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Analysis of the mechanism of counter-current spray drying

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Abstract Results of experimental investigations of the effect of drying and atomization parameters on counter-current spray drying are discussed. Based on 96 experimental tests, the local and global distributions of velocity, temperature, drying air humidity and moisture content of material dried in the drying tower were determined. Analysis of the results showed that the process of agglomeration during counter-current spray drying depended mainly on air temperature in the atomization zone.

Keywords Particle size distribution · Agglomeration · Atomization zone · Discrete and continuous phase recirculation · LDA · PDA

1 Introduction

Spray drying permits a significant extension of the contact area of heat and mass transfer, which can reduce the time of drying to a few seconds and makes this technique applicable to dewatering porous materials, such as many food and pharmaceutical products (Masters 1985).

Spray dryers of different constructions are used in industry. In most cases drying takes place in a concurrent system. Only ca. 5% of spray dryers operate in the counter-current system (Rahse and Dicoi 2001). The process with a counter-current flow of material and drying agent has many advantages, the most important being the reduction of energy input for evaporation and integration of several unit operations in one apparatus, e.g. drying, agglomeration, segregation. However, due to complicated flow hydrodynamics of both phases (recirculation of the continuous phase and agglomeration of particles), drying in the counter-current system is a poorly understood process.

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At the Faculty of Process and Environmental Engineering, Lodz Technical University, a drying tower equipped with advanced measuring systems was constructed. These systems permit complex studies on momentum, heat and mass transfer during concurrent and counter-current spray drying (Zbicinski and Piatkowski 2004).

Extensive experimental investigation of spray drying in the counter-current system was performed within this research project.

2 Experimental set-up

A diagram of the experimental set-up is shown in Fig. 1. The main element is a drying tower made of stainless steel, 0.5 m in diameter and 9 m high.

In the experiments a drying agent was hot air aspirated from the atmosphere through a filter. The air was forced into a heater of total power 73.5 kW. The air heated to the required temperature flowed into a distributor (Fig. 2) (Strumillo et al. 2001) equipped with 12 blades. A special mechanism enabled a smooth change of blade position. The slit between the blades could be adjusted in the range 0–10 mm. Depending on the amount and temperature of forced air, inlet tangential velocities ranged from 7 to 57 m/s, which corresponded to mean linear velocities in the column from 0.4 to 0.8 m/s.

Behind the dryer outlet there were 2 cyclones and a bag filter. Purified air was removed to the atmosphere.

Experiments were conducted on a water solution of maltodextrin of initial mass concentration 50%. The solution was prepared in a 150 l tank. The tank was heated with steam supplied to the heating jacket. The solution was supplied from the tank to the atomizer by a mono-pump. It was pumped to a pipeline made from stainless steel and equipped with a heating jacket. A pneumatic nozzle (Spraying System) with a thermostatic water jacket was used to spray the solution.

The spraying nozzle could be located at different distances from the drying air inlet. Tests were made for three distances of the nozzle from the air inlet: 2.4, 4.7 and 6.7 m.

After heating the air to the required inlet temperature and stabilization of thermal conditions in the whole set-up, material spraying was commenced.

Dried (agglomerated) particles of large mass and diameter fell down to the lower cyclone. Small particles that were not agglomerated were entrained by the air and received in the cyclones and bag filter.

During the drying process a number of tests were performed to determine drying kinetics in counter-current spray dryers. The temperature of continuous and disperse phase and air humidity in the dryer were tested on-line. Commercial Dantec systems (Particle Dynamics Analyzer (PDA) and Laser Doppler Anemometry (LDA)), were used to determine the distribution of particle diameters and velocities (Zbicinski and Piatkowski 2004). Samples of material were taken to determine changes in moisture content along the drying path (Delag 2002).

Extremely important drying parameters are the temperatures of material being dried and the drying medium. In the spray dryer, material particles appear as a disperse phase which is in close interaction with the continuous phase. The use of classical thermocouples allows us to measure the approximate temperature of the mixture of particles and drying air. The approximation is a result of the fact that particles moving in an air stream collide with the thermocouple and deposit on it.

