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Research proposal for IFPRI-Grant

Spray-Drying of Pastes with ACLR-Nozzle for Process Intensification

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Description of research proposal

1 Topic of the research project

Spray-Drying of Pastes with ACLR-Nozzle for Process Intensification

2 Introduction and background

Spray-drying is a widely used process for the production of powdered products from liquid formulations. The spray-drying process can be divided into three main steps: Atomization, convective drying in a hot air stream, and powder separation [1]. A scheme is shown in Figure 1.

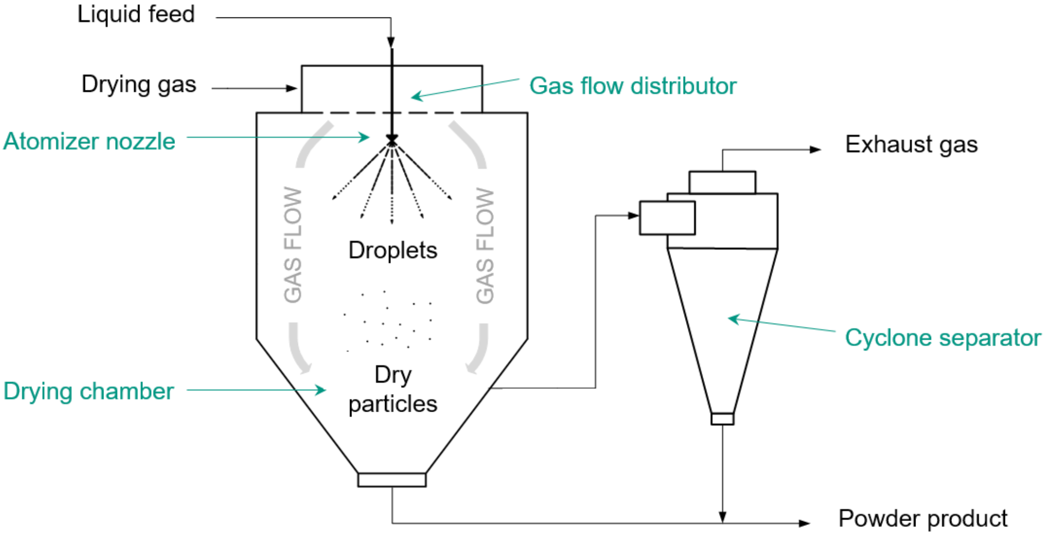

Figure 1: Schematic of the spray-drying process

The atomization of a bulk liquid into small droplets leads to a drastic enlargement of the surface-to-volume ratio, enhancing heat and mass flow in the subsequent convective drying step. The surface-to-volume ratio conditions product quality, since particles that are too small can burn, while particles that are too large do not dry sufficiently. The latter leads to increased stickiness of the particles, powder adhesion in the process equipment and storage problems [2]. Therefore, a narrow droplet size distribution is generally favorable. As with all drying processes, water removal is a very energy-consuming process, especially as internal-energy recovery is restricted in spray-drying [3]. Moreover, the powder throughput of a spray-dryer is limited by its specific water evaporation rate at given process conditions [2]. In industrial applications, the aim is therefore to feed the media into the drying process with the lowest possible water content (high dry matter content). This allows a higher product throughput with a constant water evaporation rate.

Using more energy-saving methods, such as membrane processes or multi-stage evaporation, for the upstream concentration, the total energy consumption and process footprint can be reduced. According to a model calculation on industrial spray drying by Fox et al. [4], an increase in feed dry matter content by 1% leads to a decrease in thermal energy consumption of the spray dryer by 3.8%. It also leads to a decrease in total energy consumption of 2.5%, when the energy for pre-concentration in an evaporator is taken into account. However, the dry mass content cannot be increased at will, since the viscosity of the liquid also increases with increasing solid concentration [5]. At high viscosities, the atomization step becomes more energy intensive, since the volume-specific atomization energy requirement for droplet breakup increases sharply as the internal resistance forces of the liquid increases. Larger droplets and/or wider spray droplet size distributions are then produced, up to the point where atomization is no longer possible.

