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Comparing different single milk droplet drying process with spray drying process

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ABSTRACT

Spray drying is currently the main method of industrial milk production because of its high drying speed and high process control accuracy. By controlling the parameters of spray drying process to control the characteristics of the milk powder is well researched. However, the drying mechanism in spray dryer is still not very clear. Methods of measuring single droplet drying kinetics are widely used instead of directly modelling spray drying kinetics. In this study, single droplet drying methods including sessile drying, filament hanging drying, and levitator drying in room temperature are used to compare and simulate the drying kinetics in spray dryer. Experiments show that the droplets in levitator are more spherical, but the final dry particles tend to be more donut-like due to the compression of the upper and bottom sides by the ultrasonic waves. In the filament hanging drying, the droplets are only suspended on the filament by friction. Due to gravity, the shape of the droplets and dried particles is non-sphere droplet shape. In sessile drying, the droplet is half-sphere shape, and the contact area is not changing because of capillary force. By comparison, the final particle morphologies of these are significantly different from those in the spray dryer, therefore, explore the characteristics of spray-dried droplets by monitoring the drying characteristics of single droplets may not suitable on some situation especially on multi-component materials.

1. Introduction

Milk is a highly nutritive, protein-rich food that is primarily acquired from a variety of animals, including cows, goats, sheep, buffaloes, camels, and mares, which constitute the mainstay of commercial milk production across the globe. As the first food consumed by a young mammal, milk has remained the only component of the human diet (Pereyra-Elías et al., 2022). Due to its benefits in lowering shipping and storage costs, a variety of dried milk products are produced. Spray drying is a common method for creating dry powders in the food and pharmaceutical sectors. They are less prone to microbial deterioration, resulting in high-quality goods.

Spray drying is defined as an atomized sprayer is used to apply slurry, which is then dried using a high-temperature air. It is widely utilised across a variety of sectors. For instance, it is used to make milk powder, coffee, and starch in the food business. The development of spray drying technology also gives the opportunity of producing food additives with better performance and longer shelf life. In pharmaceutical industry, it is used to produce antibiotics, micro-capsule. During

spray drying process, operator can control the process variables such hot air flow rate, input drying temperature, atomizer compress air flow rate, and liquid to atomised rate (Patel et al., 2014). It's challenging to collect samples and properly monitor changes occurring in each droplet during the intermediate drying stage since the drying of so many droplets are carried out in a spray drying chamber. These challenges make it more difficult to research the kinetics of drying in a spray drier. Online single droplet drying in spray dryer and temporal morphology monitoring, however, are challenging to achieve. As a result, offline single droplet drying techniques and other efficient approaches are frequently used to study single droplet drying dynamics. Single droplet drying is a well-researched technique that simulates the droplet convective drying process in spray drying by tracking the drying kinetics and morphological changes of an individual droplet in a controlled drying environment (Patel et al., 2014). These methods include sessile droplets, a syringe or a thin wire/glass filament hanging, sonic levitation, electromagnetic levitation (Usui et al., 2023) (Hyers, 2005). All these single droplet drying processes all have some limitation compared with the droplet drying process in spray dryer. Aiming to forecast the single

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Brownian Brownian Gravity Deegan

Fig. 1. Transport mechanisms happening inside a droplet sessile on a surface.

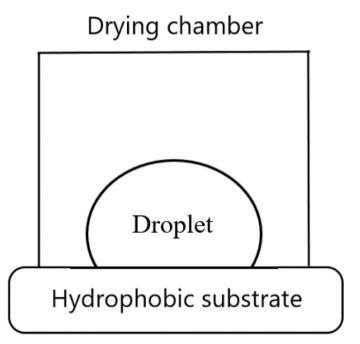


Fig. 2. Setup of Sessile drying process.

droplet drying rate, shape, shell formation, and temperature change curve, attempts have been undertaken in recent years to simulate the drying kinetics of single droplets on both insoluble and dissolved powders. The droplets in single droplet dying study will go through different drying processes compared with spray drying, including droplet size in millimeters instead of microns, drying time in minutes instead of seconds. Beyond this, the droplets do not experience droplet-droplet interactions, droplet-wet particle interactions, or droplet-wall collisions (Hyers, 2005). The deviation between these modelling prediction results is experiment from spray dryer exist mainly because they are based on above single droplet drying process with above limitation. This research is to look on the possibility of scaling up and simulation from the single droplet drying process to spray drying process.

