Spray Drying Process Scale-up

Technology Development & Scale up Sharing

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Why the scale up is a big challenge in Chemical process?

Process Understanding: Know what is the process in the system

1. Momentum transfer: force transfer, change of velocity, velocity profile, mass flow, mixing
2. Mass transfer: more than 2 phases in the system
3. Heat transfer: heat generate/consume system, need to control the precise temperature
4. Reaction

Why the scale up is a big challenge in Chemical process?

Process Understanding: Know what is the control mechanism

1. Momentum transfer rate (mixing related)
2. Mass transfer rate
3. Heat transfer rate
4. Reaction rate

The slowest rate the controlled mechanism.

Why the scale up is a big challenge in Chemical process?

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Problem: the physical rate change with scale but the chemical reaction rate does not change with scale.

1. Physical rate (Momentum, Heat, Mass transfer rate)
2. Chemical reaction rate

Why the scale up is a big challenge in Chemical process?

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Problem: the physical rate change with scale but the chemical reaction rate does not change with scale.

↓

The controlled mechanism may change with scale especially the more complex process (compose of more than 2 small processes)

↓

Challenge to design or sizing the large scale unit (Scale-Up Method)

Why the scale up is a big challenge in Chemical process?

How to Scale Up ?

↓

Using the experimental data from small scale to design the large scale unit

↓

Physical Rate: Rearrange the correlation in a function of dimensionless group

Reaction rate: Using reaction rate expression / rate law

Why the scale up is a big challenge in Chemical process?

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How to ensure the control mechanism in the large scale ?

↓

Design and validation in the suitable scale up ratio (depend on the complicate of system)

↓

Pilot scale was use to validate the scale up method for each step size of scale up ratio.

Process Scale Up Objective

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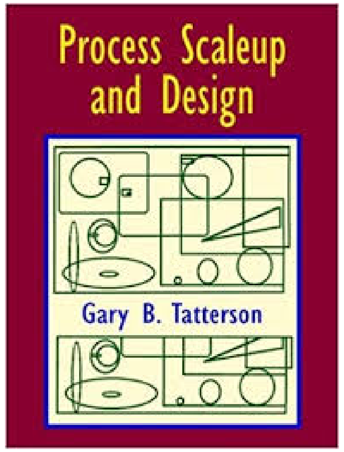
The objective of scale-up is to obtain process similarity between the two scales.

Process Similarity

Process similarity exists between two processes when the processes accomplish the same objectives by the same mechanism and making the same product.

Only one or the most important objective is selected.

7 Steps for Systematic methods for Unit Operation scale up



Tatterson, Gary B (2002). Process scaleup and design, Greensboro, NC

Systematic Method for Scale up

1. **State the scaleup objectives: This usually means seeking process similarity. What are the important aspects of the process? What is being attempted?**
2. **What is doing the work in the process? What is the controlling mechanism? What is the size of the job? Where is the process occurring?**
3. **What are the process physics and chemistry? What are the process flow regimes?**
4. **At this point, *geometric similarity may be assumed for scaleup*. Geometric similarity is really not needed but serves as a starting point for this method.**

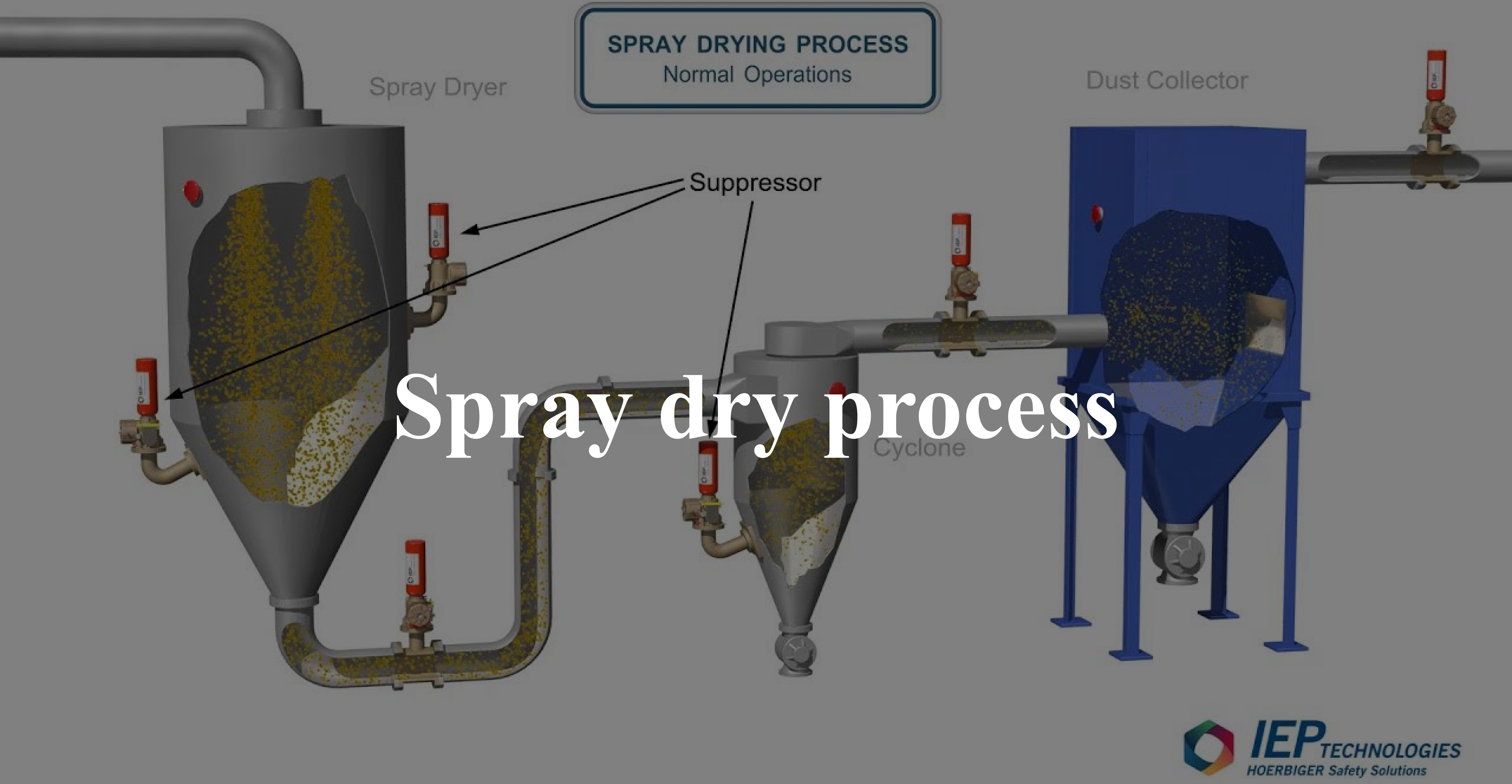
*Tatterson, Dr. Gary Benjamin. Process Scaleup and Design

Systematic Method for Scale up

5. **From the various data and correlations, the scaleup method is obtained. Geometry is obtained from geometric similarity. Operating conditions are obtained from analysis of data and correlations.**
6. **At this point, *a judgment is made as to whether the scaleup is appropriate*. Are the results reasonable? Can the scaleup be accomplished? Will it be a working design?**
7. **If yes, stop. If no, go to other options (Plan Bs). One Plan B that is particularly effective is to change geometry to advantage.**