For droplet formation, pressure-swirl (PS) nozzles are commonly used in spray-drying processes on industrial scale [1]. However, for PS nozzles the highest processable viscosity is considerably lower, compared to pneumatic nozzles [6].

Pneumatic atomizers are usually used for atomizing highly viscous liquids. The atomization energy is transferred via a gas stream [7]. In this type of atomizers, a distinction is also made between external-mixing and internal-mixing atomizers, depending on where the gas and liquid flows are combined [8]. An advantage of external mixing nozzles is that the gas and liquid flows can be set independently of each other. However, they tend to produce wide droplet distributions unless high rates of atomizing gas are used [9]. This makes the use of external-mixing pneumatic atomizers on an industrial scale not economically feasible.

In internal-mixing nozzles, an already formed two-phase flow exits through the nozzle channel. This allows for a more efficient transfer of energy from the gas to the liquid [9], as well as the ability to handle higher-viscosity liquids than with pressure swirl nozzles [10]. A drawback is that gas and liquid flow cannot be set entirely independently of each other [11].

With that in mind, the LVT-Institute developed a specific type of internal-mixing nozzle atomizer, the Air-Core-Liquid-Ring (ACLR) atomizer. A schematic of this nozzle can be seen on Figure 2 (left). The device is composed of two concentric tubes. The outer tube is where the liquid feed flows, while a capillary at the center carries the gas and injects it in the center of the mixing chamber. This forms an annular liquid flow, with a gas core. As this two-phase flow exits the nozzle through the outlet channel, the gas phase expands, and the liquid phase forms a cone like lamella that then disperses into droplets. The thickness of the lamella is correlated with the droplet size while the outlet diameter of the nozzle is much larger, typically larger than 1.5 mm. This nozzle design allows for relatively large particles in the feed stream, as the risk of clogging is low. The studies so far published, have involved both experimental and numerical analysis of the atomizer, to better understand its performance and its potential (see Figure 2 (right)). The ACLR nozzle has been proven successful for the atomization of highly viscous liquids up to 690 mPa·s [11].

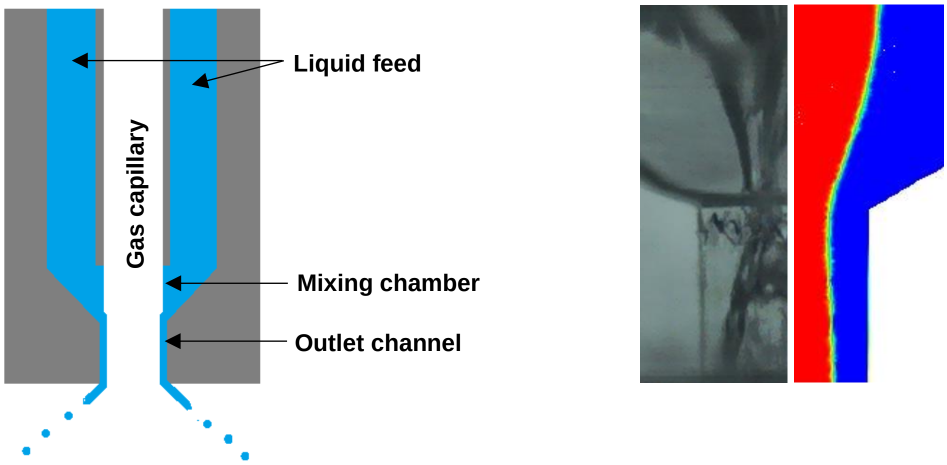

Figure 2: Scheme of the ACLR nozzle (left). Experimental high-speed image of the flow inside the nozzle and the computational simulations performed to represent the internal flow in the atomizer (right)

With the aid of high-speed images of the two-phase flow inside the atomizer, Stähle et al. [10] showed that, if a continuous liquid annular flow forms in the nozzle outlet channel, a stable spray is formed with small droplets. Wittner et al. showed the potential applicability of the ACLR for reducing total energy consumption in spray-drying processes, with theoretical energy savings of up to 29% [12].