2. Literature review

A variety of transport pathways for micro-particles occurring during the droplet's evaporation have been explored using the sessile droplet technic as shown in Fig. 1, a single droplet is placed on a hydrophobic

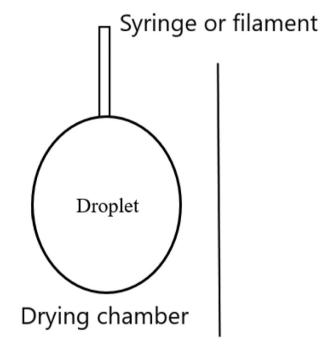


Fig. 3. Setup of Filament hanging drying process.

surface, which can be used to explain all movement in a droplet drying process. Deegan flow and Marangoni flow, also known as the Gibbs-Marangoni effect, which refers to the mass transfer across a fluid interface caused by a gradient of surface tension, compete inside the sessile droplet during evaporation. This phenomenon may be referred to as thermo-capillary convection when it is temperature dependent, and evaporative flux may occur (Fig. 1). There are studies on these flows in the literature (Majumder et al., 2012; Maki and Kumar, 2011; Sadek et al., 2015).

Different single droplet drying method is used to study the drying process including sessile drying, contact hanging drying, levitator dying, free drop drying, air suspend drying. In this research, only following methods are reviewed.

2.1. Sessile drying

Using this technique, a droplet between a micrometre and a millimetre in size is precisely positioned on a hydrophilic or hydrophobic

Acoustic or arodynamic

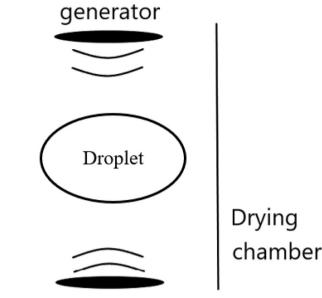


Fig. 4. Setup of Levitator drying process.

surface inside a drying chamber with controlled climatic factors (Fig. 2). The hydrophobic surface, which is produced by utilising a certain surface treatment or specific surface topography, is required to maintain the droplet's spherical shape (Xu and Choi, 2012). Ranz investigated the evaporation process of a droplet over a hydrophobic flat surface, and this approach was later widely utilised to examine the heat transfer inside a droplet, the particle shell formation, the surface mechanical characteristics (Ranz W, 1952). For the majority of other similar setups, optical equipment was employed to photograph the change in droplet shape during drying as well as to validate data of mass loss recorded using a micro-balance. Despite the fact that this method has been in use since 1962 and that it has several potential applications, a parallel between sessile droplet drying behaviour and what might occur in a spray dryer has been put out only these days (Wu et al., 2014; Lu et al., 2022).

2.2. Filament hanging drying

In order to conduct contact levitation research, a droplet which diameter was below 1 mm is hanged on the end of a syringe, glass filament, or glass capillary tube and set in a drying environment with temperature adjustable air as shown in Fig. 3 (Eslamian and Ashgriz, 2007). By attaching a microbalance to the structure or using a digital sensor to translate and calculate the deflection of the wire into the loss of droplet mass, the mass loss may be detected. A camera and thermocouples monitor the drying parameters, including the progression of the droplet's diameter, temperature, and mass. The temperature difference between the environment and the wire may be recorded in real-time thanks to thermocouples. Due to the simple design, the updated arrangement, proposed by Ranz, has frequently been used in drying studies to analyse drying process kinetics, modelling and the particle shape formation of different contents liquid, such as milk (Ranz W, 1952).

2.3. Levitator drying

The levitation method uses acoustic fields to float a single droplet in the air (Fig. 4).Davis presented a detailed explanation of the acoustic levitation principle: an ultrasonic generator creates a standing wave in a levitation chamber where an infrared thermometer and a dino-camera

Table 1
Content of whole fat milk.

Protein	24.5% - 27.0%
Lactose	36.0% - 38.5%
Fat	26.0% -
Ash	5.5% - 6.5%
Moisture	2.0% - 4.5%



Fig. 5. The droplet sessile on a glass surface.

