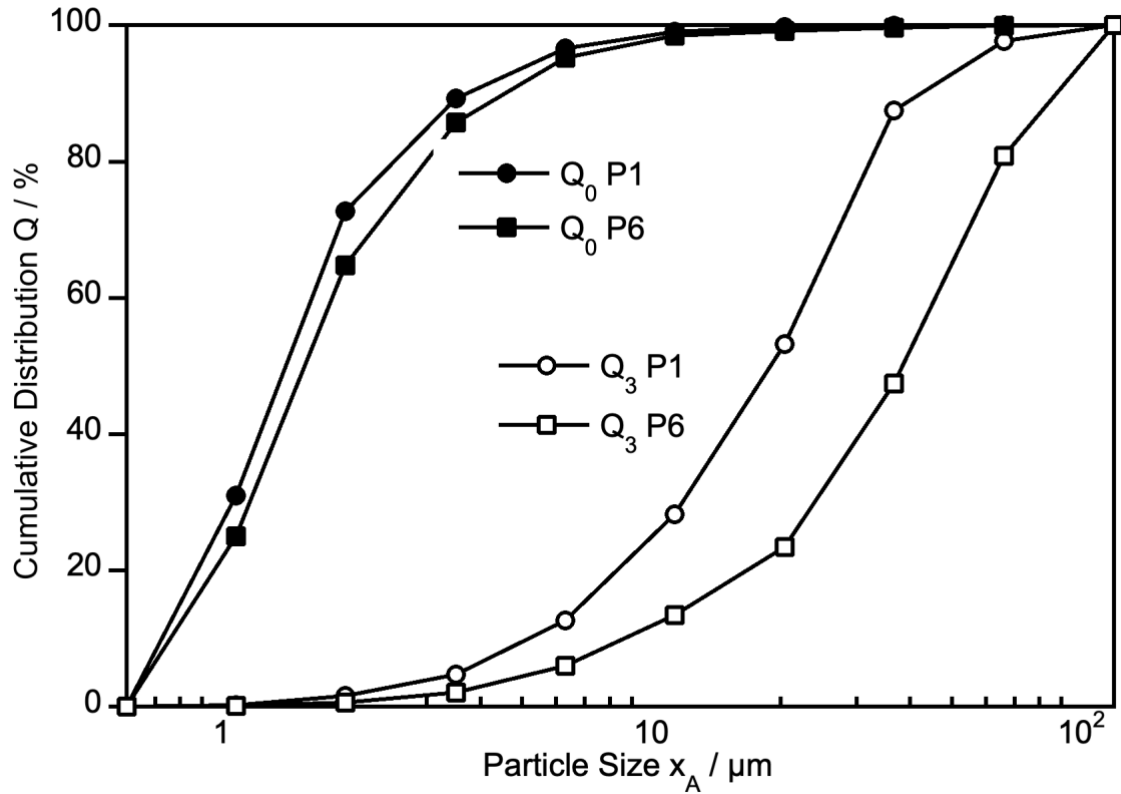


Fig. 4. Averaged number- and mass-related PSD of the investigated samples at P1 and P6.

Fig. 5 shows exemplary SEM images with EDX element overlays of samples at positions P1 and P6, as well as the corresponding element maps for P and Mg. A qualitative increase in phase mixing is evident from P1 to P6. In addition, for P6, the hetero-contact mask generated based on the extended phase overlap and its overlay with the SEM image are shown to visualize the hetero-contact areas.

The quantitative results regarding mixing quality and the formation of hetero-contacts are shown in Fig. 6. The data are based on the same samples used in the PSD analysis (see Fig. 5), although, in contrast to the PSD analysis, the figures here show the mean values per sample rather than the mean values per parameter.