Despite these major benefits of the ACLR nozzle, further studies on the scale-up potential of ACLR atomizers are still needed before a widespread application of the technology in industrial processes is conceivable. As reported by Wittner et al. [11], moisture content and water activity in the final powder product should be decreased to meet industrial demands. Additionally, based on Wittner et al. [13], we assume that in order to properly understand the process-function of the nozzle, and how spray performance relates to operating conditions and feed composition, we need to comprehend the flow behavior inside the ACLR nozzle. For this purpose, a CFD model is being developed in an ongoing project for the simulation of the internal flow of the ACLR-nozzle. Based on these investigations, it is assumed that the spray performance of the ACLR-nozzle can be enhanced by geometrical improvements. This should lead to a further reduction of the spray droplet size widths and increase the maximum possible viscosity that can be atomized for spray drying applications. In addition a further nozzle scale-up can be based on these results. Comprehending this process-function is therefore not only important for the atomization itself, but also for the subsequent drying step.

This process-function establishes the relation between the entry feed composition and droplet morphology. Nonetheless, it is well known that the droplet composition and morphology together with the process variables have a high impact on the final powder product [14–16]. Powder properties such as powder flowability, particle size, as well as particle and bulk density relate directly to the morphological structure of the powder particles. Although morphology is material-specific, it is also dependent on, for example, initial feed solids content, atomization and drying temperature [15]. The work of Hecht and King [17] also showed, that morphological changes, such as vacuole formation, can influence mass transfer rates and therefore drying kinetics. Consequently, it is very import to investigate the dynamic interactions between particle composition, morphology development and drying kinetics.

One way to examine the drying kinetics and morphology development is to directly observe the structure of the drying spray-droplets. This also allows acquiring accurate insights into the structural changes during the drying process. Direct observation of the drying droplets is unrealistic in a commercial spray-dryer or a modular nozzle test rig, due to the number of droplets and the difficult accessibility of the equipment. Furthermore, the transition from a droplet to a particle during spray-drying occurs quasi-instantaneously, which makes it almost impossible to track the drying kinetics and the morphology development of the particle. Consequently, for mechanistic studies of the drying process, experimental setups that center around drying a single droplet have been more and more the focus of research [18].

In single droplet drying (SDD) experiments, an isolated droplet is dried under a controlled drying environment [19]. The drying temperature, air humidity and air mass flow is set to mimic the convective drying process during spray-drying. Several experimental setups exist for SDD and are usually divided into levitation methods and free flight drying methods. Free flight drying methods are rather impractical for the examination of drying kinetics, as a continuous tracking of the droplet during drying is not possible. A commonly employed contact levitation method is a SDD experiment with a droplet suspended on a thin filament [20–22]. This allows easy monitoring of droplet mass and temperature changes of the droplet [23]. A drawback of this method is the intrusiveness of the filament on heat transfer.

An alternative to contact levitation is the immobilization of the droplet by acoustic levitation. In acoustic levitation, a single droplet is suspended due to a counterbalancing acoustic force while drying. [24]. A major benefit of this setup is that the drying of a droplet can be investigated non-intrusively, while one drawbacks is the not well-understood influence of the ultrasound on the drying kinetics.

Another tool to investigate one-dimensional drying kinetics is thin film drying [25]. Previous work has shown that a film thickness of micrometer- to nanometer-scale is feasible [26]. Schutyser et al. [18] has suggested that thin film drying technology could be a valuable tool to compliment the results generated by single droplet drying experimental setup.

