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Advanced experimental analysis of drying kinetics in spray drying

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Abstract

A method for measuring drying kinetics of different products in a disperse system has been developed in the study. In order to carry out the experiments a 9 m long spray drying tunnel was designed, built and tested.

The phase Doppler anemometry (PDA) technique was used to determine initial spray atomization parameters, the structure of spray during drying, particle size distribution, velocity of the particles, mass concentration of the liquid phase, etc. Measurements were made at different distances from the atomizer and in the cross-section of the spray stream. Maltodextrin was used as a raw material in the experiments. Extensive spray drying tests were performed to determine the influence of operating process parameters on spray. Examples of the drying kinetic tests are presented and discussed in the paper. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: PDA analysis; Microseparator measurements; Droplet-particle transformation

1. Introduction

The key problem in spray drying which has not been solved yet is the determination of drying kinetics and degradation kinetics for heat sensitive products. The lack of appropriate experimental investigations is due to technical problems in carrying them out. The residence time of particles in the spray dryer does not usually exceed 30 s, and is often even shorter. Thus the whole process of dehydration, formation of solid structure, degradation, etc. takes a very short time, e.g. [1]. Therefore, a few attempts made so far to determine the kinetics of product drying and degradation have been restricted to the analysis of relevant parameters only at the dryer inlet and outlet, e.g. [2,3].

A complete experimental determination of the drying and degradation kinetics to get better knowledge of the mechanisms involved in transforming a droplet to the particle is the main aim of the whole project. Accomplishment of this task requires the application of a special equipment which would make it possible to take samples and to ensure residence time long enough. The main difference of our approach and the approach encountered in the literature in a systematic investigation of spray drying process for chemical and biological systems is involving in situ analysis of the properties of continuous and dispersed phases from atomization to collection of dry product.

2. Experimental equipment

2.1. Drying tunnel

In order to carry out studies mentioned above a 9 m long concurrent spray drying tower was designed, built and tested (Fig. 1). The tunnel is equipped with a 60 kW heating system, waste air cooling system, dedusting system and optical glass windows (50 in the whole tunnel) to perform measurements using laser technique (LDA, PDA).

The construction enables to take samples at subsequent time intervals and to make laboratory determination of moisture content, size distribution, etc. as well as the quality index specific for a given product at a different distance from the atomizer. The holes made to install the windows are also used to carry out temperature and humidity measurements by the microseparator inside the spray envelope.

To obtain uniform air flow in the test section, eight dense damping grids are installed before the inlet to the measuring section where a pneumatic nozzle was mounted. Feed is delivered to the nozzle by a peristaltic pump.

Dry material is conveyed to the dedusting and cooling system where dust is collected and the clean and cool air is discharged to the atmosphere.

The transport system of laser unit was designed for laser Doppler anemometry (LDA) and phase Doppler anemometry (PDA) measurements at an arbitrary height of the tunnel and in selected points in a given cross-section. Main elements of the transport system include a stable supporting structure and mobile structure carrying an optical bench and traverse mechanism. The mobile element slides along the

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Fig. 1. View of experimental rig.

stable structure by means of a drive. Elements of the laser unit, i.e. transmitter and receiver are placed on the optical bench.

The range of sliding distance of the entire measuring system is about 5.5 m. The spray tower is equipped with a data acquisition system which enables a continuous observation and control of drying process parameters (Fig. 1).

2.2. Microseparator

One of the most difficult parameters which must be measured during spray drying are temperature and humidity of a drying agent inside the spray envelope. Measuring of air temperature is interfered by the presence of particles being dried. The particles, hitting the probe, make correct determination of the air temperature impossible.

After literature analysis we decided to apply and modify the microseparator developed by Kievet and Kerkhof [4].

The basic idea of the design is to remove particles from the gas stream and then to measure temperature of the cleaned gas stream. To achieve this, a difference in inertia is used in the system in which the pathway of air cannot be followed by the particles due to sharp curve; in this case: 180° curve, fully reversal flow (Fig. 2).

The microseparator consists of two tubes: an outer tube of 300 mm long and 20 mm in diameter to deliver gas and particles to the measuring system and an inner tube (5 mm in diameter) supplying clean air to the thermocouple.



Fig. 2. The microseparator: (1) Vaisala humidity probe, (2) thermocouple (air temperature), (3) thermocouple (dispersed phase temperature), (4) glass window, (5) clean air outlet and (6) contaminated air outlet.

We decided to hide the inner tube in the outer tube which makes the separator move easier along a diameter of the tunnel. Clean air, leaving the inner tube, is delivered to the Vaisala humidity probe. A glass window was installed in one of the microseparator walls which allow us to check if vapor does not condense in the separator during the measurements. We also put another unsheltered thermocouple at the inlet to the outer tube to measure temperature of the wet dispersed phase.

To facilitate the read-out of the information, the microseparator is connected to the data acquisition system.