*Tatterson, Dr. Gary Benjamin. Process Scaleup and Design

Spray dry process



Spray dry process

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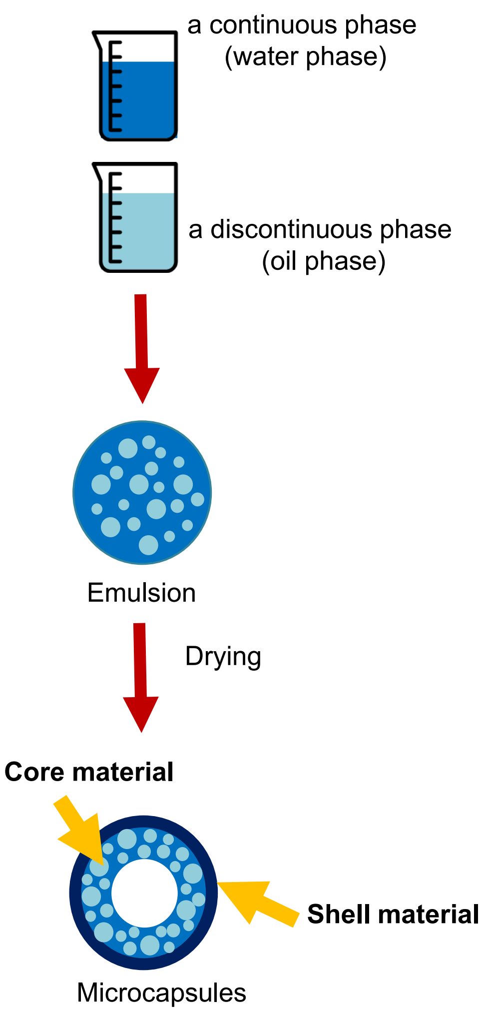
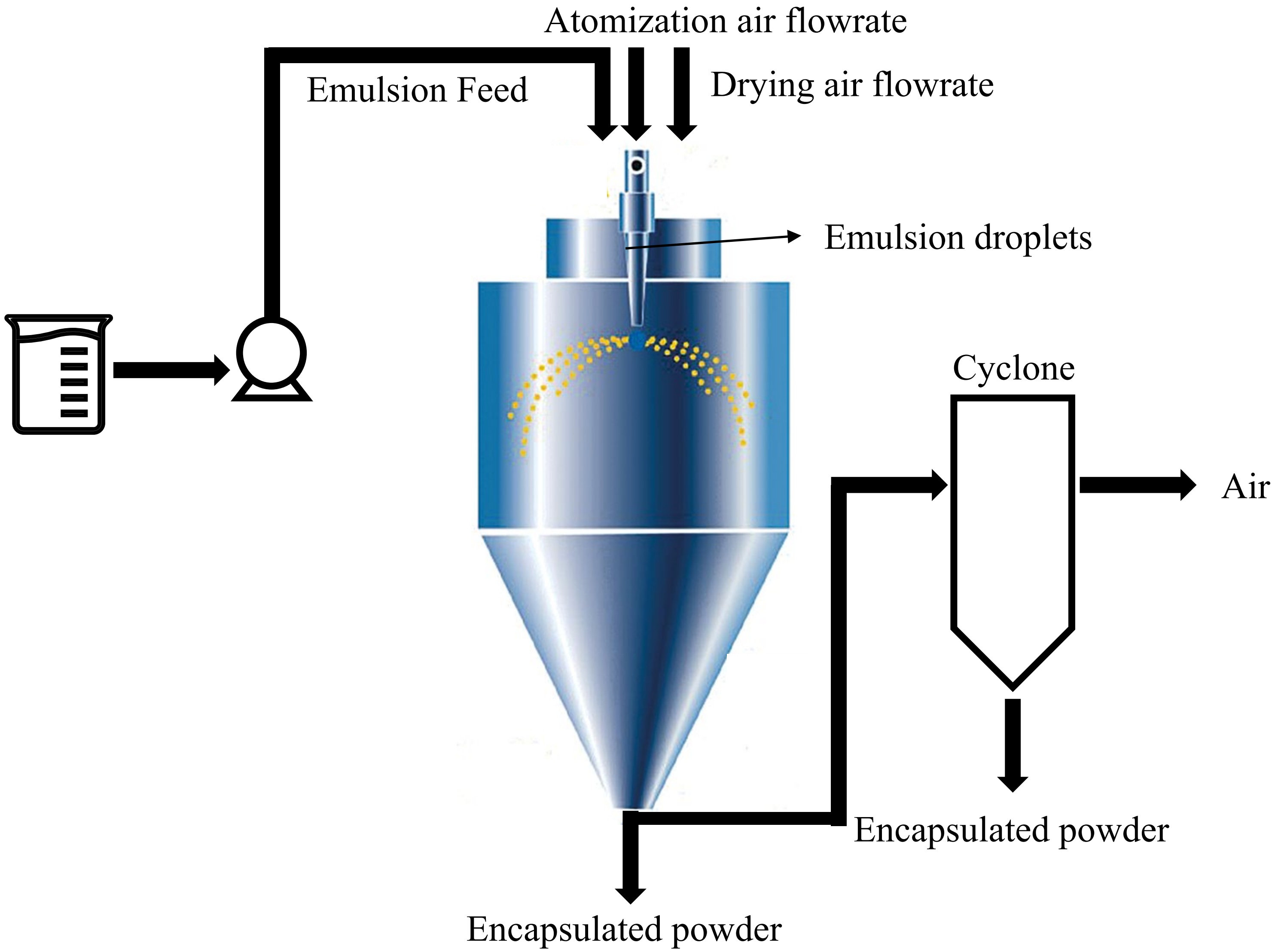
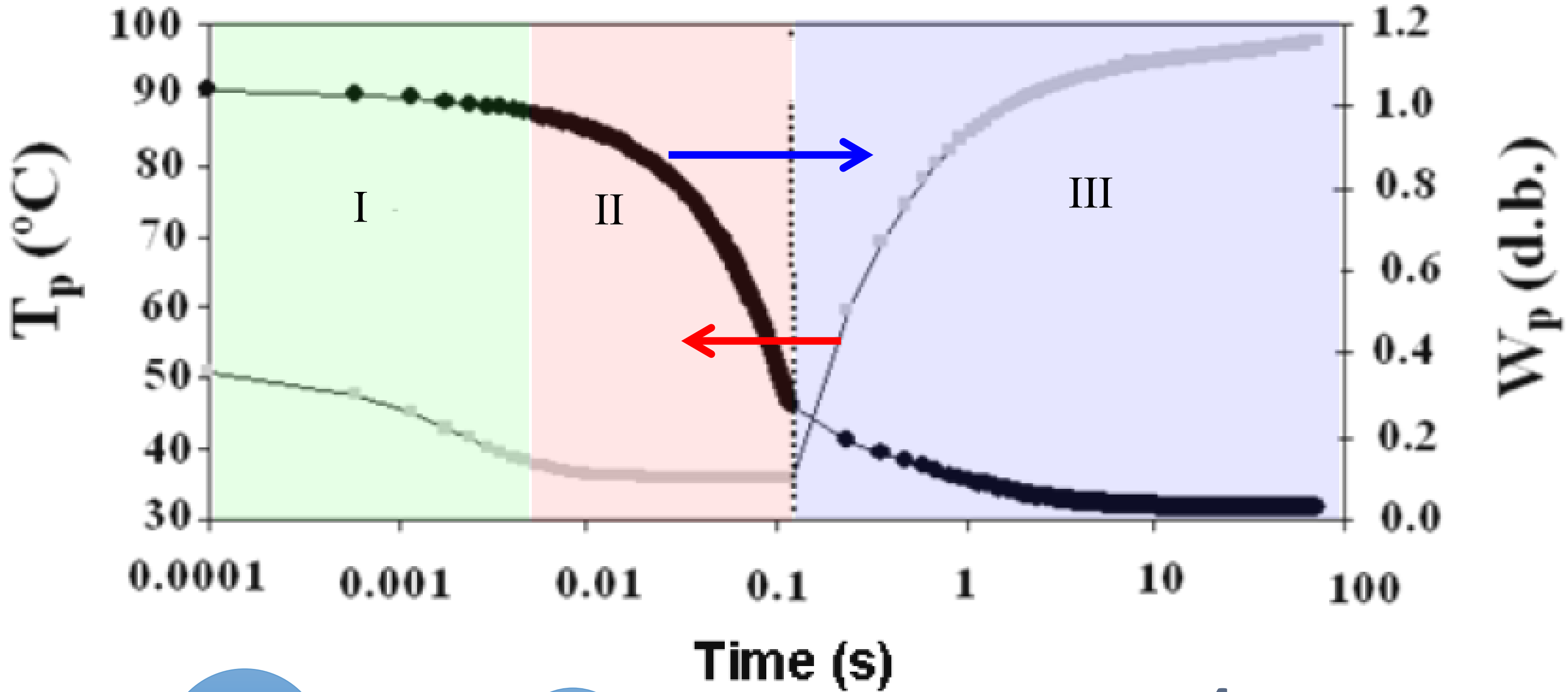
What is spray dry process ?