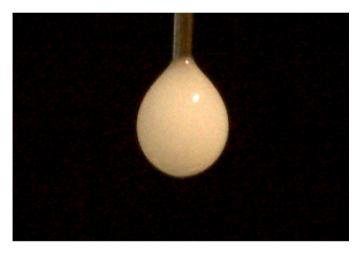


Fig. 6. The droplet hanging to a filament.

are applied to capture heat transfer and shape data as a millimeter droplet evolves (Davis et al., 1981).

3. Material

Whole fat milk powder used in this research is from Nestle and the content is shown in following table, the raw size D50 is $78.9~\mu m$. Powders were reconstituted at $20^{\circ}C$ in de-ionized water with magnetic stirrer with 10%, 20%, 30%, 40% w/w solid content. Pure de-ionized water droplet is added red dye (EBEST RHODAMINE B acid red 52) for better observation.

3.1. Method

Different solid content whole fat milk single droplets are dried in three different methods. Cameras (Dino-lite Digital Microscope, Edge AM73915MZT) with 45FPS and 1280×960 resolution are used to capture the drying process. 2.5 μl volume droplets are generated by Eppendorf pipettes manually. The distance between camera and droplet is 20 cm and the magnification rate is set at 40x.



Fig. 7. The droplet suspended in levitator.

3.1.1. Sessile drying

Single droplet is placed on a hydrophobic substrate as shown in Fig. 5, and the hydrophobic glass surface is formed by a liquid glass SiO2 and specialist silicone waterproof spray (CAR-CHEM, UK). The milk droplet form in half-sphere shape on the surface.

3.1.2. Filament hanging drying

Single droplet is attached to a filament and placed in a drying chamber as shown in Fig. 6. The droplets were attached to the filament by Eppendorf pipettes and hold by friction and capillary force.

3.1.3. Levitator drying

A single droplet wes held in the air using the acoustic force of the levitator. A drying chamber that is temperature and humidity-controlled isolates the suspended droplet (20° C, 40% RH). The droplet can be continuously observed inside the chamber during the drying process via a glass window as shown in Fig. 7. Inside the levitator, ultrasonic sound waves are generated at a frequency of 58 kHz by a generator. The sound wave is reflected by a concave reflector forming the so-called standing wave. The acoustic radiation pressure that results from the droplet in the acoustic field generates the required acoustic force to suspend the droplet against the gravity force.

3.1.4. Spray dryer

The spray dryer used is a co-current spray dryer, Mobile Minor from GEA, German. And the milk powder was produced with 160°C inlet temperature, 90°C outlet temperature, inlet air velocity 100 kg³/h (fan power 50 mmWG), 1 bar compressed air for atomizer and 2.5 kg/h feeding rate. 20%, 30% 40%, 50% w/w whole fat milk powder were

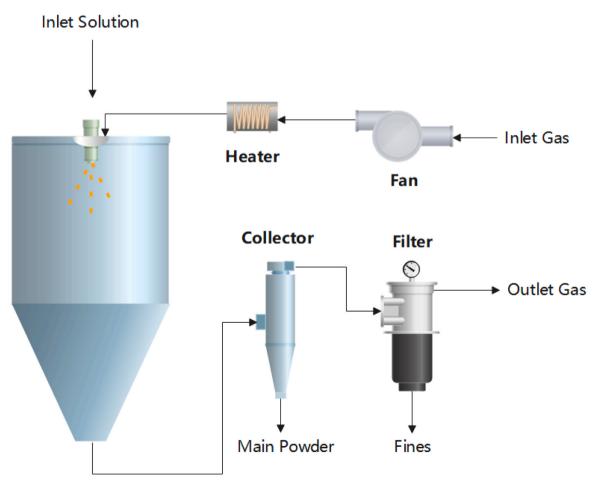


Fig. 8. Sketch of Mobile Minor spray dryer.



Fig. 9. The typical drying process of sessile drying droplets (40% w/w).

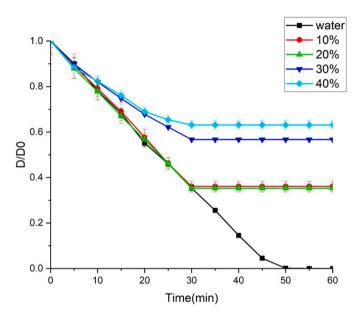


Fig. 10. The drying rate curve of different concentration milk droplets of sessile drying droplets.

produced. The sketch is shown in Fig. 8.