Both the Lacey index and the HCAF increase continuously during the mixing process from P1 to P6. Already at P1, both parameters reach relatively high values ($M \approx 0.8$; HCAF up to 16%), confirming early agglomeration and mixing in the mixing zone (see PSD analysis, Fig. 5). At P6, even higher values are observed, indicating progressive phase mixing and increased hetero-contact formation. Taking into account the size distribution of the agglomerates (see Fig. 5), it becomes clear that progressive agglomeration is not only accompanied by improved spatial homogeneity but also promotes the formation of hetero-contacts. A positive correlation between M and HCAF is observable for the majority of the samples, though it does not occur consistently. This inconsistency is likely due to the different images, which capture varying local

compositions. For example, a sample may be well mixed overall (high M) but still exhibit a relatively coarse structure with larger homo-agglomerates. In such cases, the extent of hetero-contacts (HCAF) remains limited despite good spatial distribution, as contact formation strongly depends on the fineness of mixing and the resulting interfacial structure.

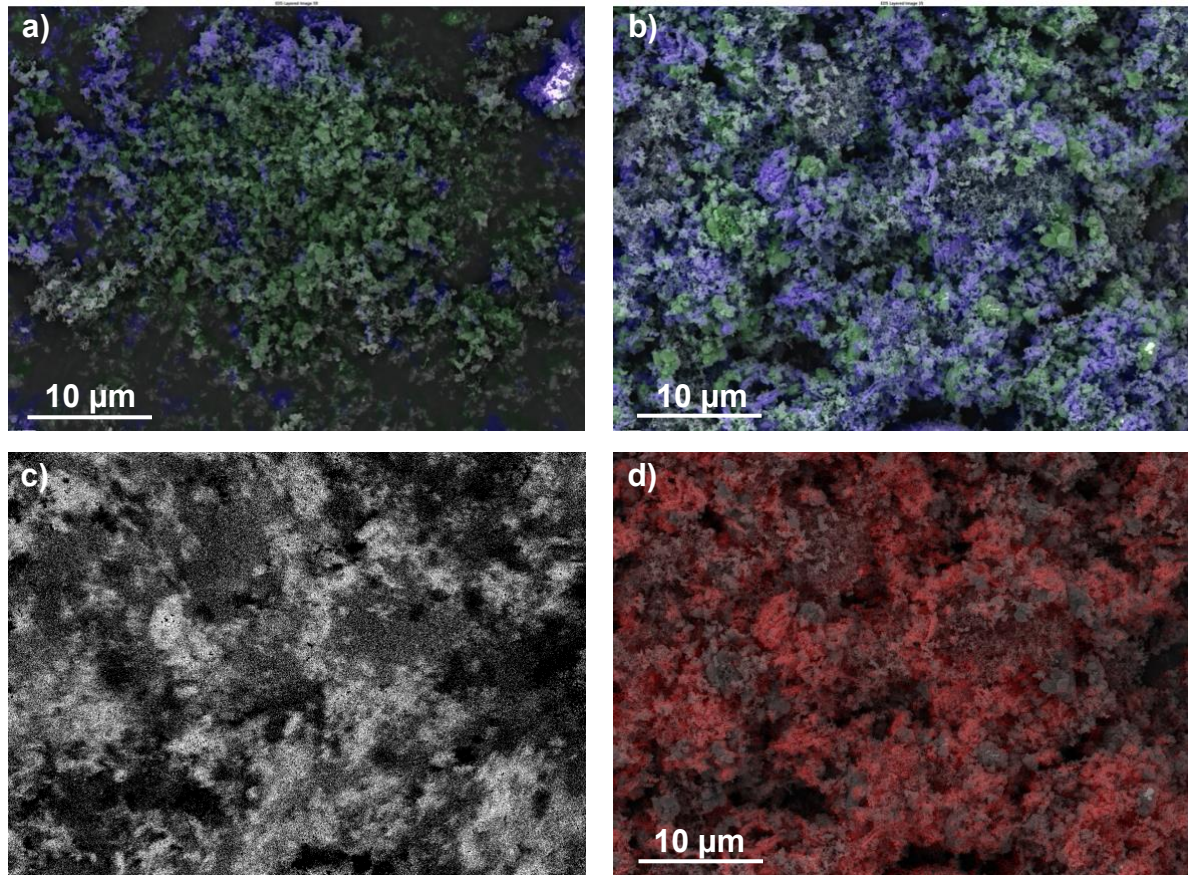


Fig. 5. SEM image with EDX elemental overlay for sample at positions 1 a) and 6 b), created hetero-contact mask for the sample at P6 based on dilated phase overlap c) and overlay of the hetero-contact mask on the corresponding SEM image for spatial visualization d).