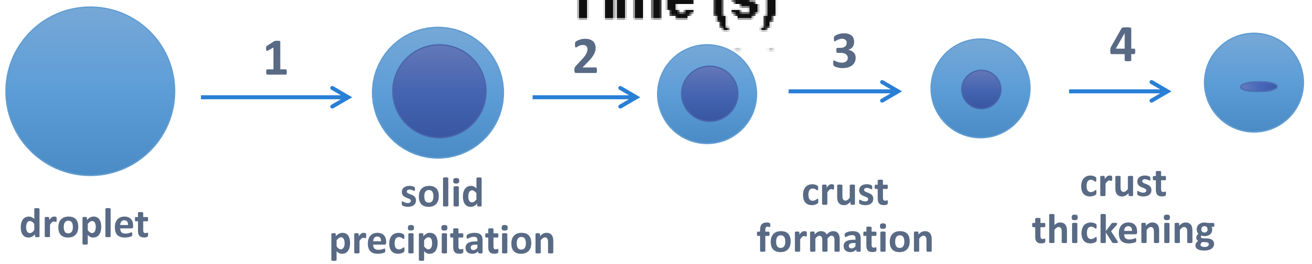




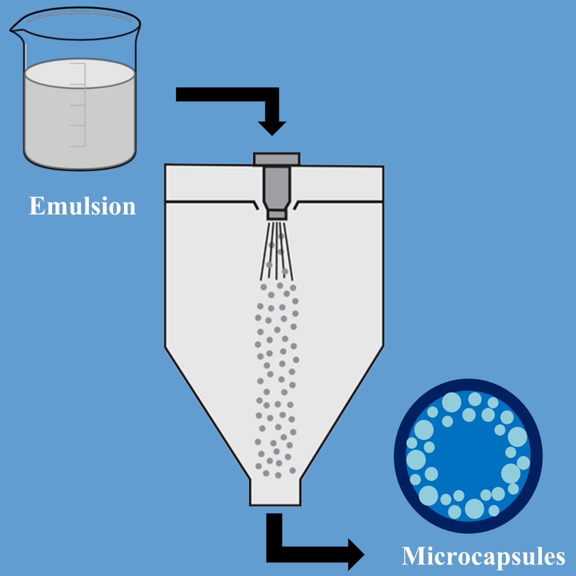
Powdering by Spray dryer

History of particle drying inside the spray dryer*





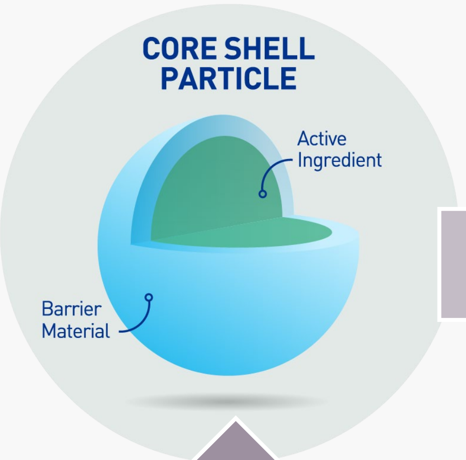
*V.S. Birchal and M. L. Passos, MODELING AND SIMULATION OF MILK EMULSION DRYING IN SPRAY DRYERS, *Brazilian Journal of Chemical Engineering,* Vol. 22, No. 02, pp. 293 - 302, April - June, 2005

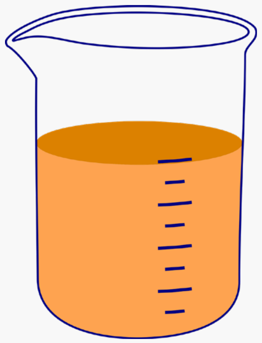


Scale up
Spray dry process

Scale up spray dry process

Encapsulation technique





Emulsion

Spray drying

Laboratory-scaled

Mini Spray Dryer B-290

SCALE UP

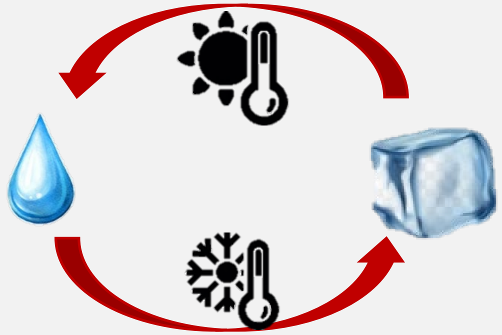
Jumbo-lab scale

MOBILE MINOR™

SCALE UP

Pilot Scale

VERSATILE SPRAY DRYER



Production of Microcapsulated PCM by Pilot-Scaled Spray Dryer

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

Production of microencapsulated PCM by pilot-scaled spray dryer :

Purpose : To study on the preparation step of emulsion and evaluate the production of phase change materials via pilot-scale spray drying process.

Solution : PCM (active ingredient) and Cetyl Trimethyl Ammonium Chloride (excipients) in DI water (solvent)

Spray dryer model and atomizer types :

Laboratory-scale

(a)

(b)

Fig.6 Spray dryer model and atomizer types in Laboratory-scale :
(a) Buchi-290 , (b) Two fluid nozzle
Ref : https://www.buchi.com/th/products/instruments/mini-spray-dryer-b-290

Jumbo-Lab scale

(a)

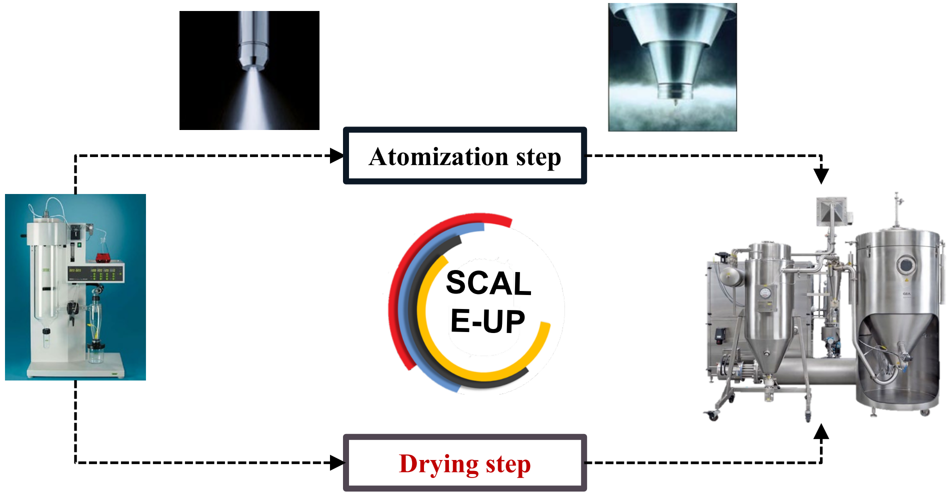
(b)

Fig.7 Spray dryer model and atomizer types in Pilot-scale:
(a) Niro A/S mobile minor , (b) Rotary disc
Ref : https://www.gea.com/spray-dryers/pilot-scale-spray-dryers/mobile-minor

Method : Scaling up the spray dryer process via vary the pressure in atomizer and using mass and energy conservation to find the appropriate hot air inlet temperature.

Assumption :

"as long as the blown-out rate from the atomizer and the ratio between the evaporated water and the solid substance in the drying chamber is kept consistent in both laboratory and scaled-up settings, the encapsulation power will be similar."



WATER EVAPORATION : SOLID CONTENT

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

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Step for scale up spray dry process :

FEED PREPARATION

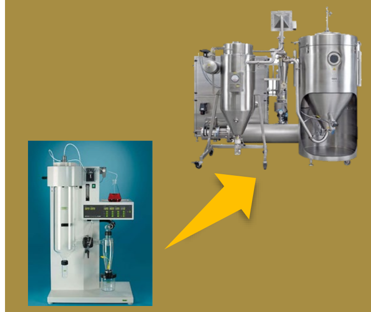
- Sol-gel process
- Emulsion size
- Effect off sol-gel time

ATOMIZATION STEP

- Correlation of atomizer air pressure vs particle size

DRYING STEP

- Varying air inlet temperature
  - 120 / 150 / 160 / 180 °C
- Varying feed flow rate
  - 0.4 / 0.84 / 1.2 / 1.6 kg/h