3.1.5. Data analyse

All droplet images are transferred to diameter data by ImageJ, ImageJ can calculate area and pixel value statistics of user-defined selections and intensity-thresholder objects. The droplet drying rate is defined as diameter reduction by D/D0, because some of the droplets are not sphere enough, so D calculated by $D=\sqrt{L*H}$ when L is the length and H is the height of droplets.

3.1.6. X-ray

X-ray images of the dried droplet were obtained using microCT35 (Scanco Medical AG, Switzerland. The X-ray beam was operated at a voltage of 45 kV, a current of 177 μ A and a powder of 8 W. The voxel size used was 0.8 μ m.

3.1.7. Scanning Electron Microscopy

Scanning electron microscopy JEOL JSM- 6010LA (Japan) was used to obtain electron micrographs of the primary powders and dried droplets. The samples were coated with gold using AGAR sputter coater (AGAR, UK).

4. Results and discussion

4.1. Sessile drying process

Different concentration milk drying rate and feature were observed by attached to a hydrophobic surface by Dino camera. The droplets were generated by pipettes and the volume of droplet is limited to 2.5 $\mu l.$ The typical drying process and shape change of sessile droplet is shown in Fig. 9. The contact surface between droplet and hydrophobic surface and its area is nearly not change. That may be mainly caused by the capillary forces and surface tension between the glass surface and liquid. The shell formation with time is not even and not sphere with high solid content which is the shrinkage area inside the droplet. The drying area is half sphere and with the hot air inlet, the difference between surface and droplet may cause further heat transfer and speed up the drying rate

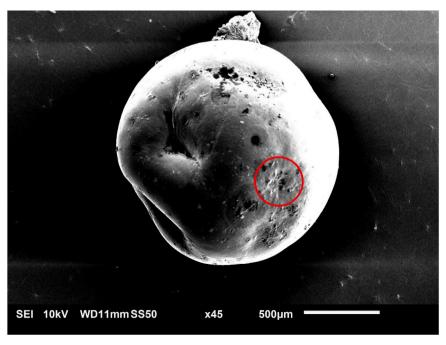


Fig. 11. SEM of 40% w/w whole fat milk droplet in sessile drying process.

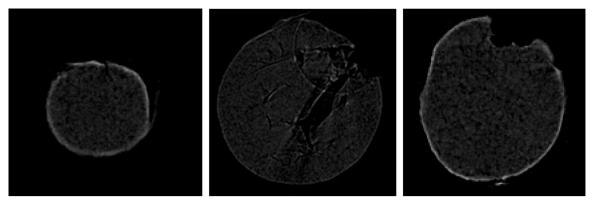


Fig. 12. X-ray images of dried droplet in sessile drying process.

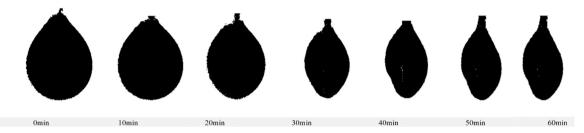


Fig. 13. The typical drying process of contact hanging drying process (40% w/w).

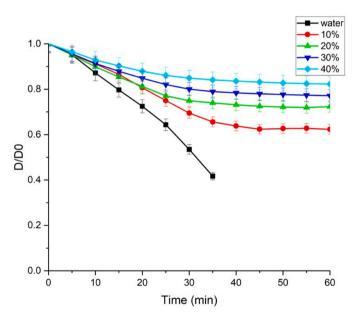


Fig. 14. The drying rate curve of different concentration milk droplets.

compared with the droplet itself.

The size reduction of different solid content droplets dried in room temperature is shown in Fig. 10. Generally, the drying rate increases with the decrease of solid content. The shell is not formed or not well formed, which result in the low concentration droplet drying curves (pure water, 10%, 20%) are overlapped. The end point is little bit higher than the solid content, which may because of the bubble area inside the droplet. 10% and 20% droplets are in low viscosity and the shell formation is slow, so that the components hardly hold the water evaporation so that cause the overlapping with pure water, but with time after 30 mins, the 10% and 20% droplet form a tiny shell so they did not end at 0. Because of their high viscosity and shell formation speed, 30% and

40% droplets drying curve are overlapped. The limitation of this method is for low viscosity droplet, high contact angle and high hydrophobic surface is necessary.

