USING AN ACOUSTIC LEVITATOR TO INVESTIGATE THE DRYING KINETICS AND SOLIDS FORMING PROCESS OF INDIVIDUAL DROPLETS DURING SPRAY DRYING

KEY WORDS: ACOUSTIC LEVITATOR, DRYING KINETICS

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Abstract

Spray drying is a widely used process to turn slurries into dry powders and is especially important for thermally-sensitive materials, that are often found in the food or pharmaceutical industry.

However, detailed insight into the drying kinetics during spray drying is difficult to investigate due to the boundary conditions in a spray drying tower. As a result, there is a lack of important information on the drying process and subsequent solidification of individual droplets.

In this context, an experimental setup for a droplet positioned in a stationary ultrasonic field of an acoustic levitator was designed to enable a non-contacting measurement of the drying kinetics and the subsequent solidification process. To generate a comparable situation like in a real spray drying process, the droplet is positioned in an airflow, where air temperature, humidity and velocity can be adjusted over wide range. Using an infrared camera to measure the surface temperature and a CMOS camera for object recognition, the droplet can be observed continuously and drying kinetics of the droplet can be determined from the measured surface temperature and decreasing droplet size.

Introduction

In food industry spray drying processes are widely used to produce dry free flowing powder starting from a liquid formulation. Since this is a one stage process with high drying capacity it has advantages compared to multi stage processes.

In order to generate large surface areas for fast drying, the slurry is disintegrated in very small droplets which are sprayed into a hot gas stream using nozzles. During the free falling period of the droplets there is a heat and mass exchange between the droplets and hot drying gas. The drying velocity is depending on the air conditions (Temperature und rel. humidity) and on the

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size and drying behaviour of the product droplets. The latter is mostly responsibly for the length of the spray tower.

Although the drying kinetics of a product is crucial parameter in this process, the experimental setup to measure the drying kinetics in an environment which comes close to the drying situation in a real spry dryer, is very difficult to establish.

In the recent years, acoustic levitation of single droplets has been developed to settle this problem. So shows (Eberhardt 1999) the development of an ultrasonic levitator apparatus. Also in publication from (Yarin et al. 1998), (Kastner 2001), (Schiffter 2006) or (Zaitone 2009) the Levitator assembly are described. The thermographic observation of levitated droplets was described by (Tuckermann et al. 2005) and (Wulsten und Lee 2008).

Materials and Methods

An experimental setup for a droplet positioned in a stationary ultrasonic field of an acoustic levitator was designed to enable a non-contacting measurement of the drying kinetics and the subsequent solidification process (Figure 1).