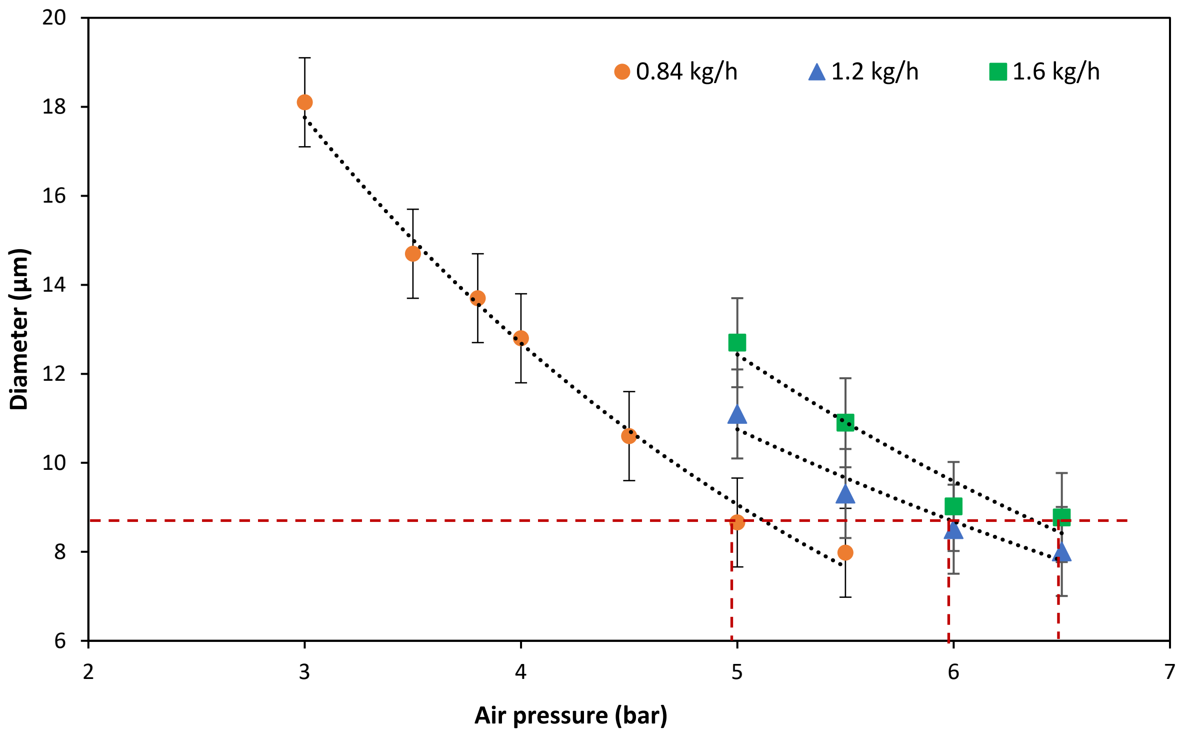


WATER EVAPORATION: SOLID CONTENT

- Varying solid content
  - 11.2 / 15 / 20 wt%
- Varying feed flow rate
  - 0.84 / 1.2 / 1.6 kg/h

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

ATOMIZATION STEP



Atomizer air pressure ↑ **Particle size** ↓

Feed liquid flow rate ↑ **Particle size** ↑

Lab-scale

Two-fluid nozzle

- **nozzle tip diameter:** 0.7 mm
- **nozzle cap diameter:** 1.4 mm
- **Atomizer air flow rate:** 0.34 kg/h
- **Achieved particle size:** 8.3 µm

Pilot-scale

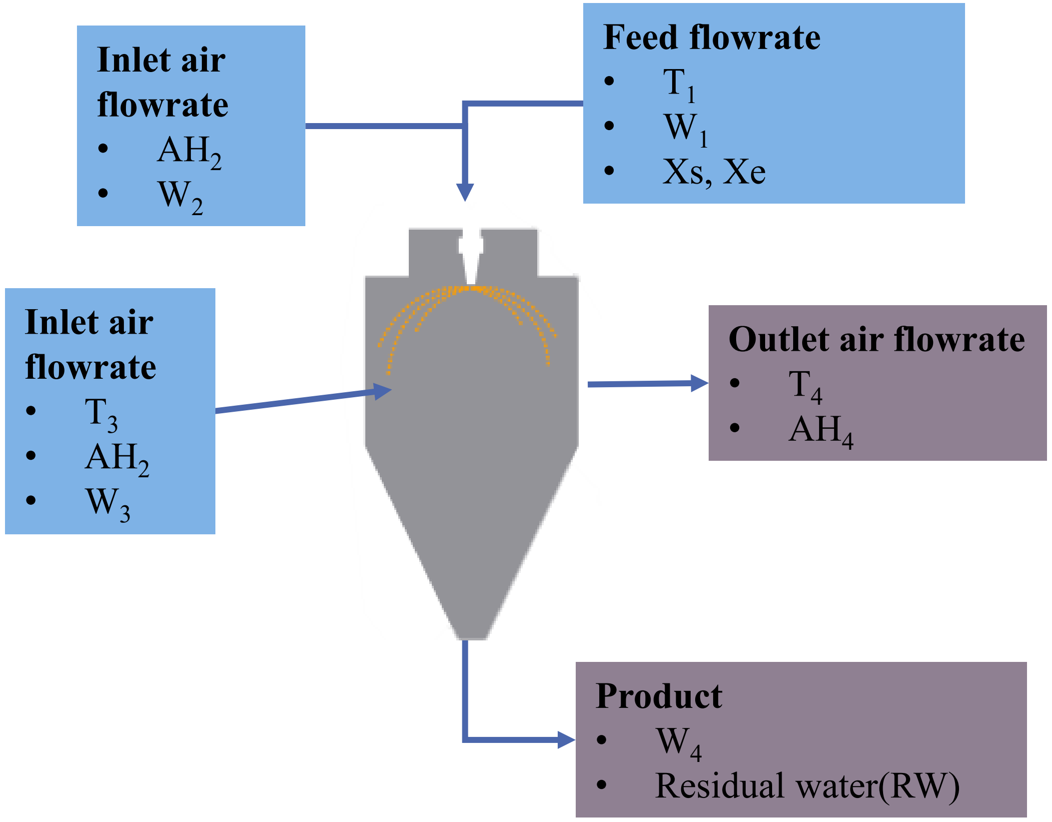
Rotary atomizer

- **Wheel diameter:** 50 mm
- **Number of channel:** 24 holes
- **Atomizer air pressure:** 3 – 6.5 bars
- **Achieved particle size:** 8 - 18 µm

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

DRYING STEP

Using Mass & Energy balance : Determine drying air inlet temperature under assumption the ratio between the evaporated water and the solid substance in the drying chamber is kept consistent in both laboratory and scaled-up settings



Mass balance

$$\left(\frac{W_2+W_3}{1+AH_2}\right)(AH_4) = \left(\frac{W_2+W_3}{1+AH_2}\right)(AH_2) + W_1(1 - Xs - Xe) - W_1X_s\left(\frac{RW}{1-RW}\right)$$

Water in outlet air | Water in inlet air | Water in feed liquid flow rate | Water in particles

Heat balance

$$W_1(1\text{-}X_s\text{-}X_e)C_{pw}(t_4\text{-}t_1)$$

$$+ \left(\frac{W_2+W_3}{1+AH_2}\right)(AH_4\text{-}AH_2)\,\Delta H_v$$ — Latent heat of water

$$+ W_1(X_e)[\Delta H_v + C_{pe}(t_4\text{-}t_1)]$$ — Sensible &latent heat of ethanol

$$+ W_1(X_s)C_{ps}(t_4\text{-}t_1)$$ — Sensible heat of solid

$$= \left(\frac{W_2+W_3}{1+AH_2}\right)C_H(t_3\text{-}t_4)$$ — Heat from hot air

$$- UA(t_4\text{-}t_1)$$ — Heat loss

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

WATER EVAPORATION : SOLID CONTENT

Scale-up

Vary 2 impact variable and Using mass & energy balance to caculate appropriate drying air inlet temperature :

- Feed flow rate
- Solid content

Operating condition in laboratory-scale and Jumbo-lab-scale spray dryer :

Feed flow rate

Spray dyer	Lab-scale	Jumbo-lab-scale		
Feed liquid flow rate (kg/h)	0.34	0.84	1.20	1.60
Air flow rate (kg/h)	40.0	88.0		
atomizer air pressure (bars)	-	5	6	6.5
Inlet air temperature (℃)	160	150	159	168
Solid content (wt%)	11.2	11.2		
Water evaporation : solid content	6.61	6.61		
Water content (wt%)	5.00	5.00		

Solid content

Spray dyer	Lab-scale			Jumbo-lab-scale		
Solid content (wt%)	11.2	15	20	11.2	15	20
Air flow rate (kg/h)	40			88		
Inlet air temperature (℃)	160	158	156	151	149	148
Water evaporation : solid content	6.61	4.38	2.71	6.61	4.38	2.71
Feed liquid flow rate (kg/h)	0.34			0.84		
Water content (wt%)	5.00			5.00		