Single droplet drying has been used extensively in literature for studying the morphology development, both by contact levitation [20,22,27] as well as by non-contact levitation [14,23,28,29]. The SDD setups are usually equipped with a CCD-camera with microscope optics for a continuous observation of the droplet during drying. Crust formation and structural changes can thus be determined directly. Nuzzo et al. [23] found for milk powder that it was possible to predict the morphology of spray-dried particles largely by means of SDD experiments, despite significantly longer drying times. The effects in the SDD tests were significantly more pronounced compared to the spray-drying tests. It is therefore expected that, despite the different drying systems, conclusions can be drawn between SDD and spray-drying tests with respect to the development of the particle morphology during drying.

The LVT-institute has many years of experience with single droplet experiments and equipment for the investigation of e.g. interfacial properties in single droplets. This mainly involves equipment with droplets suspended on filaments. In a just started research project financed by German Ministry BMWi, the LVT-Institute develops an experimental setup for SDD.

3 Project objectives

Spray-drying of liquid products is a key technology that is widely used in many industries. Its environmental impact could be reduced through process intensification by increasing the concentration of the feed liquid to be dried, thereby reduce energy consumption and process footprint. This requires the ability to atomize highly viscous liquids (especially pastes) and achieve short drying times to produce a suitably dry product that does not stick to process surfaces without changing functionality of the dried product. Key objectives of this project are:

- Validate the ACLR atomizer technology to enable spraying of highly viscous liquids for spray drying applications by improvement and upscale of the ACLR-nozzle.
- Evaluate the impact of the composition and morphology of the atomized droplets on the drying kinetics and develop drying models for highly concentrated feeds by single droplet drying.
- Investigation of the applicability of the ACLR nozzle for spray-drying of highly viscous liquids.

4 Methodology and work plan

The project is divided into three main working packages (WP). The ACLR nozzle is proposed as a suitable atomization technology for spraying of highly viscous liquids or pastes and generate droplets smaller than 100 micron. The central task of the first working package (WP) will be the validation of the ACLR nozzle to enable atomization of highly viscous liquids. CFD simulations will be used to optimize nozzle design. Process- structure-functions for the nozzle will then be established based on spray performance measurements. The gained insights can be used for further upscaling of the process. A period of 18 months is planned for this work package.

The objective of the second WP is to investigate the impact of the composition and process parameters on the drying kinetics and morphology development of highly viscous liquid or paste droplets in controlled conditions. For this purpose single droplet drying experiments will be carried out, in which droplets can be dried under precisely defined conditions. These studies will also be used to explore a possible relation between the morphology development and the drying kinetics. Taking into account the results of the first two WPs, a model for drying of highly concentrated feeds will be formulated as process-structure-function. Work on the second WP will start after viable formulations for the project are identified. It is currently estimated that the WP will be concluded after 18 months.

In the third WP, the applicability of the ACLR nozzle for industrial spray-drying processes of highly viscous liquids will be examined. For this purpose, spray-drying trials will be carried out. The results will be used to evaluate the validity of the in WP 2 formulated model. WP 3 will be the main workload during the project’s third year. It is estimated, that work on the final WP will start after the first two WPs have been completed and the process-structure-function has been elucidated in WP 2.

A working diagram is shown in Table 1.

Table 1: Working diagram for the planned project duration

Project year	1				2				3			
Quarter	I	II	III	IV	I	II	III	IV	I	II	III	IV
WP 1: Atomization with the ACLR nozzle	■	■	■	■	■	■						
WP 2: Evaluation of the impact of composition and morphology on drying kinetics			■	■	■	■	■	■				
WP 3: Industrial applicability of the ACLR nozzle for spray-drying									■	■	■	■

4.1 WP 1: Atomization with the ACLR nozzle

Numerical studies on the ACLR atomizer started with Wittner et al. [13], where an initial computational model to represent the multiphase flow inside the system was developed, using Ansys Fluent. This model is currently being refined, under a project financed by the DAAD. The objective of this research is that the predicted annular multiphase flow inside the nozzle can be used as an indication of whether a stable flow with the selected process and formulation conditions is possible, which corresponds with a stable atomization. In this way, the operational limits of the current nozzle design are determined, which serves as a basis for geometrical optimization or tailoring of the nozzle design for specific industrial applications.