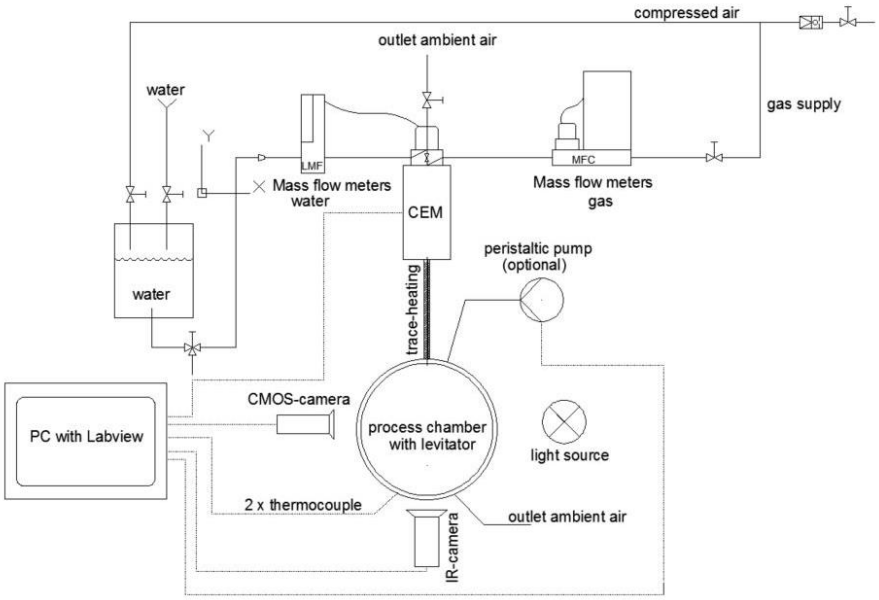

Figure 1: Experimental Setup ultrasonic levitator

The ultrasonic levitator is comprised of an ultrasonic transducer and reflector. If US-frequency and distance between transducer and reflector are set correctly, a stationary wave is established. The droplet can be positioned in one of the power nodes and is kept in place by the US-field.

To generate a comparable situation like in a real spray drying process, the reflector has been constructed as a nozzle which is used to establish an airflow towards the droplet (Figure 2). The air temperature, humidity and velocity can be adjusted over wide range.

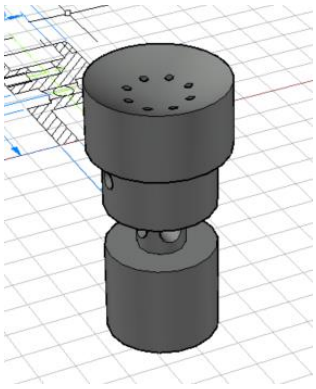

Figure 2: US reflector modified as nozzle

Using an infrared camera to measure the surface temperature and a CMOS-camera for object recognition, the droplet can be observed continuously. The data from the CMOS-camera are used to determine the size of the droplet from which the volume and mass can be derived. The drying velocity in the constant rate period can be assumed by the experimentally observed loss of mass in a given time range. During that period (constant rate period: CRP) the surface temperature of the droplet remains at the wet bulb temperature. All the energy that is transferred from the hot gas stream to the droplet is used for evaporation. As soon as the droplet stops shrinking a rise in surface temperature can be observed and is detected by the infrared sensitive camera (Figure 3).

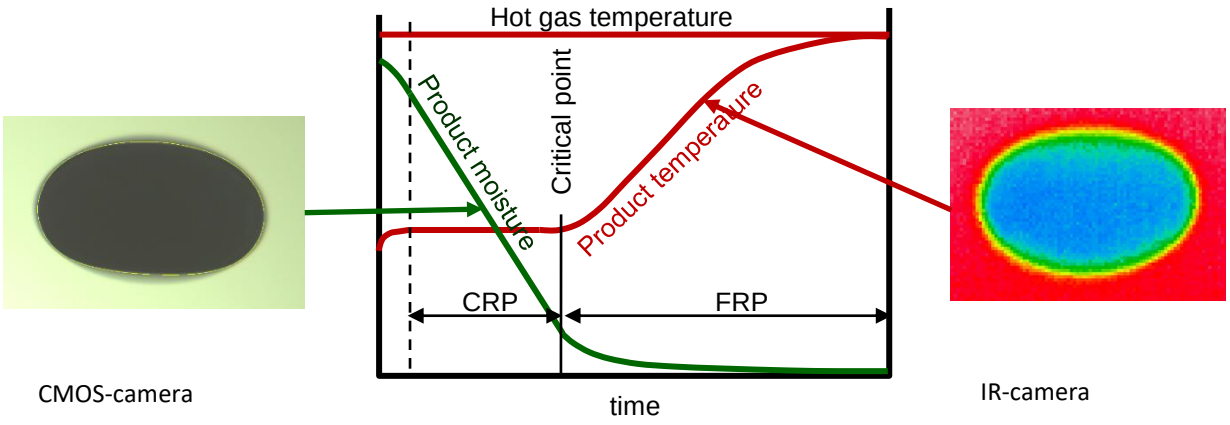

Figure 3: Determination of the drying kinetics from combined measurement of the volume reduction (moisture content) and the surface temperature of the product