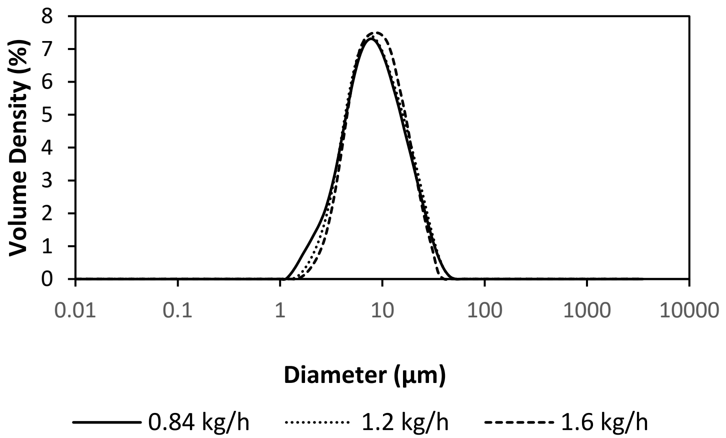
Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

Results

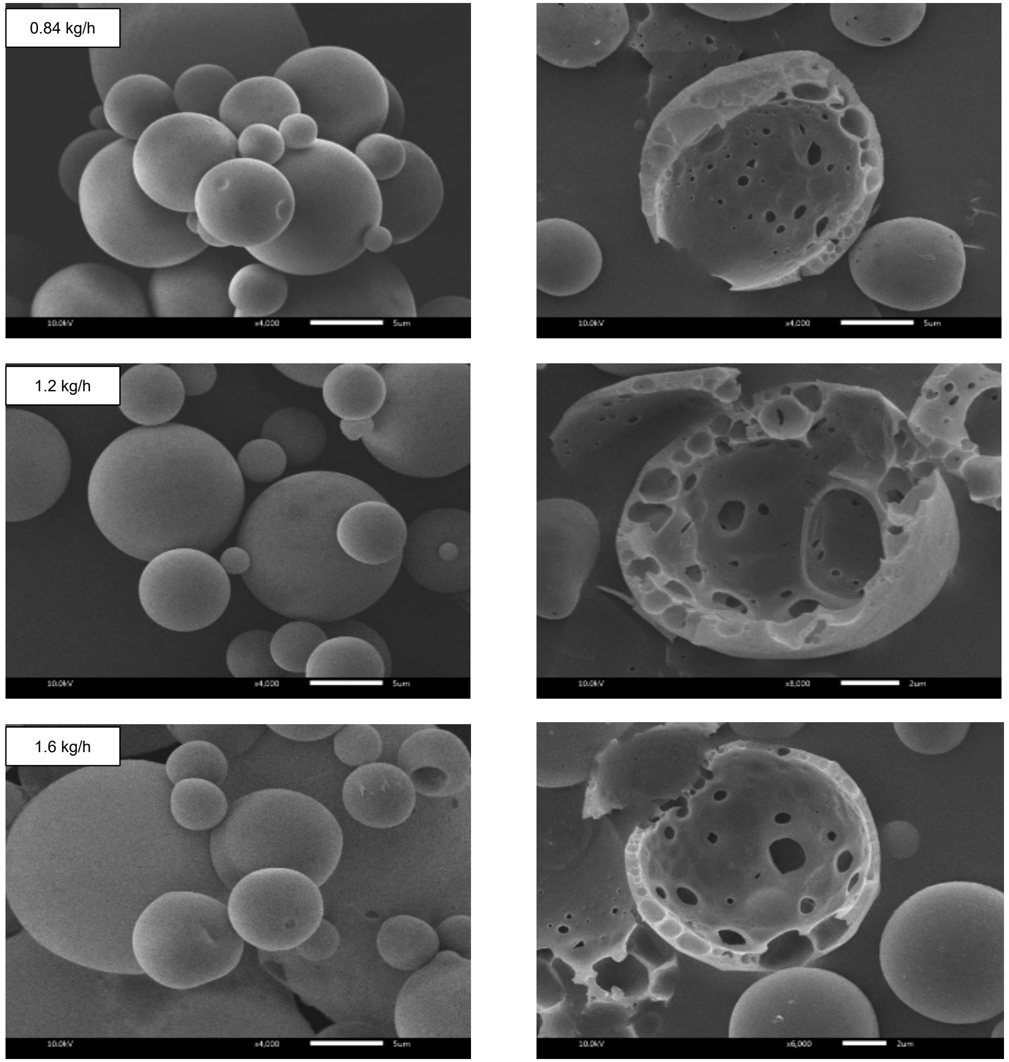
FEED LIQUID FLOW RATE

Physical properties of microcapsules

Spray dyer	%Yield	Diameter (µm)	Water content (wt%)	Water evaporation : solid content
Lab-scale	39.2	8.3	6.32	6.60
Jumbo-lab-scale				
0.84	60.7	8.63	6.58	6.60
1.2	57.6	8.51	6.79	6.59
1.6	55.3	9.02	6.9	6.59



Particle size distribution



SEM image of PCM microcapsules

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

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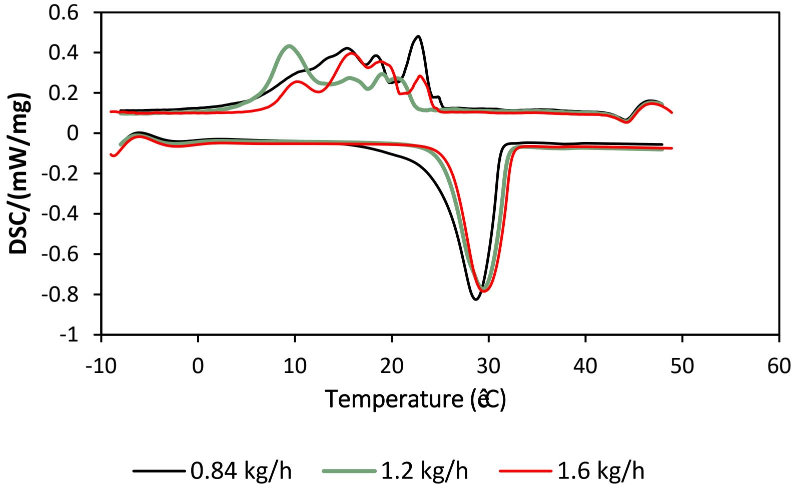
Results

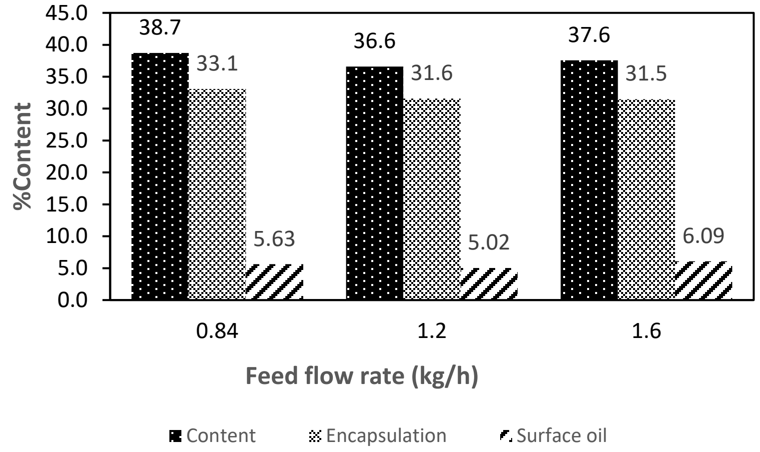
FEED LIQUID FLOW RATE

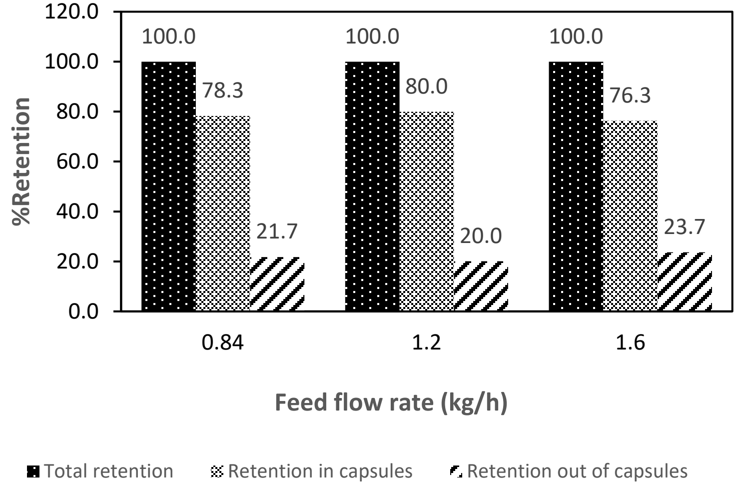
Thermal properties of microcapsules

Spray dyer	Feed liquid flow rate (kg/h)	ΔH_m (J/g)	ΔH_c (J/g)
Lab-scale	0.34	77.67	75.53
Jumbo-lab-scale	0.84	79.37	77.83
	1.2	75.01	71.64
	1.6	77.04	76.39