The experimental rig enables to perform the following measurements:

- moisture content of the sprayed material as a function of the distance from the atomizer and radius of the tunnel;
- local and average temperatures of spray stream and drying agent in the spray envelope in the cross-sectional area and along the length of the tunnel;

- determination of the quality index;
- LDA analysis of the flowfield in the spray tower;
- PDA analysis of the spray structure in the cross-sectional area and along the length of the tunnel.

3. Drying tests

To perform first experimental drying trials, a preliminary selection of suitable labile product was done.

Finally, we decided to carry out experimental trials with maltodextrin as a raw material. Maltodextrin is often used as a carrier in spray drying in food industry.

We have made extensive tests on maltodextrin spray drying for various feed rates and initial parameters of the heat carrier. Each test included the analysis of temperature distribution in the dryer, evaporation level and sprayed material structure. The structure of spray during drying was analyzed by the laser methods (PDA, FLOWLITE System by Dantec).

PDA technique was also used to determine initial atomization parameters as well as particle size distribution, velocity of the particles, mass concentration of liquid phase in the cross-section of spray stream, etc., in the spray drying tower during drying tests. Drying agent temperature inside the spray envelope was found using a microseparator. Examples of the results are presented below.

3.1. Temperature measurements

Temperature measurements were taken in five points along the diameter of the tunnel (however there are no limits here) and in 10 positions along the length of the spray tower. The microseparator enables to determine simultaneously local temperature of a heat carrier inside the spray envelope and temperature of sprayed material.

Fig. 3 shows gas temperatures inside the spray envelope and the temperature of dried material along the length of the tunnel axis for feed rate of 10 kg/h and atomizing air rate of 60 kg/h.

The profiles of the temperatures are characteristic for spray drying. The initial growth of gas temperature is observed in the spray envelope caused by an extension of the spray envelope and an increase of the amount of heat carrier which is in contact with the disperse phase, and then a decrease induced by liquid evaporation. At temperature of 220 °C (atomizing air/feed ratio 60/10) already at a distance of 2.5 m from the nozzle no difference occurs between the temperature of gas and spray (Fig. 3) which means that the evaporation process is completed. However, at temperature of 175 °C the evaporation process takes place still at the distance of about 3.5 m from the atomizer.

Fig. 4 shows changes of mean drying agent temperature along the length of spray drying tower for different atomizing air/feed rate ratios. The rate of heat carrier temperature drop is a function of the drying rate and, to a small extent, heat losses to the atmosphere.



Fig. 3. Temperature profiles along the axis of the tunnel.

For fine atomization (60/5 and 60/10) sharp drop of drying agent temperature is observed and then the average gas temperature is almost constant to the end of drying process. For coarse atomization drying agent temperature drop is gradual due to lower drying rate.

Conclusions concerned with the impact of initial parameters of atomization on drying process duration are also confirmed.

3.2. Material moisture content

One of the most important measurements carried out in this study was the determination of changes in moisture content of the material for different drying process parameters. The measurement included sampling of material from various heights of the spray drying tower using holes designed as for control windows.

Examples of the results of measurements of material moisture content are shown in Fig. 5. Fig. 5 shows moisture content profiles as a function of a distance from the atomizer for different feed rates and atomization air/feed rate ratios. The length of the tunnel necessary to complete drying process ranges from 1.2 to 5.5 m. For the worst atomization ratio (30/10) the drying process was not completed despite high initial gas temperature 220 °C.





Fig. 4. Mean air temperature in cross-sectional area of the tunnel.



Fig. 5. The moisture content profile as a function of a distance from the atomizer.

The results show that in the spray drying tower we may achieve a wide spectrum of drying conditions which enables a profound analysis of the mechanism of droplet to particle transformation.

4. PDA measurements

The most powerful tool we applied to analyze the structure of spray during drying to determine droplet to particle transformation was particle dynamic analysis (PDA, FLOWLITE System by Dantec). The PDA technique was used to determine initial atomization parameters as well as particle size distribution, velocity of the particles, mass concentration of liquid phase in the cross-section of spray stream, etc., in the spray drying tower during drying tests. Figs. 6–11 show examples of the results obtained from PDA analysis.

Figs. 6 and 7 show average concentration of fractions 1-8 and $58-66 \,\mu\text{m}$ during drying process. Concentration of small fraction increases during drying process, while concentration of bigger fractions decreases. Results described above are logical, confirm our understanding and reflect the run of drying process.

Figs. 8–10 display an example of local particle size distribution for three distances (0.2, 0.5 and 3.5 m) from the



Fig. 6. Average concentration of fraction 1-8 µm during drying process.