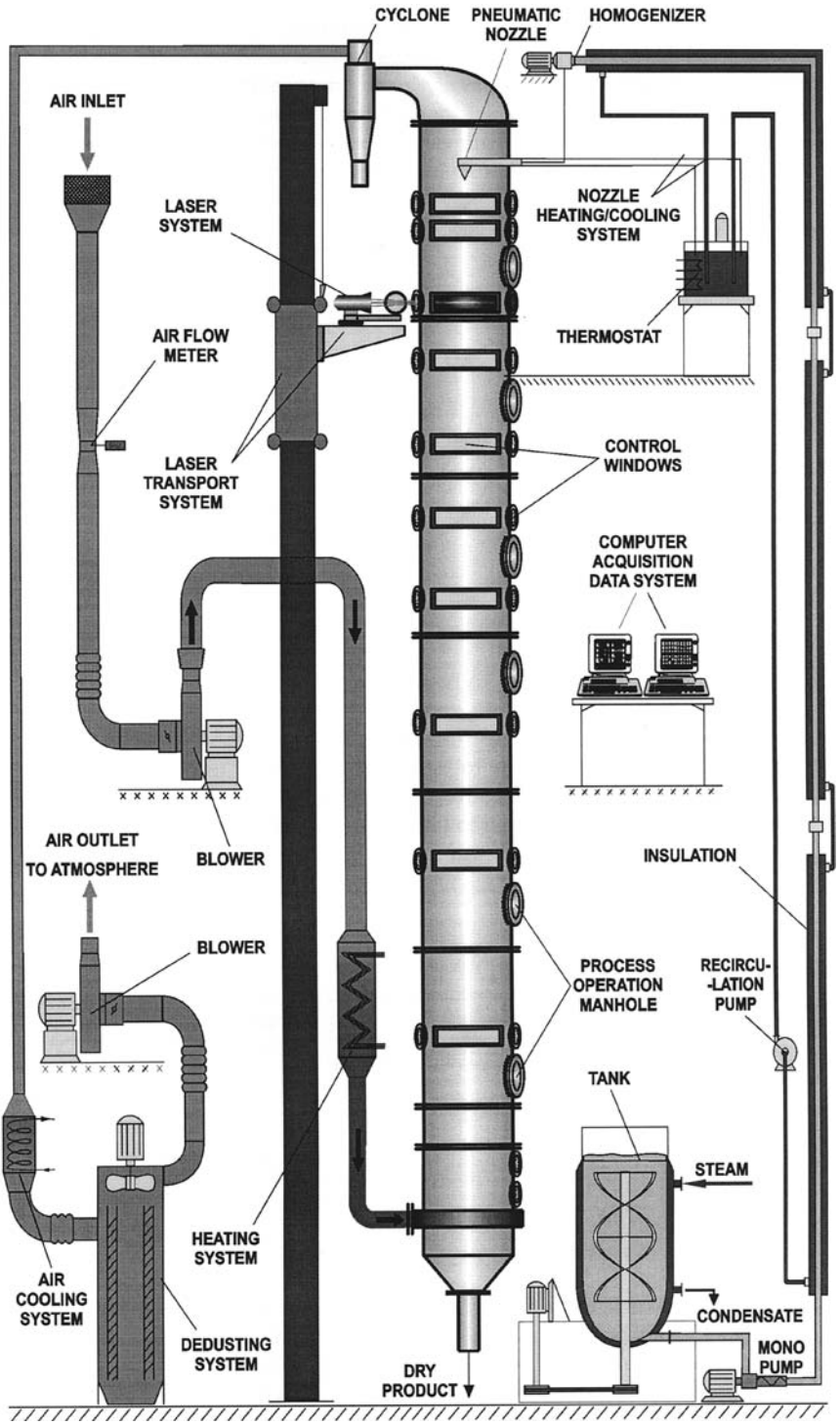


Fig. 1 Experimental set-up

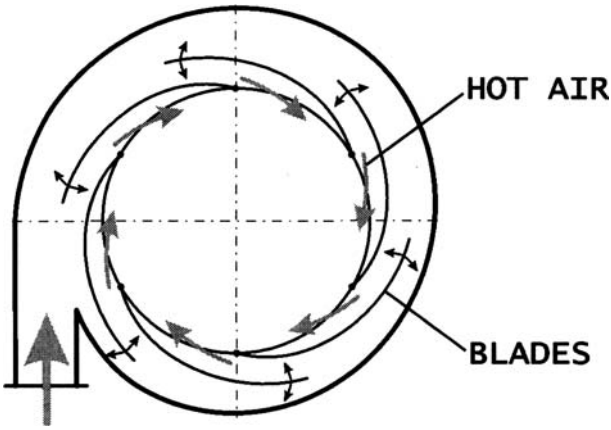


Fig. 2 Schematic of air inlet

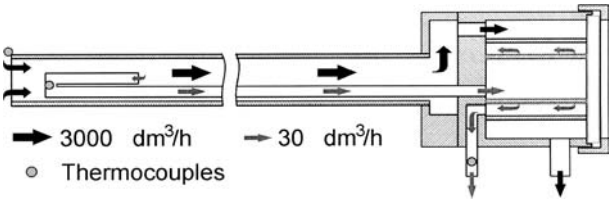


Fig. 3 Schematic of the microseparator

Many methods were tried to eliminate errors that appear as a consequence of this phenomenon. The most frequently applied method was to use a shielded thermocouple. A partly shielded thermoelement enables determination of the continuous phase temperature with satisfactory accuracy (Papadakis and King 1989; Zbicinski 1995). However this method cannot be applied in the counter-current drying system because dried material particles move in all directions in this process.

