IFPRI PROJECT EXTENSION PROPOSAL

ONLINE ANALYSIS AND CONTROL OF PRODUCT PARAMETERS BY CONTROLLING THE VISCOSITY OF THE ATOMIZED SOLUTION

by

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1 INTRODUCTION

Spray drying is a widely used industrial process employed to convert liquid or slurry materials into dried powders or granules. This technique is particularly valuable in various industries including food, pharmaceuticals, chemicals, or ceramics. The process involves atomizing liquid feed into small droplets, which are then introduced into a hot gas stream inside a drying chamber.

The fundamental principle behind spray drying is rapid moisture removal through evaporation, which enables the transformation of a liquid substance into dry powder or granules while preserving its essential characteristics. The liquid feed material is typically a solution, suspension, emulsion, or paste, containing the desired product.

During the process, the atomized liquid feed enters the drying chamber and encounters a stream of hot air or other drying gas medium. The heat causes the tiny droplets to quickly give away moisture, allowing the formation of solid particles. The resultant dried particles are collected from the chamber's bottom directly or with the use of cyclones, and the gas, collected at the top, is usually filtered and recycled.

Spray drying offers numerous advantages, such as the ability to produce fine and uniform powders with controlled particle sizes, enhanced product stability, improved shelf life, and the retention of product properties, such as its flavor, color, and bioactivity. Additionally, this method enables efficient and continuous production of dry powders on industrial scale.

However, the process parameters, including inlet temperature, feed rate, droplet size, and drying gas characteristics, must be carefully controlled to achieve the desired product properties and prevent potential issues, like product degradation or undesirable particle agglomeration.

Studies on the influence of the solution properties on particles' morphology during co-current spray drying have shown that the structure of the final material is largely influenced by the process of droplet formation during the atomization process.

Incorrectly selected operating parameters of the nozzles have significant negative influence on the final result of the drying process. The rheological properties of the liquid phase, measured in a separate laboratory under isothermal conditions, have shown that for correct results the nozzle should effectively break up the liquid flow for given parameters of the atomization process. During preliminary experiments it was found that in some cases the nozzle did not produce uniform droplet streams, even though the solution parameters fulfilled the requirements of the operating window. Changes in liquid properties related to pumping (shear rates) and local variations in temperature or concentration were found to be crucial for the effective spray drying process. To have full control over the atomization process, one needs to know the exact parameters of the material sprayed at the inlet of the nozzle. Only such precise control of the atomization process makes it possible to determine the effects of the rheological parameters on properties of produced powders.

2 THE SPRAY DRYING TUNNEL

The semi-industrial scale spray column, built and owned by the Faculty of Process and Environmental Engineering, can operate in co-current and counter-current mode. The size of the equipment allows to conduct experiments and analyze samples of dried material in conditions very close to those of fully industrial scale. The equipment consists of a spray-drying tower itself, of 8 meters in height and 0.5 meters in internal diameter, a system for preparation and distribution of the raw feed, a system for preparation and distribution of the drying air, a product collection system, and a system of control and data acquisition. The P&ID schematic of the equipment is presented in Figure 1.

The raw feed is prepared in an unpressurized tank with a bottom, low-speed ribbon mixer, and equipped with additional wave breakers. The tank is equipped with a heating jacket and its volume is 0.15 m^3. The initial temperature of the feed stream is controlled and stabilized with the T31 temperature sensor coupled with flow rate control of the heating medium. The liquid is transported from the mixing tank to the spray nozzle with the use of a high-pressure pump (CAT, USA or Netzsch, Germany) through double-jacketed piping, the temperature of which is controlled with the Therm 2 thermostat. The control of the pump is carried out via reads of the F35 Coriolis mass flow rate sensor (Emerson, USA), and the stability of the liquid over the course of the drying process is verified with the D36 density meter (Emerson, USA). The operation of the nozzle is verified by measurements of the P40 pressure sensor (Wika, Germany). The solution temperature is controlled by thermocouples: T41 after the pump, T73 between the piping thermostat system and the lance thermostat system, and T63 inside the lance just before the nozzle. The multi-level control allows to properly adjust and stabilize solution parameters over the whole feeding section. The regulation is being carried out automatically, based on the reads from the sensors, which have been implemented into PID regulators.