Fig. 7. Average concentration of fraction 58-66 µm during drying process.

atomizer (air/feed ratio 5/10, initial gas temperature $220 \,^{\circ}$ C). At the distance of 0.2 m from the atomizer two peaks are observed. Along the axis and in its vicinity (on the radius of about 6 cm) small particles are concentrated (Sauter mean about 40 µm). In the remaining volume of the spray (from about 6 cm up to the spray edge) larger particles occur (Sauter mean about 100 µm). The analysis of subsequent figures shows that this distribution is flattened due to spray dispersion at the distance of 3.5 m from the atomizer. In

each point of the spray there is practically an identical diameter distribution, e.g. [5].

This conclusion is of great importance to understand the phenomenon of simultaneous momentum, heat and mass transfer during spray drying.

Fig. 11 presents initial velocity of particular fractions for atomization ratio 60/10. The most important conclusion obtained from the analysis of this figure is that the initial velocity of particles is not only the function of a diameter but



Fig. 8. Local particle size distribution at the distance of 0.2 m from the atomizer.



Fig. 9. Local particle size distribution at the distance of 0.5 m from the atomizer.

is also the function of the distance of the axis. Velocity of a given fraction is not constant at the start and changes (for instance from 100 m/s at the edge of spray envelope, Fig. 11). If we assume that particles start travelling under a random atomization angle, such velocity distribution promotes intensive mixing of a spray and results in uniform particle size distribution, which explains effects, presented in Figs. 8-10. from the atomizer. Average linear velocity of the drying agent is also shown in the figures. At the distance of 0.2 m from the atomizer a significant differentiation of particle velocities from 1 to 14 m/s is observed. It was found that particles accumulated along the axis and in its vicinity have much higher velocities than these on the spray edge; the larger is the diameter, the higher is the velocity. At the distance of 3.5 m from the atomizer the particles have almost the same velocities close to mean linear gas velocity in the tunnel.

Figs. 12 and 13 show the distribution of axial velocity of different droplets encountered at a distance of 0.2 and 3.5 m



Fig. 10. Local particle size distribution at the distance of 3.5 m from the atomizer.

Air 30 kg/h, 220°C, 1 m/s



Initial velocity; air 60kg/h, feed 10kg/h





Distance from the atomizer 0.2 m Air 30 kg/h, feed 5 kg/h, 200°C, 1m/s

Fig. 12. Distribution of axial velocity at the distance of $0.2\,\mathrm{m}$ from the atomizer.

Distance from the atomizer 3.5 m Air 30 kg/h, feed 5 kg/h, 220°C, 1 m/s



Fig. 13. Distribution of axial velocity at the distance of 3.5 m from the atomizer.

5. Drying kinetics

First in the literature attempt was made to determine full trajectory of spray drying kinetics in a pilot-plant scale tower.

Application of the PDA technique enabled us to determine the relation between time and length scale for drying of maltodextrin and as consequence to find drying kinetics and residence time of particles in the tunnel. In the first step the relation of weighted velocity of particular fractions as a function of the distance from the atomizer was found. Integration of particle velocity along the length of the column allowed us to determine an average velocity of the spray and time necessary to cover a given distance from the atomizer.



Fig. 14. Drying kinetics of maltodextrin.



Distance from the atomizer 5.5 m

Fig. 15. Average residence time for particular fractions.

Having this information we recalculated the length scale to time scale (Fig. 5) and present changes of moisture changes of moisture content versus time (Fig. 14).

Fig. 15 displays average residence time of particular fractions. Particle residence time in the column is always shorter than the average drying agent residence time due to high initial particle velocity. However, there is not simple relation between changes of gas and particle residence time.

A significant differentiation of the initial velocity of particular fractions, Fig. 11, does not affect the residence time of fractions in the column. A rapid deceleration of particle velocity in the initial stage of the process makes residence time similar for all fractions (from 1.7 to 2.4 s).

6. Conclusions

A spray drying tower which was designed, built and tested allows to perform a profound analysis of disperse and continuous phase parameters. Sophisticated measuring techniques were applied and developed to determine spray to determine spray structure (PDA) and gas temperature (microseparator).

The tower capacity enables the identification of the effect of various drying process parameters like the effect of feed properties, feed rate and feed temperature, drying agent temperatures, air flow rate on drying and degradation kinetics.

The analysis of the results showed that particle size distribution became uniform due to spray dispersion during drying. No aerodynamic segregation of particles was found.

The analysis of initial velocity of the particles showed that it was not only a function of diameter but also a function of the distance from the axis of the spray. Particles injected into the continuous phase under random angles promote self-mixing of a spray resulting in a uniform particle size distribution. The concentration of a small fraction increases during drying process, while the concentration of bigger fractions decreases. These results confirm our understanding and reflect the run of drying process.

Full trajectory of spray drying kinetics has been presented for the first time in the literature.

Application of PDA measuring system enabled us to prove that residence time for all fractions is similar and shorter than drying agent residence time. There is no simple relation between changes of gas and particle residence time. Drying time is mostly affected by air flow rate and atomization ratio.

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