Particle formation via droplet drying: observing morphological evolution and measuring aerodynamic diameter

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RESUME

Dans le présent article nous étudions l'influence de la cinétique de séchage d'une gouttelette, sur les caractéristiques morphologiques des particules produites, cela dans le but de modéliser les propriétées aérodynamiques des aérosols ainsi formés.

Nous présentons le développent d'un nouveau dispositif permettant d'analyser en détail l'évaporation de gouttelettes, depuis leus production jusqu'a la formation d'une particule sèche, en passant par la nucléation des premiers cristaux. Cette expérience permet d'étudier l'évaporation des gouttes avec une résolution temporelle inférieure à la microseconde, et ainsi de détecter trés présisément, par analyse d'images, le dédut de la cristalisation. Nous présentons une première étude réalisée sur un sel inorganique et qui montre, suivant les conditions de séchage, une grande variété de morphologies de particules.

ABSTRACT

An investigation into the relationship between factors governing droplet drying and resultant particle morphologies, with a specific interest in the aerodynamic properties of dried particles.

This work describes a new Falling Droplet Column (FDC), which offers the capability to analyse in detail the entire evaporative lifetime of individual droplets, from generation to dry particle formation, with capability for sub-microsecond temporal resolution and subsequent offline analysis of dried particles by SEM. A comparison of evaporative profiles and resulting morphologies produced in a range of conditions for different inorganic salts is presented. We will explore the specific crystallisation events through detailed imaging of aerosol droplets.

KEYWORDS: spray drying, droplet crystallisation, evaporation kinetics, particle morphology

1. CONTEXT

Powder production via spray drying is pivotal in a range of applications including the food and pharmaceutical industries.[1] The motion and evaporation of droplets are coupled processes and the resulting morphology of a dry particle is dependent upon the drying process through which it is formed.[2] Similarly, in the nuclear industry, large amounts of radioactive materials are handled in the liquid phase. In an accident scenario this material can be aerosolised, producing droplets which dry rapidly forming solid particulates. Spray drying typically involves the heated production of aerosol droplets which then dry quickly. A jet formed from a clean break in a heated container may be considered similarly. Thus, the phenomena observed in spray drying and the underlying principles may be applied directly to particle formation from ruptured pressurised systems.

Measuring the evaporation and morphological evolution of individual droplets through this highly dynamic process presents significant technical challenges but is critical for particle engineering and the quantification and mitigation of risk from aerosolised material.

2. INTRODUCTION

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The parameters governing the droplet-drying process impact final dry particle morphology. This, in turn, determines the aerodynamic and transport properties of the dry particles. One way to understand the effect of evaporation rate on final morphology is to use a Péclet number.2 The Péclet number compares the evaporation rate of a droplet to the rate of diffusion of a solute. For values greater than one, the evaporation rate is dominant and surface enrichment is likely. In this case droplets have a propensity to form a skin or crust. For a Péclet number less than one the rate of diffusion is dominant and surface enrichment does not occur. There are a limited number of techniques to make measurements on single droplets during evaporation. Observation of droplets from droplet generation to dry particle formation is rarely achieved and instruments are often limited by temporal resolution or ability to collect dried particles for further analysis. Evaporative processes and morphological changes can occur in a matter of milliseconds under industrially relevant conditions. To probe the phenomena that occur in these systems a sub-millisecond temporal resolution is required.

3. EXPERIMENTAL

This work presents a Falling Droplet Column (FDC), shown in Figure 1, capable of analysing in detail the entire evaporative lifetime of droplets, with a sub-microsecond temporal resolution.[3] The instrument operates by establishing a chain of uniform falling droplets within a temperature and humidity-controlled environment. Stroboscopic imaging of droplets enables direct measurement of droplet diameter and calculation of aerodynamic diameter by measuring droplet settling velocity. Additional measurements of aerodynamic diameter for individual particles are achieved using multiple exposure imaging. Dry particles are deposited at the bottom of the FDC and imaged using SEM. This allows study of evaporative dynamics with comparison against final morphologies.

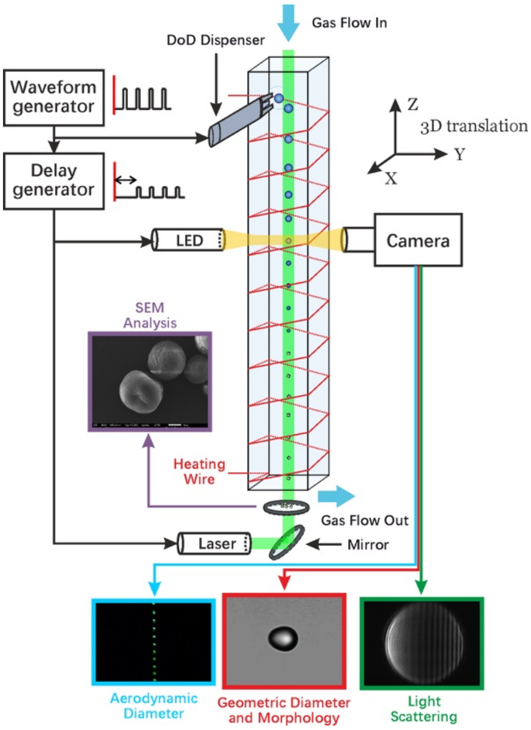

Figure 1. Schematic of the Falling Droplet Column, displaying key features and measurement capabilities.