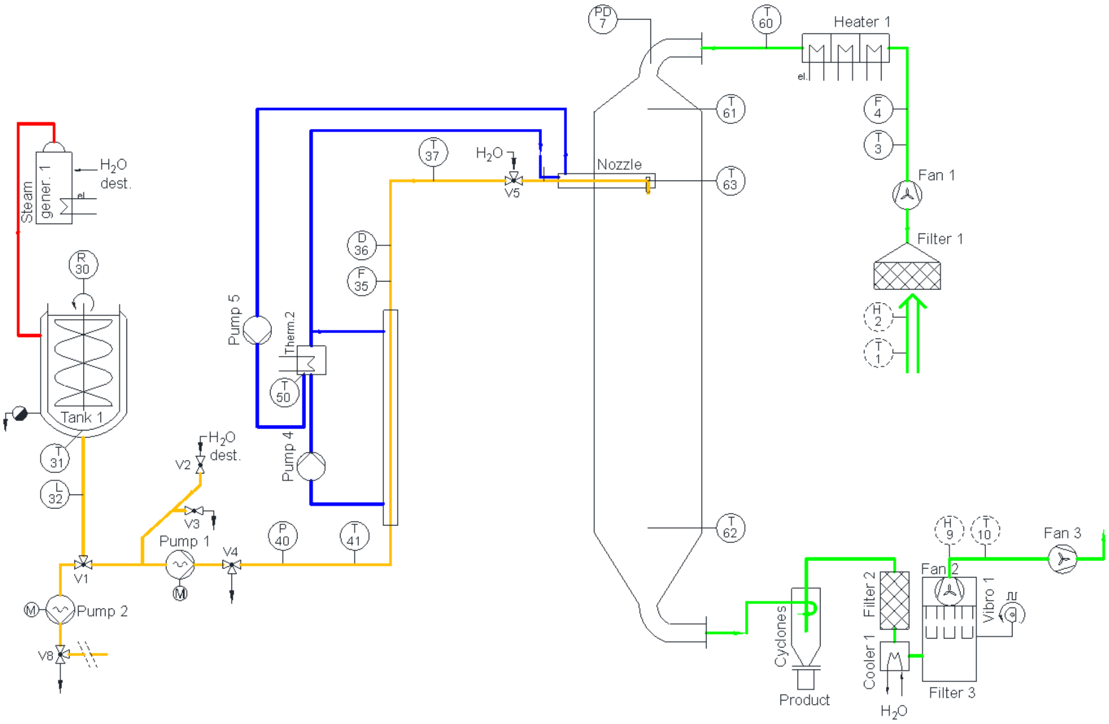

Figure 1. P&ID schematic of the co-current spray drying equipment

The drying medium (air) is taken directly from the technological hall, filtered with the use of an ePM1 class filter according to ISO 16890 standard, and directed toward the heater by a fan blower coupled with a PID regulator, equipped with the F4 vortex flow meter (Endress+Hauser, Germany). Thus prepared drying medium is directed toward the spray column. To ensure uniform flow of the gas, special meshes have been installed at the inlet. Under the meshes, the temperature is verified with the T61 temperature sensor. Then, the air contacts the dried medium within the column and then leaves the drying chamber. The dried particles are separated in a system of cyclones and bag filters. The implemented regulatory systems based on the CompactRIO platform (NI/Emerson, USA), allows to measure and control the following parameters over the course of the drying process:

- Flow rate and temperature of the drying medium
- Pressure inside the drying chamber
- Process evaporation efficiency
- Control of atomization parameters
- Drying process progression
- Local and average temperatures of the sprayed medium and the drying medium in the cross-section and along the drying column on 9 levels, in situ measurements with the use of additional sensors
- Local and average velocities of the dispersed medium in the cross-section and along the drying tunnel height, in situ measurements with the use of additional sensors (np. Dantec LDA or CTA, Denmark).

3 POWDER MORPHOLOGY

The analysis of the quality of powders obtained during the experiments can be determined on the basis of testing the morphological properties of the particles. The analyzed properties are:

- Particle size distribution (PSD) measured by laser diffraction methods.
- Bulk density, apparent density and specific density using a helium pycnometer and bulk measuring cylinders in accordance with EU standards.
- Equilibrium moisture content and sorption izotherms (Novasina, LabMaster.
- Particle structures with optical spectroscopic microscope and SEM (scanning electron microscope).
- Moisture content in final product, angle of repose and wettability.
- Internal structure of the particle will be characterized using nitrogen absorption system, allowing determination of surface area, total pore volume, and pore size distribution inside the particles, (ASAP 2020 V3.01 H automated sorption system, Micromeritics) and SEM images of crushed particles to determine the shape and position of pores.

In drying experiments, only the rheologically stable solutions which do not change the properties with time in the feed tank will be used. As a base products, we can use maltodextrin (skin forming material), washing powders (porous materials) and water suspensions like yeast, chalk or coffee. We can also use the other test materials provided by IFPRI members, e.g. individual components of washing powders.

4 CHANGES IN THE DESIGN OF THE DRYING COLUMN

Currently, the drying column used in the Department of Process Engineering and Environmental Protection at the Lodz University of Technology was originally designed to investigate the kinetics of material drying along with measurements of particle size distributions using a Phase Doppler Anemometry (PDA) system.