During this period (falling rate period: FRP) the heat flow from the gas to the particle stays constant but only a part of the energy is still used for drying. The rest is heating up the droplet. From the slope of the temperature rise and the thermodynamic properties of the slurry the drying velocity in the FRP can be calculated.
The drying air is controlled in terms of temperature, relative humidity and velocity. Thus the complete characteristic drying kinetics of a single droplet in dependence of the major boundary conditions in a spray dryer can be measured.
To start the experiment, the complete setup is heated up to the desired temperature and the droplet is positioned in one of the nodes, using a micro-syringe. After the experiment the resulting particle is removed from the apparatus for further investigation (final moisture content, strength etc.)
The product system under investigation was a titanium dioxide (TiO_2) suspension. TiO_2 was supplied by Merck KGaA (CAS-No. 13463-67-7). The primary particle size distribution (PSD) shows Figure 4. It is obvious that the PSD is a bi-modal distribution, where the bigger particles have an average size of 45 µm and the smaller fraction has an average size of 2,5°µm.

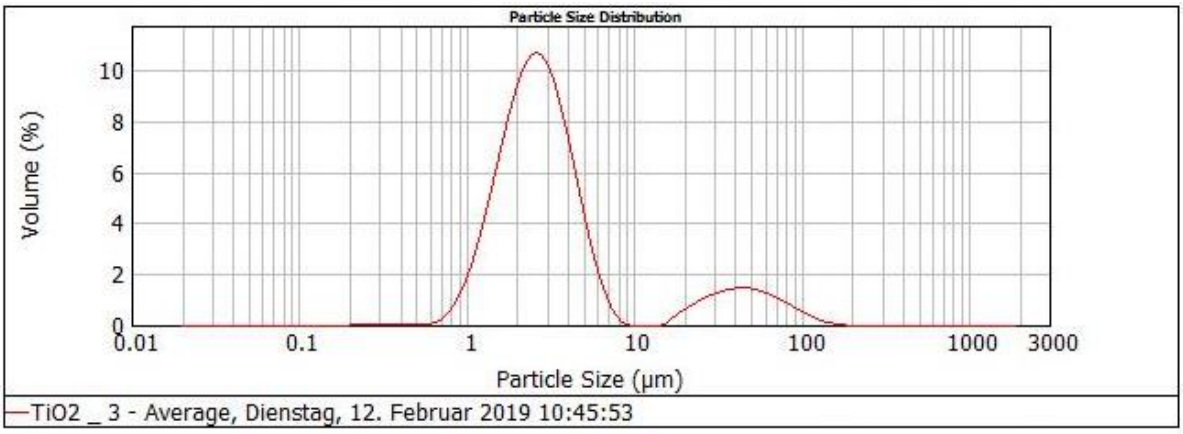

Figure 4: Particle size distribution TiO_2-Suspension

Results

TiO_2 was suspended into water with an initial water content of 90%. The suspension was positioned in a node of the ultrasonic field. Here it displayed a very stable levitation behaviour. The resulting TiO_2 particle had an almost perfect spherical shape.

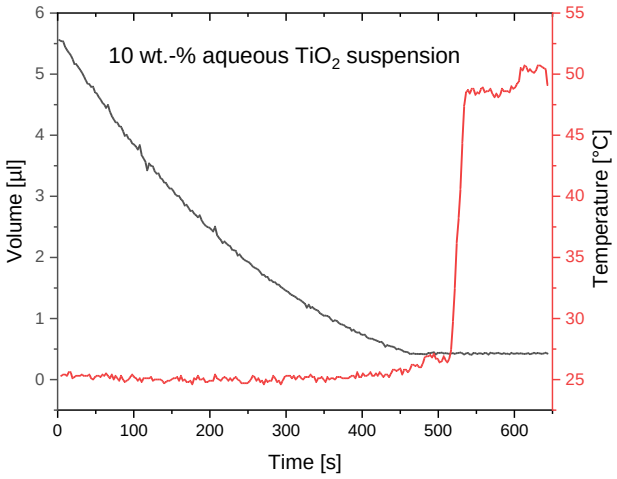

Figure 5: Results for a TiO_2 suspension at an air temperature of 50°C and an air velocity of 1 m/s