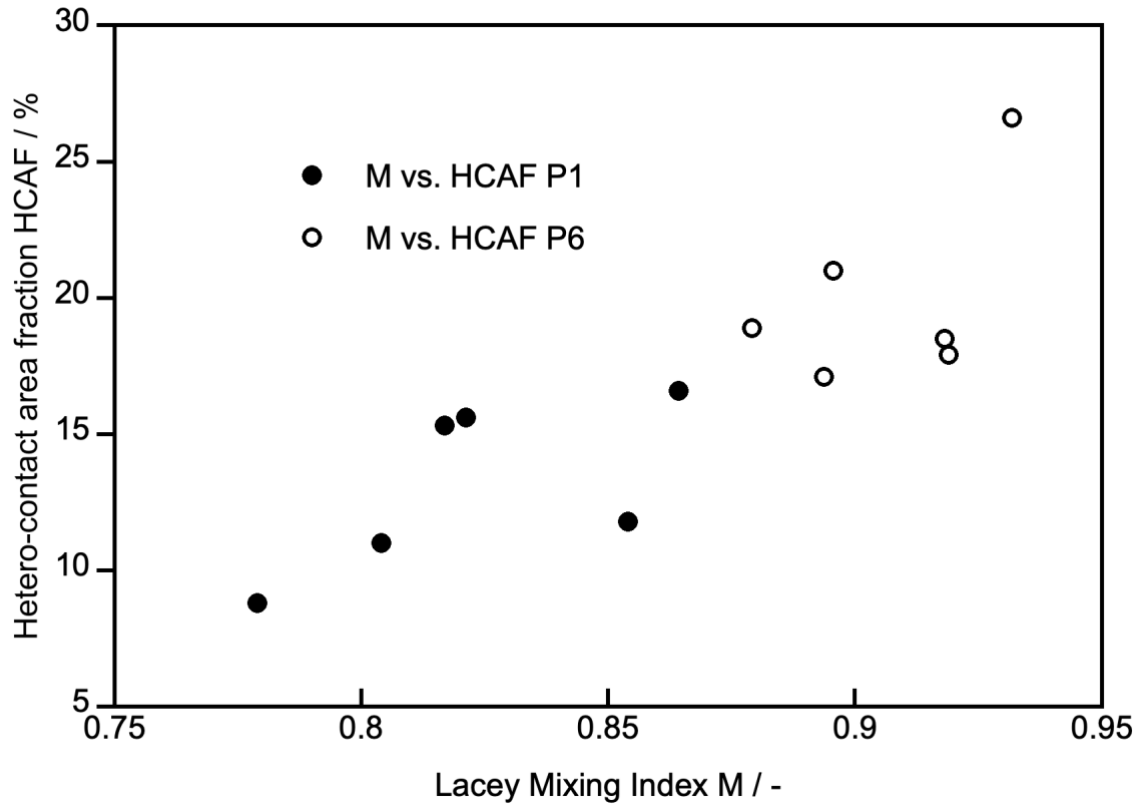


Fig. 6. Correlation between mixing state (Lacey index) and hetero-contact area fraction (HCAF) across all analyzed samples at P1 and P6.

4 Summary and outlook

In this work, a method was developed for the quantitative characterization of the mixing state and hetero-contacts in gas-phase-based hetero-agglomerates. By integrating EDX maps, intensity-based analysis, and image analysis techniques, spatial homogeneity (Lacey index) and the hetero-contact formation (HCAF) could be reliably determined.

The method consistently captures structural changes during the mixing process. From P1 to P6, there is a significant increase in phase mixing, which manifests as higher homogeneity and an increased hetero-contact fraction. The combined analysis of distribution and contact parameters provides a deeper understanding of the agglomeration and mixing mechanisms.

To validate these trends, further measurements at additional sample positions are required to systematically capture local inhomogeneities and position-dependent effects. The established methodology forms the basis for future studies on the influence of process parameters with the aim of targeted process optimization.

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