DSC curves





PCM in microcapsules after spray drying
(a) %Content (b) %Retention

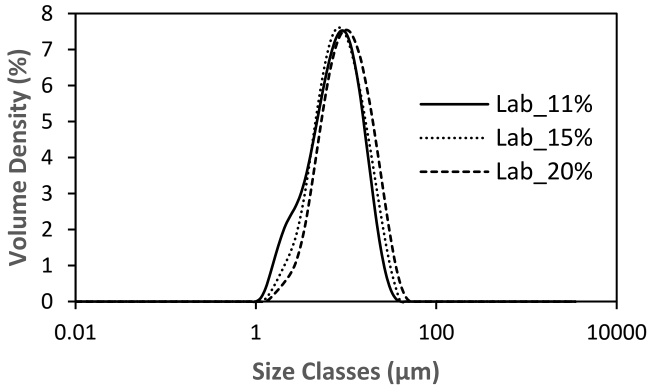
Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

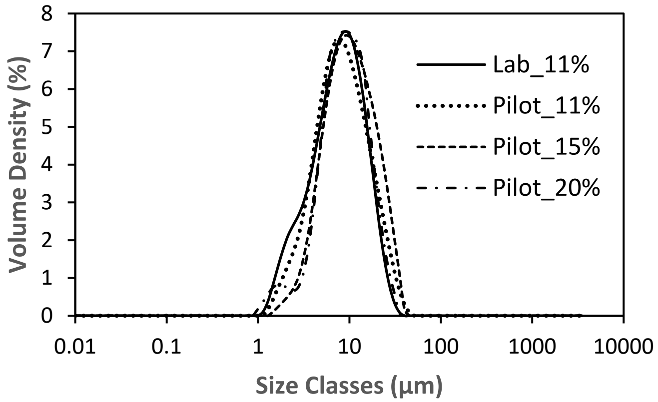
Results

SOLID CONTENT

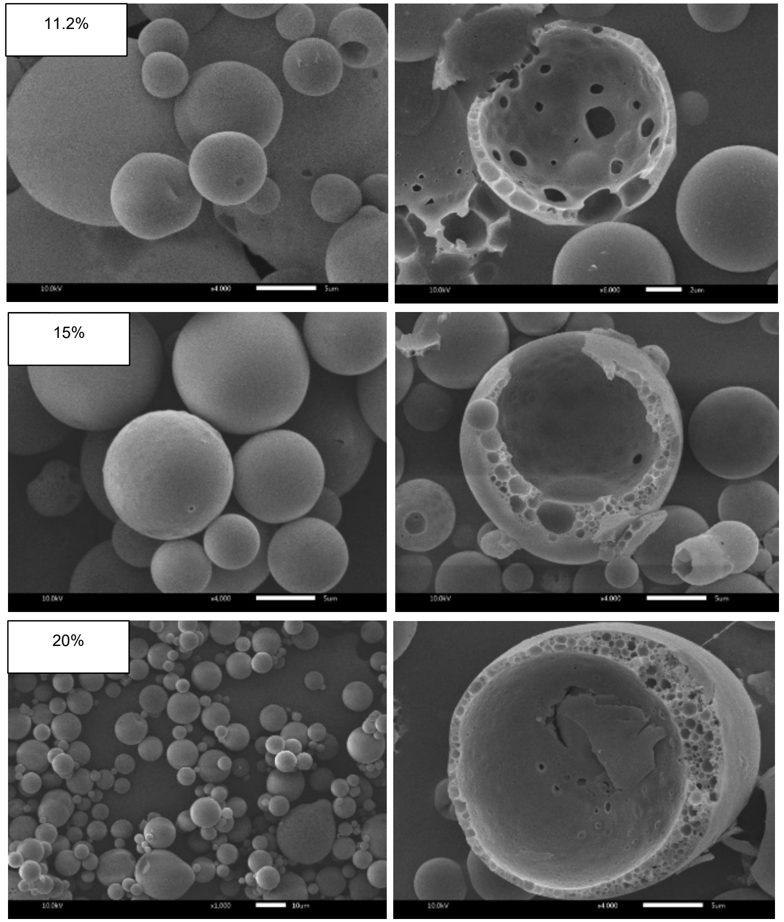
Physical properties of microcapsules

Spray dyer	%Yield	Diameter (µm)	Water content (wt%)	Water evaporation : solid content
Lab-scale				
11.2	39.2	8.3	6.32	6.60
15	40.0	9.3	6.74	6.59
20	39.6	10.4	6.41	6.60
Jumbo lab-scale				
11.2	60.7	8.63	6.58	6.60
15	64.0	10.8	6.75	6.59
20	63.3	9.47	6.88	6.59





Particle size distribution



SEM image of PCM microcapsules in Jumbot-lab-scale

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

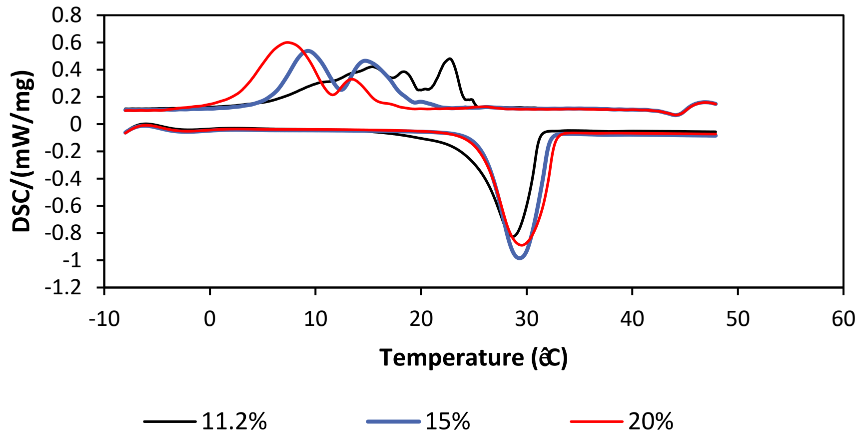
Results

SOLID CONTENT

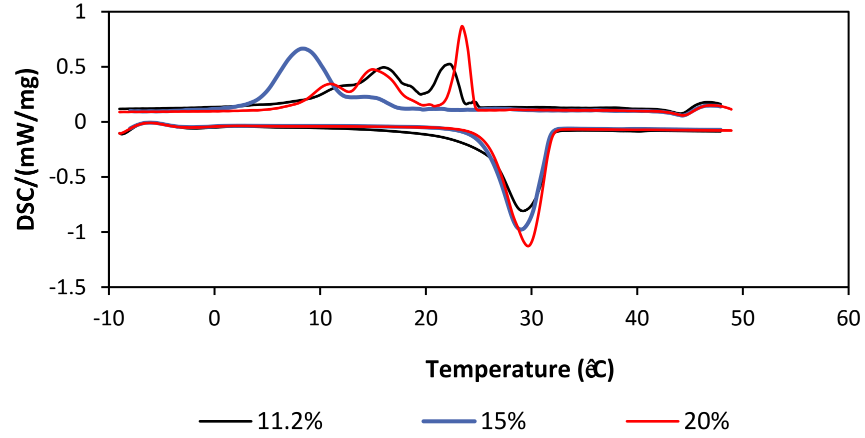
Thermal properties of microcapsules

Spray dyer	Solid content (wt%)	ΔH_m (J/g)	ΔH_c (J/g)
Lab-scale	11.2	77.7	75.5
	15	77.26	74.76
	20	81	77.4
Jumbo-lab-scale	11.2	79.37	77.83
	15	76.7	71.28
	20	82.74	79.54