Figure 5 shows a typical result for a drying experiment. Here the surface temperature and the volume of droplet are displayed as a function of time. The volume of the droplet is reducing continuously the drying during the constant rate period, while the surface temperature stays constant. In the case of the used TiO_2 suspension ideal shrinkage can be observed. The critical point where the constant rate period is switching to the falling rate can easily be detected. Here the shrinkage comes to a stop, because the particle has reached its final size while the temperature starts to rise. Finally, the droplet reaches the surrounding air temperature and the drying experiment is stopped.

The removed particle (figure 6) shows a spherical shape.

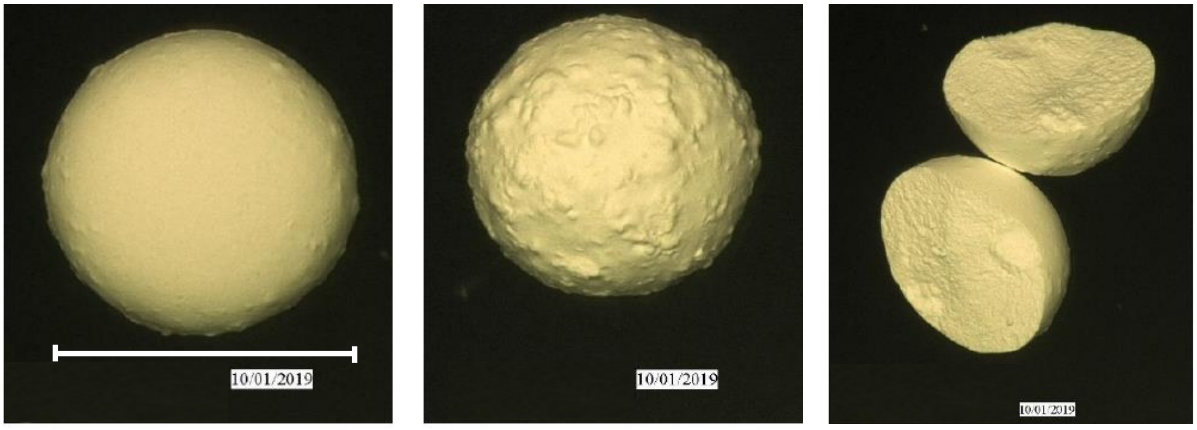

Figure 6: Dried TiO_2 particle a) top view, b) bottom view, c) cross section

Discussion

Figure 5 exemplifies that the drying of a 10 wt.-% aqueous TiO_2 suspension occurs mainly in the CRP. The loss of size occurs continuously during the drying process and the shrinkage of the droplet stops immediately when the particle solidifies. A time gap of ~130 s can be observed between the end of the shrinkage and the onset of the temperature rise. This indicates an internal structure with broad capillaries which results in a fast moisture transport to the particles surface. In this case, the particle stays in the CRP regime.

The steep slope of the temperature rise in the FRP regime indicates that no water content is left inside dried particle. Only quite short FRP regimes are detected.

Figure 6 shows microscopy images (VHX-1000, Keyence, Japan) of obtained dried particles. The surface of the resulting particles in Figure 6 a and b show different topographies. This result may be traced back to the bi-modality of the PSD and a simultaneous sedimentation process during the drying. Larger particles sediment faster to the bottom of the droplet, resulting in an altered particle surface.

Conclusion

In summary, the implementation of an acoustic levitator for drying kinetics experiments of individual droplets has been shown. First experiments prove the applicability of the setup and yield results with good accuracy.

Using a 10 wt.-% aqueous TiO_2 micro particle suspension, the different drying phases of the drying process can be measured and identified. Additionally, details of the solidification process during the drying can be observed. Therefore, the acoustic levitator proves to be an effective and accurate tool to investigate the drying behaviour of individual droplets.

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