In our tests we applied a dynamic method of temperature measurement in the continuous phase, in which inertia forces were used to separate the disperse phase. The idea of this method, called "microseparator", was given by Kievet and Kerkhof (1996). The name of the method is related to the phenomenon of aerodynamic separation of particles from the air.

The solution proposed by Kievet and Kerkhof has been improved in our project. Fig. 3 shows a diagram of the device and the principle of operation.

The outer pipe of greater diameter is used to aspirate the mixture of drying air and dried particles from the dryer. In the pipe, the air and particles flow at a significant rate (ca. 3.5 m/s) in the pipe. A pipe of smaller diameter, bent at an angle of 180° is placed inside the outer pipe. This pipe is used to aspirate the air at velocity ca. 1 m/s. Due to flow rate difference in both pipes and the opposite flow direction, the small pipe aspirates air that is almost completely devoid of dried material particles. The continuous phase temperature was measured using a thermocouple placed inside the small pipe. Additionally, a thermocouple was installed at the end of the larger diameter pipe to measure the jet temperature.

Separation of the continuous and disperse phase allowed us to measure one more important process parameter—moisture content of the continuous phase.

A precision S4000 Michell dew-point thermometer (accuracy 0.01°C) was used for air humidity measurements. After the particles were removed, the air was supplied to a hygrometer by a heated Teflon pipe 5 m long. Construction of the microseparator enabled free displacement of the device and the performance of tests on 12 measuring levels. On each level the temperature and humidity were determined at 7 points along the dryer radius.

3 Scope of investigations

The full scope of the investigations covered many aspects of spray drying kinetics in counter-current systems. In this paper we focus on the effect of process parameters on the degree of particle agglomeration in the drying tower.

4 Analysis of results

A laser anemometer (LDA) was used to measure local velocity distributions of dried material particles. Disperse phase velocity was determined in the tower axis and at 6 points located on the dryer radius. Fig. 4 shows the directions of particles in the dryer.

Particles and droplets of dried material were displaced with drying air axially, tangentially and radially. Lighter particles were entrained upwards to the atomization zone where they encountered the stream of sprayed material. As a result of contact between particles (droplets), agglomerates were formed, which dropped down the column and contacted the drying agent of growing temperature. Mass loss due to evaporation might cause the agglomerate to be raised again by the drying air to the atomization zone where it was further agglomerated.

The process of agglomeration proceeded until the moment when the force of gravity on the agglomerated particle was greater than the forces of aerodynamic resistance of the drying agent. The particle then fell below the air inlet to the lower receiver.

Diameters of dried material particles were measured by a PDA laser measuring system below the atomizer and one level above it, nearest to the air outlet from the dryer.

The laser system provided information on the diameters of several thousand particles from each measuring point. The data were sorted and divided into ranges, every 10 μm. On the basis of the number of particles in particular fractions, their percentage was calculated.

Figure 5a–f show local particle size distribution in such drying conditions in which final particle diameters were the biggest. The applied PDA configuration enabled data acquisition for particles of diameters up to 1000 μm. Since the fractions of particles with diameters greater than 700 μm did not exceed 0.2%, the range of diameters shown on the graphs was limited to 0–700 μm. Analysis of Fig. 5a leads to the conclusion that the diameter distributions are most unified above the atomizer. In this part of the column, there were particles entrained with the drying air whose diameters were relatively small with moisture content not exceeding 5%.

The diagram shown in Fig. 5b (distance from the air inlet 4.6 m) shows particle size distribution at a distance of 0.1 m from the atomizer. In the dryer axis and at a