The laser system necessitates specialized construction of inspection windows, limiting the column width to 0.5 meters. Such a small column diameter mandates the use of nozzles with a very narrow spray angle, which generate a highly dense droplet stream. As part of this project, we propose the reconstruction of the upper part of the drying chamber to install a chamber with a diameter of 1 meter. Expanding the column will enable the use of nozzles with a larger spray angle (closer to the nozzle used in industry) and will facilitate the observation of droplet formation zones and measurement of size distributions using optical systems.

The new chamber section will be directly connected to the existing older element, where particles will undergo final drying before being measured for morphological studies. The evaluation of spray efficiency and particle size distribution will be conducted using the Shadowgraph ParticleMaster system by LaVision (Germany). This system enables macroscopic observation of forming droplets and determination of their size through image analysis. Unlike Doppler systems, this system does not have constraints regarding the column width. We propose the implementation of elongated windows to scan the droplet formation zone and accurately determine the initial size distribution.

The atomized solution will be delivered to the nozzle through a thermostatically controlled pipeline and a lance. Both the lance and the nozzle head maintain a constant temperature. Ahead of the lance, a monitoring system for the properties of the atomized solution will be installed. The mass flow rate of the solution, density, and temperature will be measured using a Coriolis meter (Emerson, USA). The aforementioned parameters in industrial installations are typically controlled (or at least monitored). However, due to measurement difficulties and the historically limited availability of reliable devices, continuous (in-line) monitoring of the viscosity of the sprayed medium is seldom carried out. Previous studies (including those within the IFPRI project) have indicated that even minor changes, such as temperature variations, can lead to significant viscosity alterations (by orders of magnitude). Consequently, this disrupts the atomization process and adversely affects the repeatability of the properties of the produced powders.Subsequently, the viscosity of the solution will be investigated utilizing a in-line process viscosity meter (e.g. XL7 Hydramotion, UK). Main parameters of the viscosimeter include: viscosity measurements in the range of 0 ÷1000 cP or broader, the range of pressure for measurements of 0 ÷ 50 bar or broader, the range of temperature for measurements of 10 ÷ 100°C or broader, ability to recording the measured value over time.

The prepared measurement system will respond to changes in the solution temperature and the flow rate, indicating the current state of the slurry before it is sprayed through the nozzle. This will enable the verification of the operational window of the nozzle for various sprayed materials and refine the relationships allowing the determination of the initial droplet size distribution based on the system state parameters.

5 AIM OF THE PROJECT

In the previous experimental stages it was observedhat viscosity of the slurry is one of the main factors determining the initial droplet size distribution in the atomization process, and thus, the final properties of the resulting powder. Therefore, the main goal of this project is to deeply investigate the dependency between powder properties and slurry viscosity, and create an on-line system for controlling the spray-drying process with viscosity of the slurry. With proper parametrization of the system, in the future it could be universal technique for controlling of product properties produced in industrial spray dryers for any kind of skin-forming material.

The project will consist of the following stages:

- Retrofitting and adaptation of already existing research equipment. This stage will include designing of the new top section of the column and selection of a proper spray nozzle for the system.
- Update of the existing laser measurement system. For proper measurement of the initial droplet diameter distribution, the currently existing laser measurement system needs to be adapted for the new tower section.
- Determination of the spray nozzle's operational window. Since a new spray nozzle has to be selected, preliminary experiments have to be conducted to determine the limits of the nozzle's operational conditions.
- Spray drying experiments with the use of the updated co-current setup.
- Examination of physicochemical properties and morphology of the particles. Upon collection of the dried material, samples from each experiments will be investigated in terms of their physicochemical properties.
- Data analysis and determination of mathematical relations between slurry viscosity and initial droplet size distribution. Dependencies between properties of the product and the slurry viscosity will serve as base for development of the control system.
- Validation of developed control system/mathematical relations by additional spray drying experiments

The outcome of the project is design of a pilot "atomization control system" for the industry with continuous monitoring of changes in viscosity, density and temperature of sprayed liquids.

6 PROJECT TIMETABLE

No.	Topic	Time
1	1. Formulation of the design of a new, wider top section of the spray-drying column, along with measurement windows for laser access 2. Selection of dried material 3. Selection of a proper nozzle for the drying process 4. Ordering and manufacturing of all the necessary equipment parts and fittings	1st year
2	1. Construction and retrofitting of the existing equipment 2. Preliminary experimental works for determination of operational windows and process conditions 3. Carrying out experimental drying processes and analysis of powder morphology obtained from different slurries and under different process parameters: - Identification of the effect of feed properties, feed rate, air flow rate and drying temperature on powder morphology; 4. Determination of process control methodology: controlling temperature, concentration and feed flow rate (pressure), according to the initial size distribution data	2nd year
3	1. Analysis of physicochemical properties of the obtained powder samples 2. Determination of mathematical dependencies between process conditions and product properties 3. Determination of design methodology of on-line atomization control system for spray-drying processes 4. Validation	3th year