The SEM is shown in Fig. 11. The surface is not smooth and sphere. Some areas show different darkness could be different components. Besides, the net shape marked in the image could be protein. During the drying process, the shrinkage was observed on the top of the droplet.

The X-ray images of the dried droplet on different layers are shown in Fig. 12. As observed during drying process and SEM photos, there are some shrinkages in the middle layer of the dried droplet and the area of the droplet bottom is nearly the same with the initial droplet.

4.2. Filament hanging drying process

Different concentration milk drying rate and feature were observed by attached to a thin metal wire and Dino camera. The droplets were generated by syringe and the size of droplet is limited to 2.5 μl and were held by the friction of the wire/filament surface. The typical drying process is shown in Fig. 13. The pure water is mixed with pink dye then captured by Dino camera and Image J. And 10%, 20%, 30%, 40% solid content milk droplets were dried for 1 hour until dry. The shape is not spherical because of the gravity. Besides, because of this method is an intrusive filament, the shape may change because of the capillary force as well. The shell formed is not smooth and the shrinkage is still existed at the bottom of the droplet, which happened at the end of the drying process because of the shrinkage of shell.

The size reduction rate is decreasing with the increase of concentration but need more time until total dried as shown in Fig. 14. The drying rate is obviously increases with the decrease of solid content. The shell is well formed that all the end point is much larger than the solid content. The milk droplet of 40% solid content end around 0.8 which refers to high porosity. Pure water curve end by the half of time because it's almost dried and the filament will be calculated as the area of droplet by ImageJ.

The SEM is shown in Fig. 15. The surface is typically combined by different component which include fat, protein and lactose. The shape of the dried droplet is sphere on the bottom part of the droplet, the top

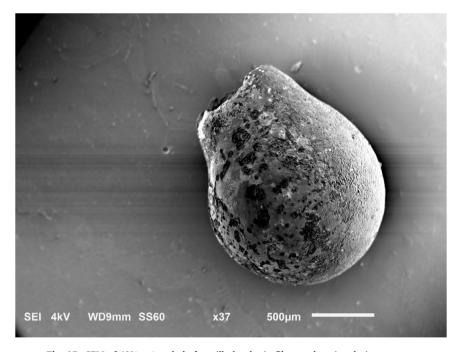


Fig. 15. SEM of 40% w/w whole fat milk droplet in filament hanging drying process.

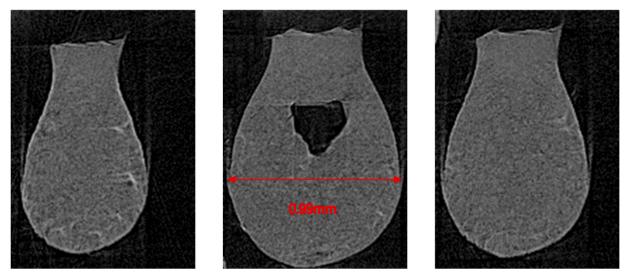


Fig. 16. X-ray images of 40% w/w whole fat milk dried droplet in filament hanging drying process.

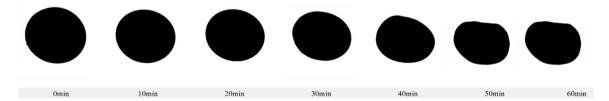


Fig. 17. The typical drying process of levitator drying process (40% $\mbox{w/w}$).

shape is formed by the friction and capillary force between the liquid and the syringe metal surface.

The X-ray images of the dried droplet on different layers are shown in Fig. 16. It shows 40% w/w whole fat milk droplket droplet end at 0.8 in Fig. 14 which meaning the volume reduced by 20% compared with the initial liquid droplet while the water content is 60%. After checking the

SEM and X-ray images, the hole was caused by the filament intrusion, which means the porosity need to be considered by deleting the intrusion area. So even the droplet is seen to be solid in low temperature drying, the real structure should be porous.