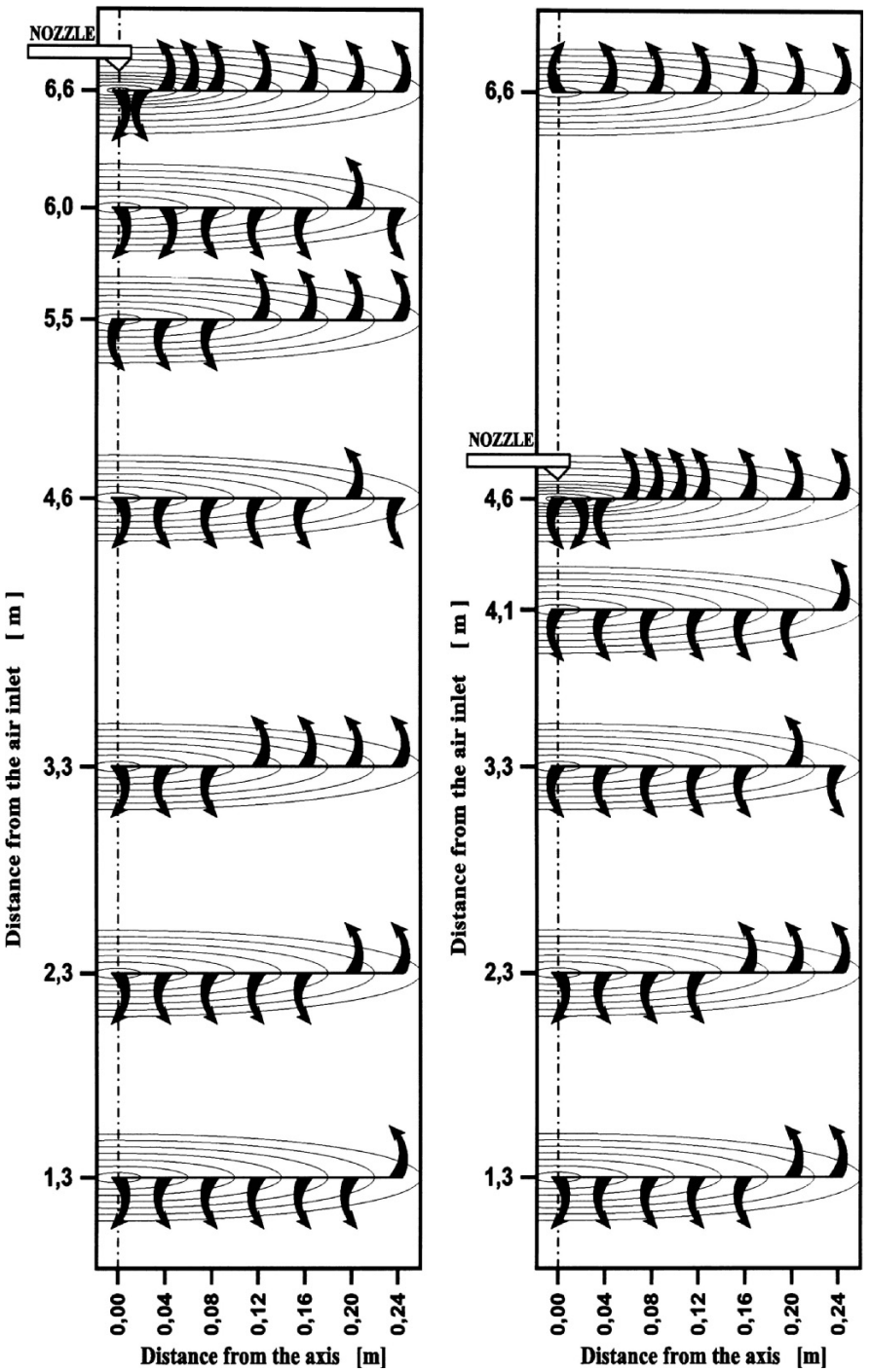


Fig. 4 Directions of particles in the dryer (air 300 Nm^3/h, temperature 150°C, slit between blades 10 mm, solution 6 kg/h, atomizing air 1.8 kg/h, nozzle 4.7 and 6.7 m)