Lab-scale



Pilot-scale



DSC curves

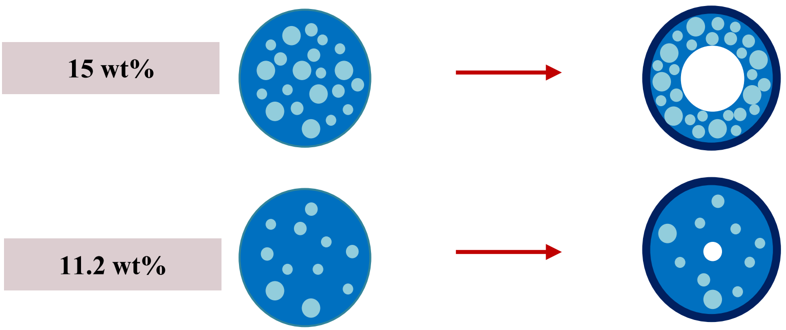
Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

Results

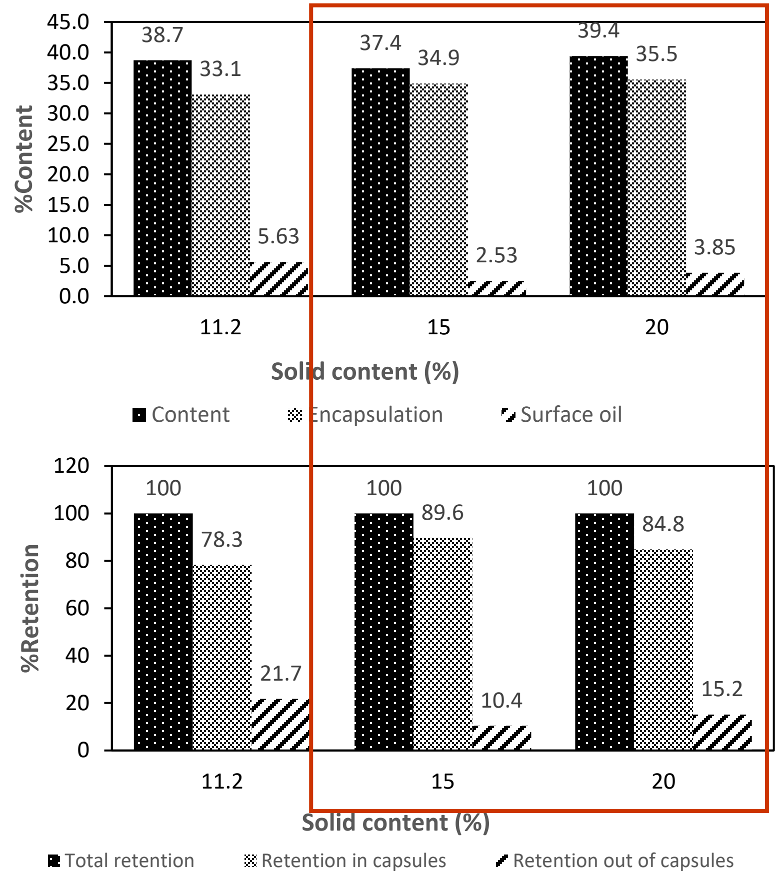
SOLID CONTENT

Thermal properties of microcapsules

Spray dyer	Solid content (wt%)	ΔH_m (J/g)	ΔH_c (J/g)
Lab-scale	11.2	77.7	75.5
	15	77.26	74.76
	20	81	77.4
Jumbo-lab-scale	11.2	79.37	77.83
	15	76.7	71.28
	20	82.74	79.54



Pilot-scale



PCM in microcapsules after spray drying in pilot scale
(a) %Content (b) %Retention

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

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When **particle size from the atomizer** and **ratio of water evaporation to solid content** were controlled equally in laboratory-scaled and pilot-scaled spray dryer, it can generate the microcapsules with similar properties including size distribution and PCM encapsulation.

The effect of **feed liquid flow rate** and **atomizer air pressure** in the atomization step

The effect of feed liquid flow rate, air inlet temperature

RECOMMENDTION

- The ratio of PCM : silica to enhance the encapsulation efficiency.
- The increasing quantities of feed preparation suitably for the pilot-scaled spray dryer.
- The others operating condition including air flow rate, influencing the microcapsules properties.

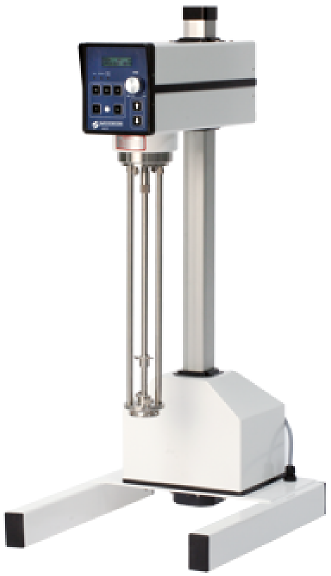
MIXER

Laboratory-scaled

Model T-25D Ultra-Turrax, IKA, German

- Maximum volume: 1 L

Jumbo laboratory-scaled



Siverson Model L5M Laboratory mixer

- Maximum volume: 12 L

Pilot-scaled

Silverson Model Verso Pilot scale in-line mixer

- Water flow rate: 2,400 L/h

SPRAY DRYER

Laboratory-scaled

B-290, Büchi Labortechik AG

- Water evaporation rate : 1 kg/h
- Production rate 0.20-0.50 kg/h

Jumbo laboratory-scaled

MOBILE MINOR™, GEA spray drying

- Water evaporation rate : 10 kg/h
- Production rate: 2-4 kg/h

Pilot-scaled

Spray dryer, Model SD-03

- Water evaporation rate: 50 kg/h
- Production rate: 10-20 kg/h

Production of Microencapsulated PCM by Pilot-Scaled Spray Dryer

Samples	ΔH_m (J/g)	ΔH_c (J/g)
Lab	97.75	94.22
Jumbo-Lab	101.43	95.65
Pilot Scale	109.80	103.69

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The production of encapsulated holy basil oil powder by different scale of spray drying process

The production of encapsulated holy basil oil powder by different scale of spray drying process