In WP1 this model will be extended to the flow analysis of highly viscous liquid feeds. On the basis of the CFD results, the suitability and limitations of the nozzle design and atomization system will be determined, which allows the development of an improved nozzle concept. The focus of the research will be on the evaluation of process-structure-function for the nozzle. Therefore, the atomization capability of two different highly viscous liquids will be investigated. Process parameters are the nozzle design, the ratio of air-to-liquid flow rate (ALR) and the air pressure. Structure variables are the spray angle and the droplet size distribution, including its temporal stability. As model system, aqueous solutions of starch and derivatives (like maltodextrin) are proposed. Starch and its derivatives are commonly used as thickener and stabilizing agent and can form aqueous solutions and suspensions. To ease the transfer of knowledge between the research results and industry partners, the partners could also provide relevant model liquids and suspensions. Nonetheless, the model system has to be defined and characterized at the beginning of the experimental study, to allow a systematic investigation of the relevant process parameters.

For validation of the CFD model, the flow pattern inside the nozzle will be analyzed. Taking into account the numerical and experimental results, a comparative analysis can be done to investigate the feasibility of spraying highly viscous liquids and pastes to generate droplets smaller than 100 µm. The current CFD model has shown so far good agreement with experimental data up to 400 mPa·s, and will be further used with higher viscosities. The experimental study can be carried out on the institute's facilities. The setup has been proven successful for viscosities of up to 690 mPa·s with droplet sauter mean diameter of around 70 µm.

On the basis that Kelvin-Helmholtz instabilities lead to a disintegration of the liquid lamella which ends in droplet formation, one can roughly estimate the maximum viscosity that can be atomized by the current ACLR nozzle. For this rough estimation The droplet diameter was calculated, taking Kelvin-Helmholtz sheet instabilities into account (Equation 1). The equation for the droplet diameter x_d is given by

$$x_d = 1{,}882 \left(\frac{8a_b}{k_{max}}\right)^{\frac{1}{2}} (1 + Oh)^{\frac{1}{6}} \tag{1}$$

with half of the width of the liquid sheet a_b in m, the wave number of the maximum instability k_{max} in 1/m and the dimensionless Ohnesorge number *Oh*, a dimensionless number that relates the viscous forces to inertial and surface tension forces [30]. For estimation of k_{max} eq. 1 was used with measured values at 140 mPa s. The development of lamella thickness was estimated in case 1 (C1) to develop linear with the droplet size for 690 mPas and then extrapolated for higher virtual viscosities. In a second case (C2) it was assumed that the lamella thickness is constant. The results are shown in table 2.

*Table 2: Estimations of lamella thickness depending on the liquid viscosity. *measured values*

Liquid viscosity η in mPa·s	**Lamella thickness d_l in mm, C1**	**Droplet diameter x_d in µm**	**Lamella thickness d_l in mm, C2**	**Droplet diameter x_d in µm**
140*	0.130*	43*	-	-
690*	0.228	70*	-	-
1000	0.290	86	0.290	86
2000	0.490	120	0.290	96
5000	1.09	195	0.290	112

Allowing the lamella thickness to increase leads to comparably large droplets e.g. 195 µm at 5 Pa s. Assuming the lamella thickness to be constant leads only to a moderate increase of the droplet size from 86 mm to 112 µm while the viscosity increases from 1 to 5 Pa s. This calculation is very limited in its significance but one can state that the lamella thickness has a much higher impact on the droplet size than the viscosity. This leads to the conclusion that if its possible e.g. by constructional means or process variables like the air pressure to limit the increase of the lamella thickness, it should be possible to produce droplets which are small enough for spray drying even at high liquid viscosities.