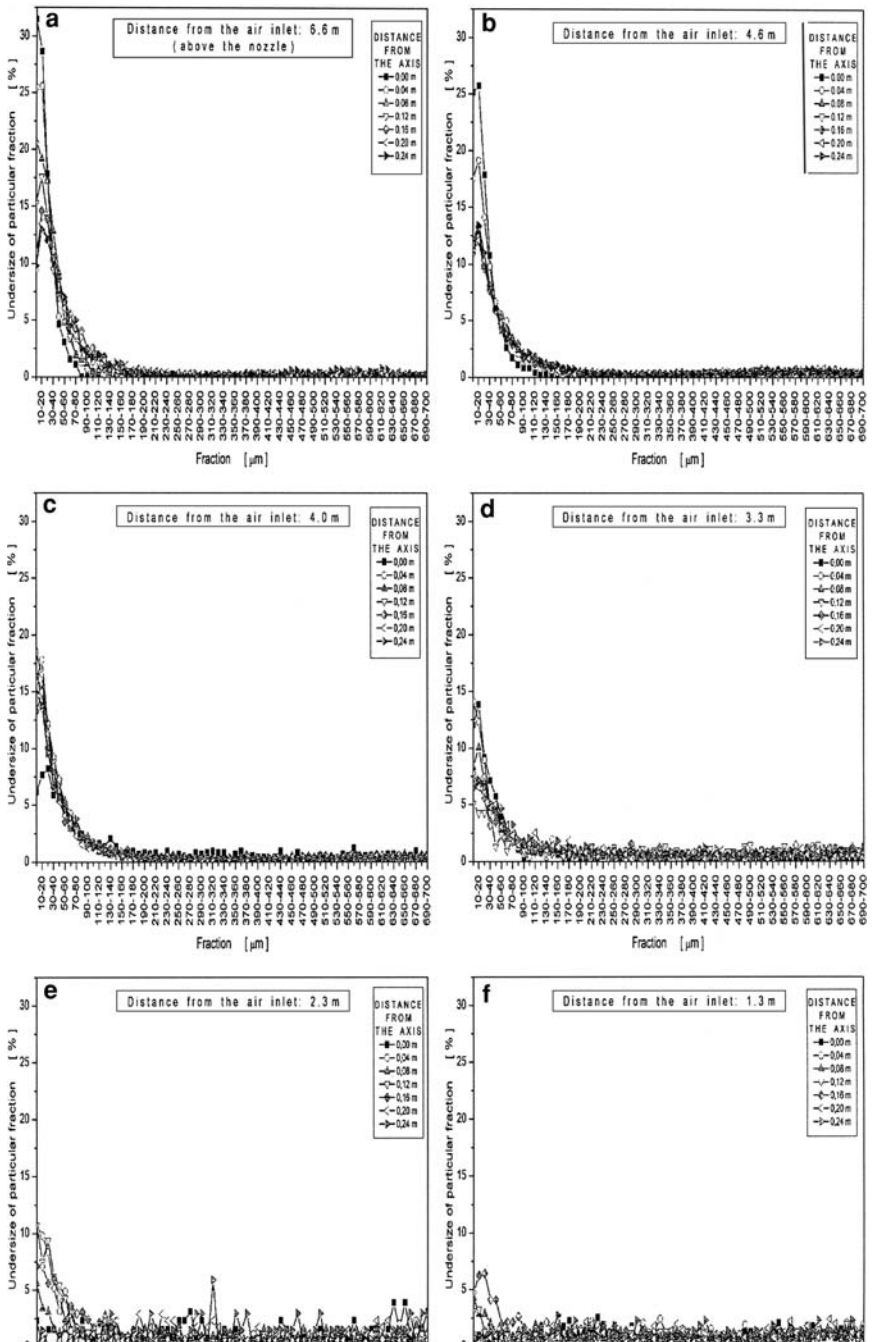


Fig. 5 Fractions of particles (air 300 Nm^3/h, 150°C; slit between blades 10 mm, material 6 kg/h, atomizing air 1.8 kg/h, distance of the nozzle from the inlet 4.7 m)

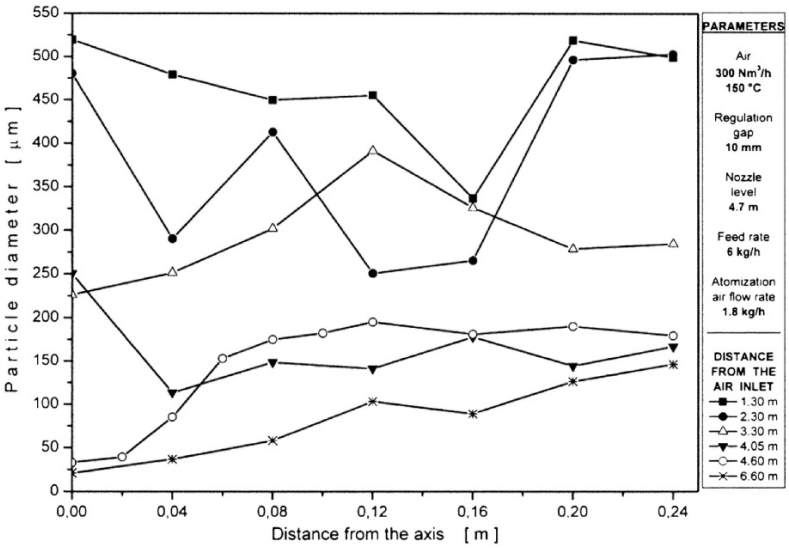


Fig. 6 Cross-sectional distributions of particle diameters

distance 0.04 m from the axis, where a compact jet of sprayed material is observed, the percentage of particle fractions with the smallest diameters reached 26%. At distance 0.24 m from the axis, the percentage of the smallest fractions did not exceed 15% and particles of diameters exceeding 400 μm appeared at the same time; this is caused by recirculation of the drying air stream and entrainment of single particle agglomerates.

On analyzing Fig. 5c–f, one can observe significant changes in particle diameter distribution during drying. With increasing distance from the atomizer, the percentage of particles with diameters exceeding 150 μm increases and those with diameters up to 50 μm decreases. There was a uniform distribution of particle diameters at a distance 1.3 m from the air inlet.

In order to better illustrate the above results, Fig. 6 shows mean arithmetic particle diameters at successive measuring points.