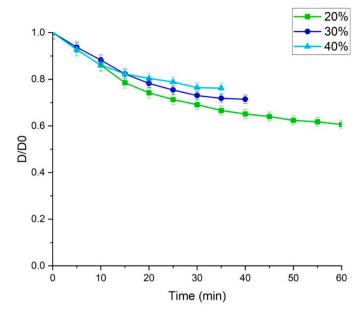


Fig. 18. The drying rate curve of 20%, 30%, 40% w/w Whole Fat Milk in Levitator.

4.3. Levitator drying process

Different concentration milk drying rate and feature were observed by suspended in a levitator and observed by Dino camera. The droplets were generated by Eppendorf pipettes in 2.5 μ l. The typical drying process and shape change of droplet in levitator is shown in Fig. 17. It shows more spherical shape and very smooth shape during drying. However, with the time, shrinkage will happen on both top and bottom, which may because of centrifugal force cause by the rotation of droplet itself forced by ultrasonic.

The comparison of 20%, 30%, 40% w/w whole fat milk powder were showing in Fig. 18 by using fitting average line. The end drying particle diameter increase with the solid content. The 20% w/w droplet drying rate is fast that others because of higher water content. The figure shows

that the drying rate decrease with solid content. Pure water droplet and 10% w/w droplet cannot be suspended for a long time in the levitator because no well form shell to precent the shape change, the droplet will be forced apart because of centrifugal force.

The SEM is shown in Fig. 15. The surface is much smoother than the dried droplet in Figs. 11 and 15. The component cannot be told from the surface difference, different components may be forced to move to surface because of the centrifugal force when the droplet rotate by itself. And the shape is not sphere. During the drying process, the shrinkage was observed on the top and the bottom of the droplet.

The X-ray images of the dried droplet on different layers are shown in Fig. 20. The donut shape is also approved by images as described above that the shrinkage happens on top and bottom of the droplet during the drying process.

4.4. Comparison of different drying process

The size reduction of different drying method of 40% solid content milk droplets are shown in Fig. 21, of which the sessile drying is fast than levitator drying and filament hanging drying. The rate difference may because of the shape and porosity is different which the sessile drying is half spherical droplet, and the filament hanging drying, and levitator drying is much more like whole spherical droplet. Besides, the higher drying rate of droplets in levitator is because during the operation, not only the ultrasonic generator will generate heat, but the droplets are also rotation by itself which will accelerate the evaporation speed.

The comparison of single dried droplet and spray dried powder with SEM and CT images contain surface formation and the porosity. The single dried droplets are much bigger than the spray dried powder because of initial droplet size. The SEM images shows the surface structure, the single droplet shows smoother surface and no breakage, because the moisture is not evaporated and break the shell structure. For the porosity, all dried single droplets are low porosity because of low temperature, however, the filament hanging drying method have an insert filament which result in a hole in the middle. The spray dried whole fat milk powder shows very hollow sphere shape both in SEM and X-ray images. The condition and size comparison are shown in Table 2.

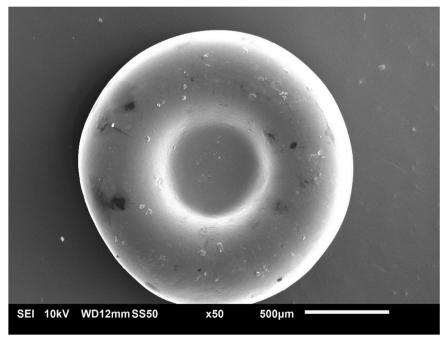
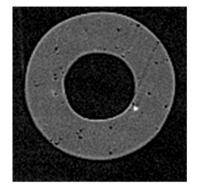
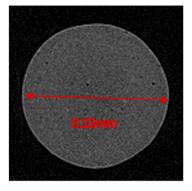


Fig. 19. SEM of 40% w/w whole fat milk droplet in levitator drying process.





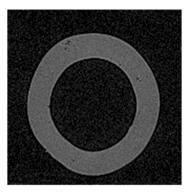


Fig. 20. X-ray images of dried droplet in levitator drying process.

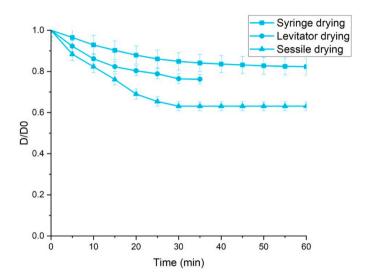


Fig. 21. Droplet Size Reduction of 40% w/w Whole Fat Milk in three different single droplets drying process.