4. RESULTS

In this talk we will compare evaporative profiles and resulting morphologies produced in a range of conditions for different inorganic salts. We will present detailed images of phase and morphological change such as crystallisation and particle deformation and corresponding variations in aerodynamic diameter, such as that shown in Figure 2.

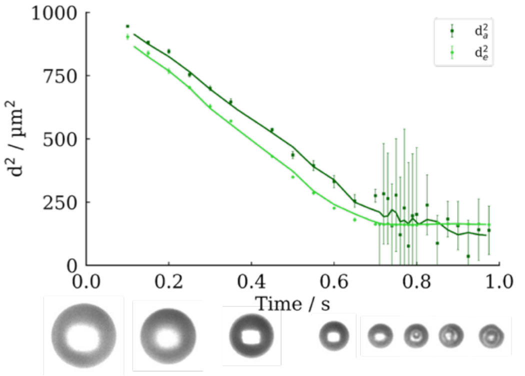

Figure 2. Sodium sulphate droplet evaporation (0.05 mass fraction) at 0% relative humidity and 296K. Surface solidification is observed at approximately 0.7 s, but solvent loss continues as sen in the deformation of the surface.

Though analysis of droplet morphology and parallel measurements of aerodynamic diameter and volume equivalent diameter we will explore solvent loss and particle deformation after solidification.

We will present a quantitative analysis of particle morphology, demonstrating the link between drying rate and final morphology as demonstrated in Figure 3.

We will also present new aerodynamic dimeter measurements of single particles, achieved through multiple exposure imaging, allowing the distribution of aerodynamic diameter of dried particles to be resolved.

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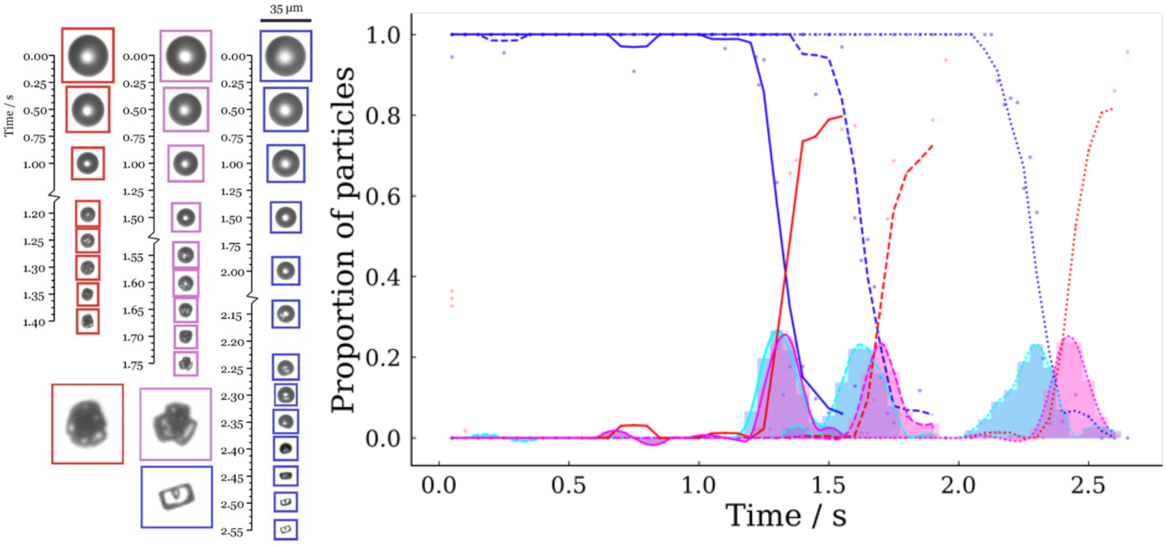

Figure 3 Left: Inflight images of the morphology of evaporating NaCl droplets in environments of 20% (red), 30% (purple) and 40% RH (blue) at 298K K. Enlarged images of final morphologies are inset. Right: The proportion of particles in liquid droplet state (dark blue) or final crystalline state (red) for NaCl droplets evaporating at 293 K in 20% RH (solid line), 30% RH (dashed line) and 40% RH (dotted line). The cyan and magenta curves represent the proportion of particles transitioning out of the liquid droplet state, and into the final crystalline state, respectively.

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