In Fig. 6 one can observe changes of particle diameters in the drying tower cross section. Above the atomizer (6.6 m from the air inlet) particle diameters are distributed along the column radius, from the smallest one in the column axis to the biggest near the wall. This distribution is a result of entrainment of the particles from the atomization zone (small particles in the axis) and aerodynamic segregation (bigger particles near the wall).

It can also be observed that at distance 0.1 m below the atomizer (4.6 m from the air inlet) at a width 0.04 m from the axis the sprayed material stream was still flowing. Particles entrained by the air flowing from the lower part of the column appeared further from the axis.

Subsequent curves show how particle diameter changed with increasing distance from the atomizer. Near the air inlet the mean particle diameter was the greatest, reaching ca. 520 μm.

Irregular changes of particle diameters along the radius at the distance smaller than 3.3 m from the air inlet (Fig. 6) are a result of particle recirculation in the column.

Figures 7–9 show changes of particle diameters as a function of distance from the air inlet for 24 drying conditions. It was found that the final agglomerate size depended on process condition and changed over a broad range.

Analysis of Figs. 7–9 shows that the biggest influence on particle diameters was the atomizer position. When it was changed, the temperature of air in direct contact with atomized particles also changed. This means that the process of agglomeration takes place mainly in the atomization zone, i.e. in the area 1–2 m below the atomizer.

It follows from analysis of Fig. 7 that higher gas temperature in the atomization zone hampered agglomeration and final particle diameters in these conditions did not exceed 150 μm in most cases. Poor agglomeration of particles was a result of rapid moisture evaporation and a lack of sufficient number of wet particles that were most susceptible to agglomeration.

Displacement of the atomizer from the distance of 2.4 to 4.7 m from the air inlet (Fig. 8) caused a decrease of air temperature in the atomization zone by about 10°C (Fig. 10). In these conditions the agglomeration process was efficient and mean particle diameters varied from 250 to 450 μm.

Location of the atomizer 6.7 m from the air inlet caused a further gas temperature drop in the atomization zone (Fig. 10). A consequence of the temperature drop in the agglomeration zone was a change of product particle diameter distribution. Agglomerates obtained in these conditions had mean diameters up to 450 μm; the smallest particle diameters obtained in this location of the atomizer did not exceed 150 μm (Fig. 9).

Figure 11a–f show the structure of particles obtained in different positions of the atomizer. The photographs presented were taken with a Hitachi S-3000N electron microscope. Photographs in the left column show material from the bottom collector, while the right one shows photographs of powder separated in the cyclones. Magnification in the right and left column differs five times.

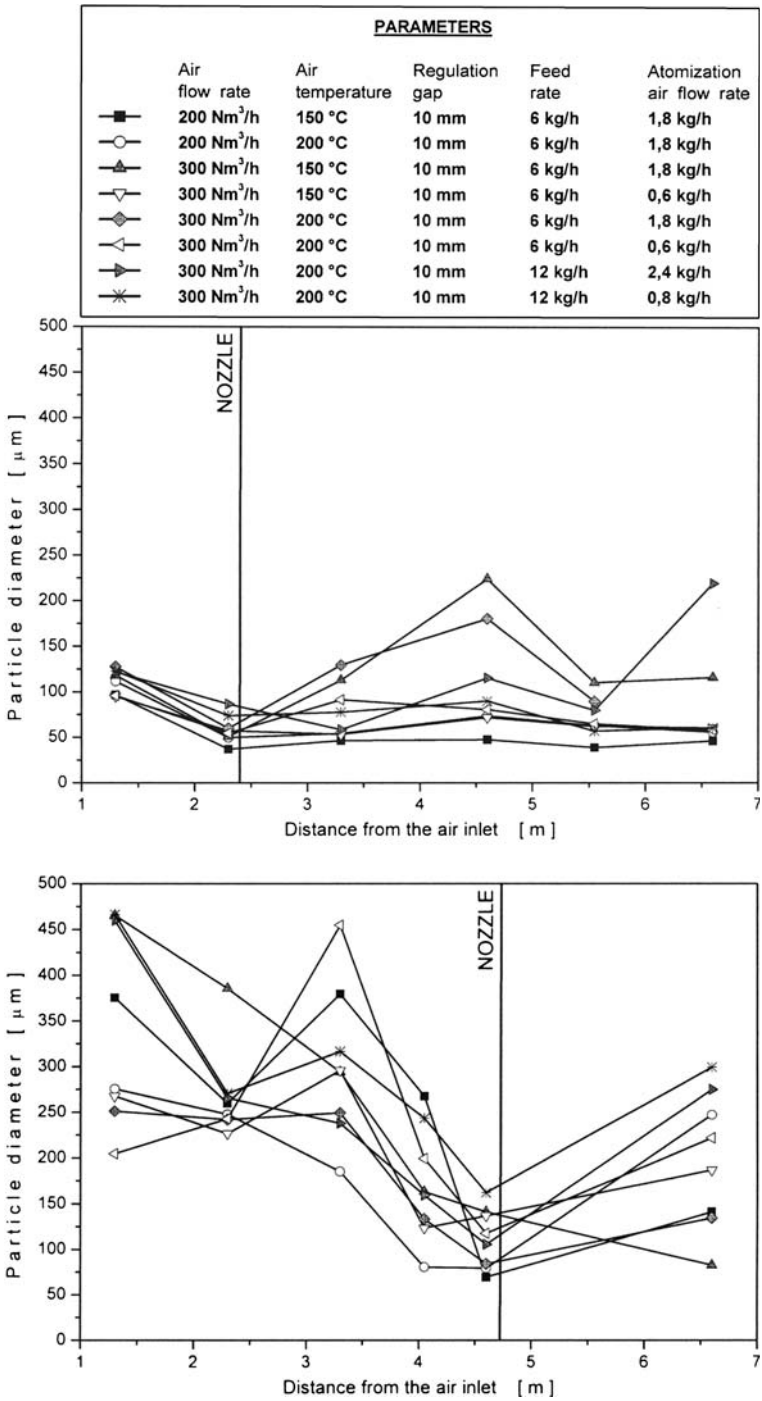
In the present drying conditions, particles obtained from the cyclones had similar shapes and diameters. These were non-agglomerated particles of diameters equal to dozens of micrometers.