The available facilities for these investigations are: Air compressor which can be operated at pressures of up to 9 bar (RSF-Top 7.5, Renner GmbH, Güglingen, Germany); A pressure vessel and various eccentric screw pumps with volume flow between 5 and 100 L/h for the liquid supply; A test rig equipped with a temperature control for the feed liquid. Here the spray droplet sizes can be measured over time using a laser diffraction spectrometer (Malvern Spraytec, Malvern Panalytical Ltd, Malvern, United Kingdom) in a size range of 2-2000 µm; A high-speed imaging system (OS3-V3-S3, Integrated Design Tools Inc., Tallahassee, FL,USA) is available to analyze the spray angle and the flow pattern inside the atomizer. The rheological characterization of the model systems can be executed with a double gap system (Physica MCR 101/103, Anton Paar, Graz, Austria).

4.2 WP 2: Evaluation of the impact of the composition and morphology on the drying kinetics and model development by single droplet drying

The development of a suitable methodology for single droplet drying experiments is already being pursued at the institute as part of the AiF project 21662 N "Spray-drying of emulsions for microencapsulation". For this project, an experimental setup is will be installed that allows continuous monitoring of the drying kinetics and particle morphology during the drying of emulsion droplets.

In WP 2, the experimental setup for emulsion droplets will be extended to droplets of highly viscous solutions. This is expected to be fairly straightforward. A potential challenge is mainly seen in the production of uniform droplets from formulations with varying solids concentrations, as the rheological behavior differs between these formulations. Using of different droplet injection systems might be necessary. Nevertheless, as the development of the experimental setup is still in its early stages, advices and experiences of industry partners on single droplet drying could prove valuable. The experimental studies in WP 2 will focus on the influence of air temperature, air velocity and total solids content on the drying kinetics and morphology development during convective drying. Therefore, the same highly viscous liquids will be used as in WP 1. By monitoring droplet size, evaporation rate and temperature profiles of the droplet, the drying kinetics can be determined. For the morphology development, different structural changes are expected. Besides changes in particle size, other phenomena may occur during drying of a liquid droplet. These include the formation of an inhibitory skin, changes in the surface structure (smooth or wrinkled), and rupture of the particle. To observe these phenomena, various analytical methods are available at KIT. In addition to light microscopy and SEM (scanning electron microscopy) images, the particles can be examined in µCT (micro computed tomography). Solubility tests can provide information about the properties of the particle skin.

Based on the results of the experimental study a model for the drying of highly viscous liquids will be developed. The model will be formulated as a process-structure-function to elucidate the impact of feed composition and morphology development on the drying kinetics.

4.3 WP 3: Proof-of-concept of industrial applicability of the ACLR nozzle for spray-drying of highly viscous liquids

It is expected that from the results of WP1 and WP2 conclusions can be drawn on possible process windows for atomization (WP1) and drying (WP2) conditions for highly viscous liquids. In WP3 the applicability of these conditions on a spray drying process will be investigated. This will be executed in pilot scale on the institute's spray-dryer (Werco SD-20, Fa. Hans G. Werner, max. water evaporation capacity of 20 l/h, max. air inlet temperature 250 °C, max. air outlet temperature 100 °C). Solutions with the highest possible viscosity at acceptable spray droplet size are selected for this purpose. The spray-drying experiments can be conducted under variation of the air temperature at the inlet (160-220 °C) and outlet (65-95 °C), the air velocity and the spray droplet size. This way, the drying rate and the surface-to-volume ratio in the particle are varied. Measurements of the residual moisture content can be used to validate the drying kinetics determined in WP 2. After spray-drying, the structure of the produced powder will be characterized and compared to the results of WP 2. The same powder characterization methods can be used as in WP 2. The results allow a validation of the process-structure-function developed in WP 2, which will be modified if necessary.

Figure 3: The institute's pilot scale spray-dryer

5 Transfer of knowledge

Based on the results, solution concepts can be derived that contribute to a targeted and understanding-based process design for the production of powder products from highly viscous liquids. These solution concepts form the basis for an implementation by the industrial user with their own recipes and processes. The research results can be presented and discussed in accompanying meetings and appropriate international conferences and are summarized in annual reports. The results will be published in appropriate international journals.

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