Table 2
Dried droplet comparison of 40% w/w Whole Fat Milk

	Size range		
Sessile (Room temperature)	1.8–2 mm (droplets)		
Filament hanging (Room temperature)	0.70–1.40 mm (dried droplets) 1–1.2 mm (droplets)		
Levitator suspended (Room temperature)	0.91–0.99 mm (dried droplets) 0.8–1.2 mm (droplets)		
Spray dryer (160°CInlet 90°C Outlet)	0.07–0.20 mm (dried droplets) 0–50 μm D50 13.9 μm (Dried powder)		

Table 3Main size of different concentration spray dried whole fat milk powder.

	20%	30%	40%	50%
D10 (μm) D50 (μm) D90 (μm)	5.2 ± 0.10 11.5 ± 0.08 20.2 ± 0.15	5.4 ± 0.12 13.8 ± 0.12 29.8 ± 0.24	5.5 ± 0.09 13.9 ± 0.15 34.7 ± 0.33	5.8 ± 0.10 14.5 ± 0.12 38.9 ± 0.52

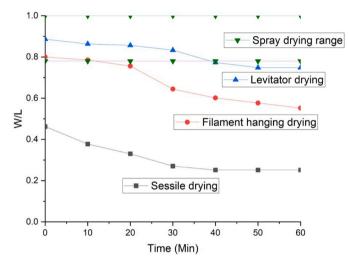


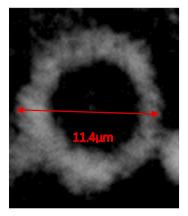
Fig. 22. Shape factor(w/l) of dried particles of different methods.

4.5. Spray drying process

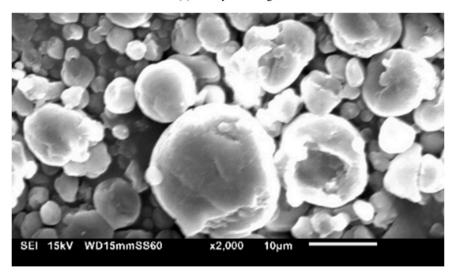
20%, 30%, 40%, 50% whole fat milk powder is produced in spray dryer within the same condition and the main size is shown in Table 3. The size is slightly increased with the increase of the solid content, which is similar to the single droplet drying process, higher concentrate droplet will end the drying process with a higher D/D0. For shape factor (W/L) of dried particle shown in Fig. 22, it shows that the droplet shape in levitator drying is the closest to the shape factor of spray dried powder in spray dryer. The X-ray CT image of spray dried particle blur shows in the table in is limited to the equipment even use the minimum Voxel size 0.8 µm because of the small particle size shown in Fig. 23 as well as SEM images of spray dried powder which showed a lot of hole sphere shape. Though some of the drying rate or size trend change with solid content are similar between single droplet drying and spray dryer drying, there're still big differences in size, shape, drying time, surface structure.

5. Conclusion and Limitation

All these single droplet methods have some limitations, the limitation of sessile drying is that even with hydrophobic surface, the droplet will only form a hemispheroid and the heat transfer from the surface generated by different specific heat capacity cannot be ignored during high temperature drying. The limitation of filament hanging drying is that the droplet shape will be changed by gravity force and the heat transfer from the contacted area generated by different specific heat capacity cannot be ignored. The limitation of levitator drying is that the droplet shape will be changed by ultrasonic to force the droplet stay and rotate in the middle of the chamber. The heat generated by the



(a) X-ray CT image



(b) SEM image

Fig. 23. X-ray CT (a) and SEM (b) images of 40% w/w whole fat milk droplet in spray drying process.

ultrasonic generator cannot be ignored as well.

The obtained experimental data can be used to determine the drying parameters of whole milk and to verify the theoretical drying model in simulation and scaling up. However, when using approaches that investigate spray-dried droplet properties by analysing the drying properties of single droplets, extra caution must be taken. The developed drying kinetics measurement method can also contribute to other materials. In order to select the simulation of different parameters in the production process of spray drying, different single droplet drying experiment methods could be critically selected based on the method limitations and different materials provided in this article and different materials.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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