Powders collected in the lower receiver were agglomerates and as such had a greatly differentiated external structure. Drying processes carried out with the atomizer installed at the level of 2.4 m caused formation of agglomerates which had the structure of combined spheres. This was a result of the previously described intensive mass transfer induced by high temperature in the agglomeration zone.

Particles obtained during spraying from the atomizer located 6.7 m from the inlet can be characterized as particles of uniform structure and large diameters. Lower gas temperature at the atomizer level caused the particles to form large homogeneous structures.

As was indicated earlier, location of the atomizer on the intermediate level, i.e. at a distance of 4.7 m, resulted in the formation of agglomerates with the greatest diameters. Figure 11c shows material particles obtained for the position of the atomizer. Their structure reveals that during drying moisture was partly removed from the particles that then got into contact, which led to the formation of extensive spatial structures.

Summarizing the results obtained, we can conclude that optimum agglomeration conditions can be determined for every process parameter of counter-current drying.



Figs. 7 and 8 Particle diameter distribution along the drying tower

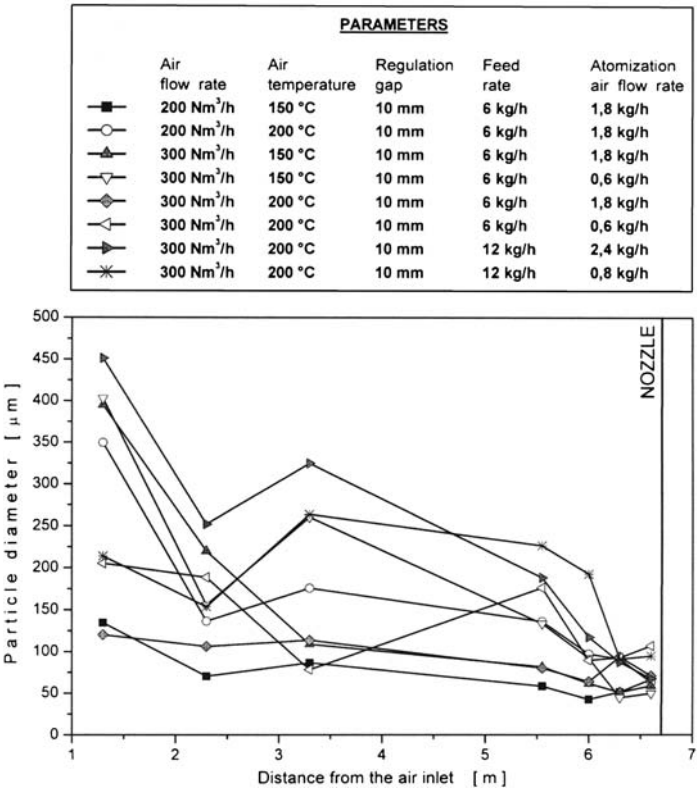


Fig. 9 Particle diameter distribution along the drying tower

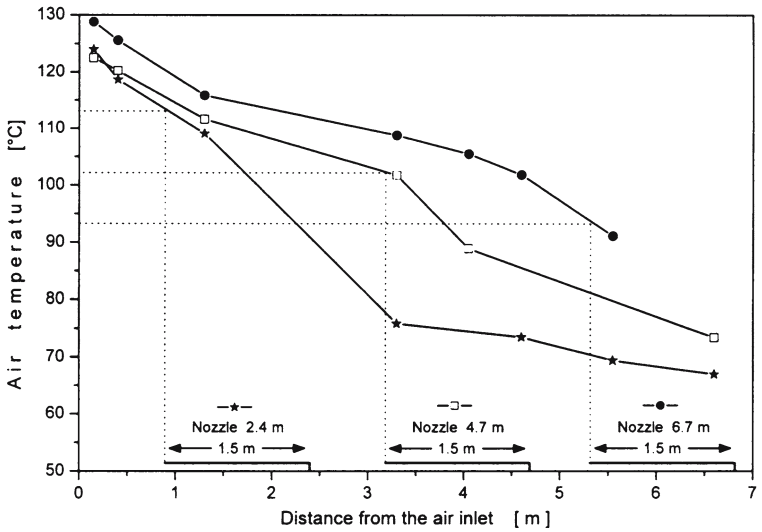


Fig. 10 Drying air temperature (Drying air 300 Nm^3/h, Temperature 150°C, Slit between blades 10 mm, Material 6 kg/h, Atomizing air 1.8 kg/h)

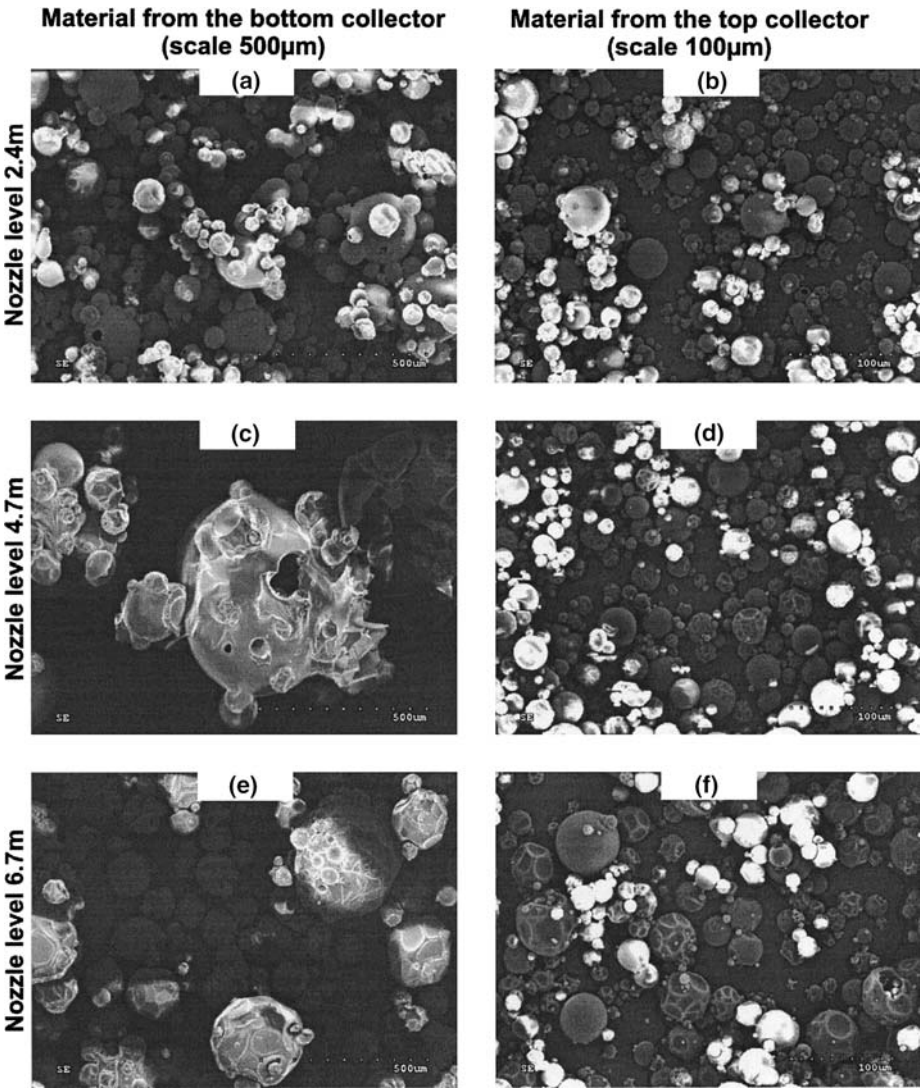


Fig. 11 Photographs of dried material particles

5 Conclusions

1. Agglomeration during counter-current spray drying depends on drying air temperature in the atomization zone.
2. For every atomization parameter optimum drying conditions, i.e. such in which agglomeration process is most efficient, can be determined.
3. By proper choice of drying and atomization parameters we can control diameters of the agglomerates obtained in counter-current spray drying.

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