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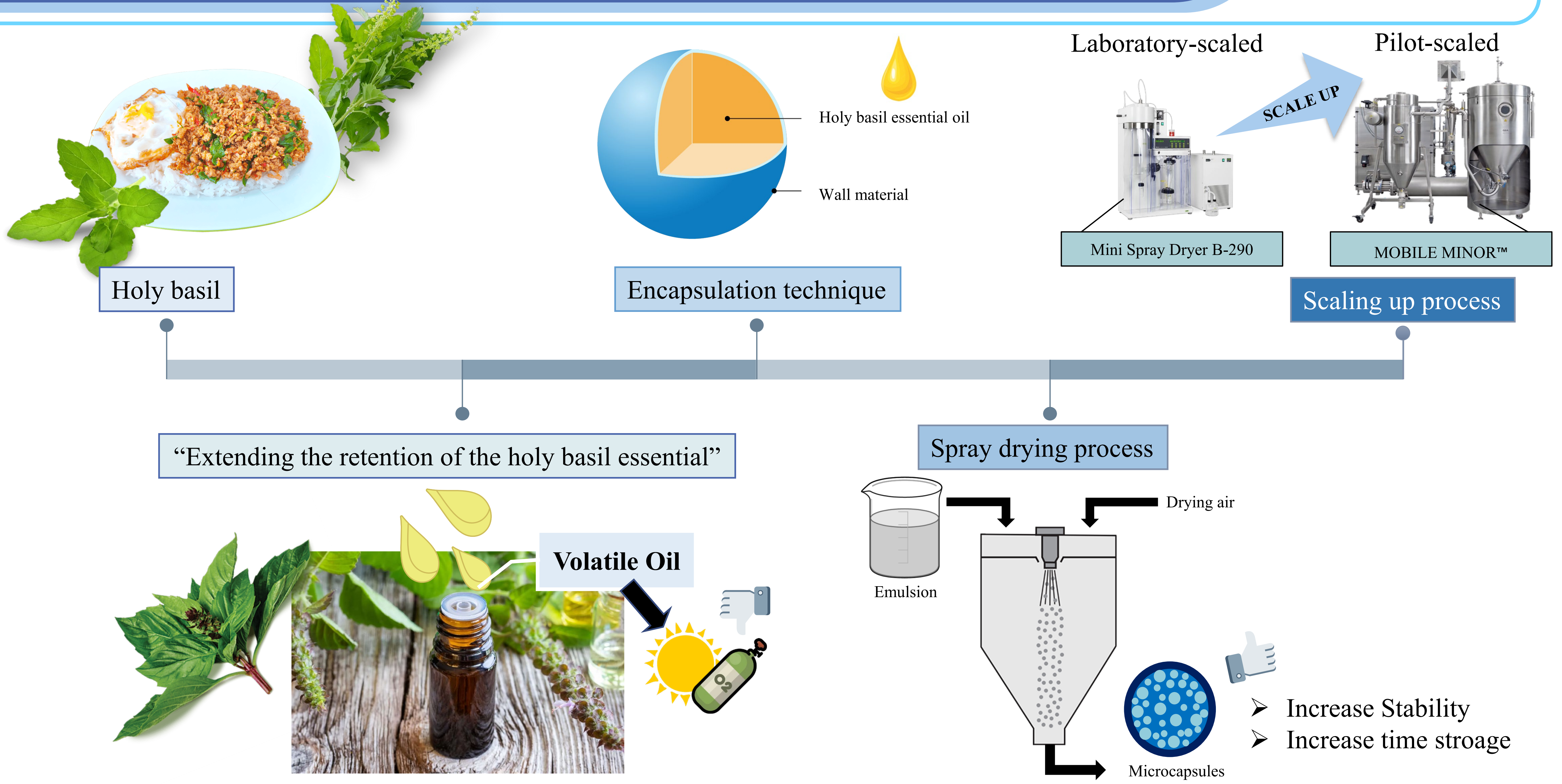
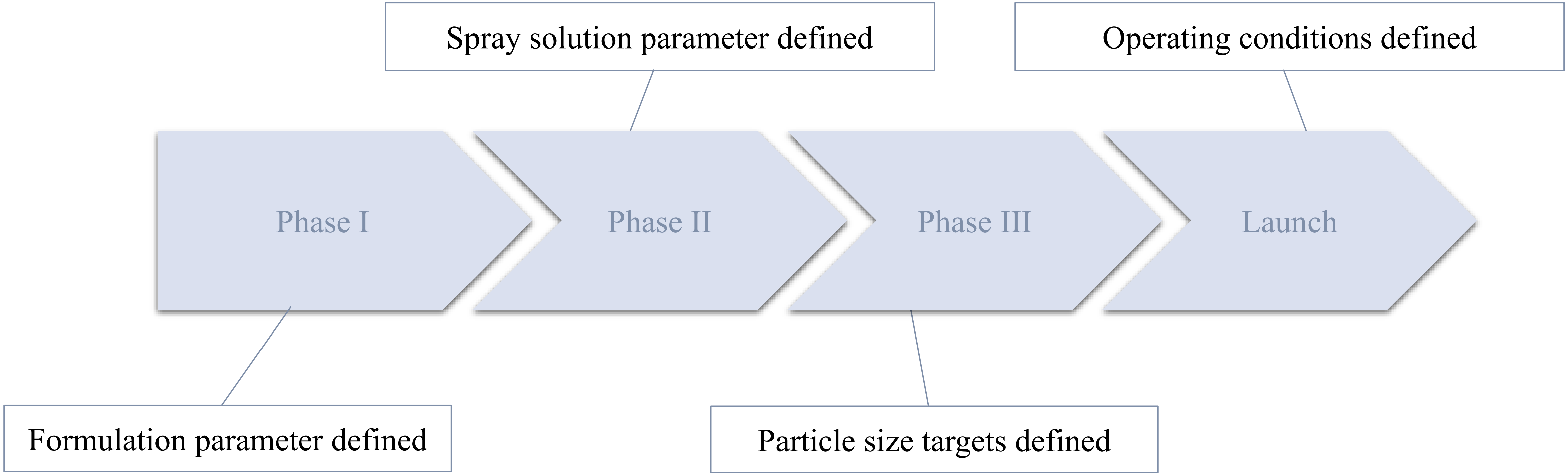


Spray Drying and Scale-Up Book

Dobry et al. (2009)

Graphically describes for method to scaling up process :



“as long as the blown-out rate from the atomizer and the ratio between the evaporated water and the solid substance in the drying chamber is kept consistent in both the laboratory and the scaled-up settings, the encapsulation power will be similar”

The production of encapsulated holy basil oil powder by different scale of spray drying process

Emulsion	Holy basil oil (active ingredient), modified starch (wall material), DI water (solvent) Holy basil oil : modified starch (w/w) = 1:4

Spray dryer model

Laboratory-scaled spray dryer

"Mini Spray Dryer B-290 (Büchi)"

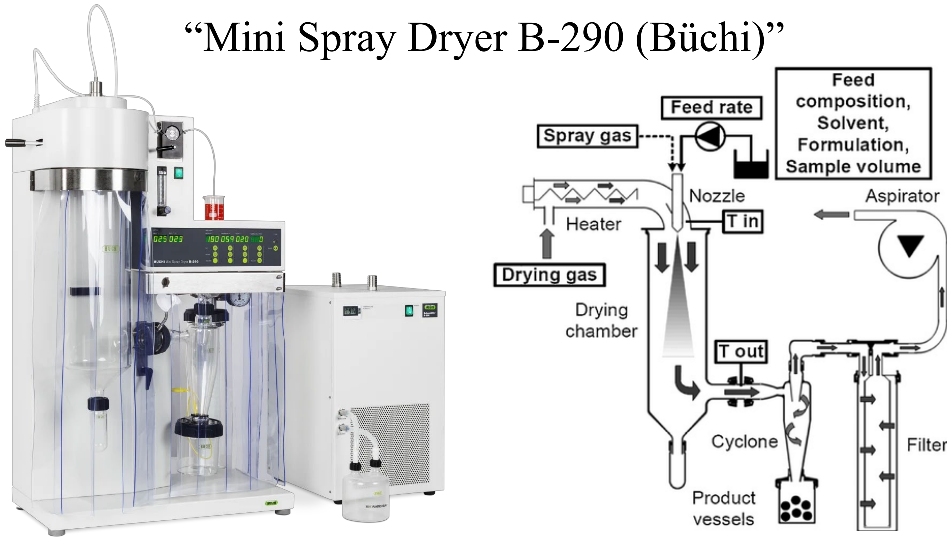


Figure 8 Mini Spray Dryer B-290 (Büchi) model

Reference : Jan P., Types of spray drying installations. Milk power manufacture, 2023

Table 1 Technical Feature of laboratory-scaled

Technical Feature	Büchi
Nozzle Types	Two-fluid nozzle
Evaporation capacity	1.0 kg/hr
Drying air flow rate	40 kg/hr
Achieved particle size	2 – 25 µm

Pilot-scaled spray dryer

MOBILE MINOR™ (NIRO)

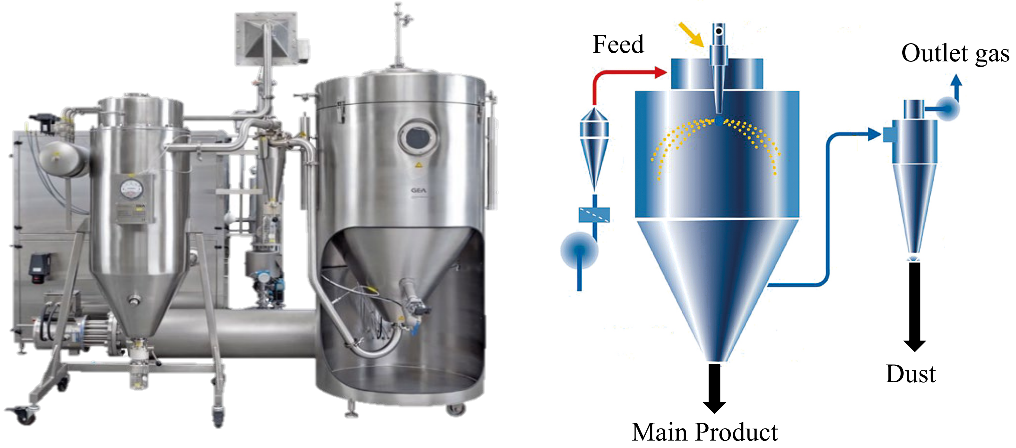


Figure 9 MOBILE MINOR™ (NIRO) model

Reference : Jan P., Types of spray drying installations. Milk power manufacture, 2023

Table 2 Technical Feature of Pilot-scaled

Technical Feature	NIRO
Nozzle Types	Rotary Atomizer
Evaporation capacity	0.5 - 6.0 kg/hr
Drying air flow rate	80 kg/hr
Achieved particle size	2 – 80 µm

Methodology

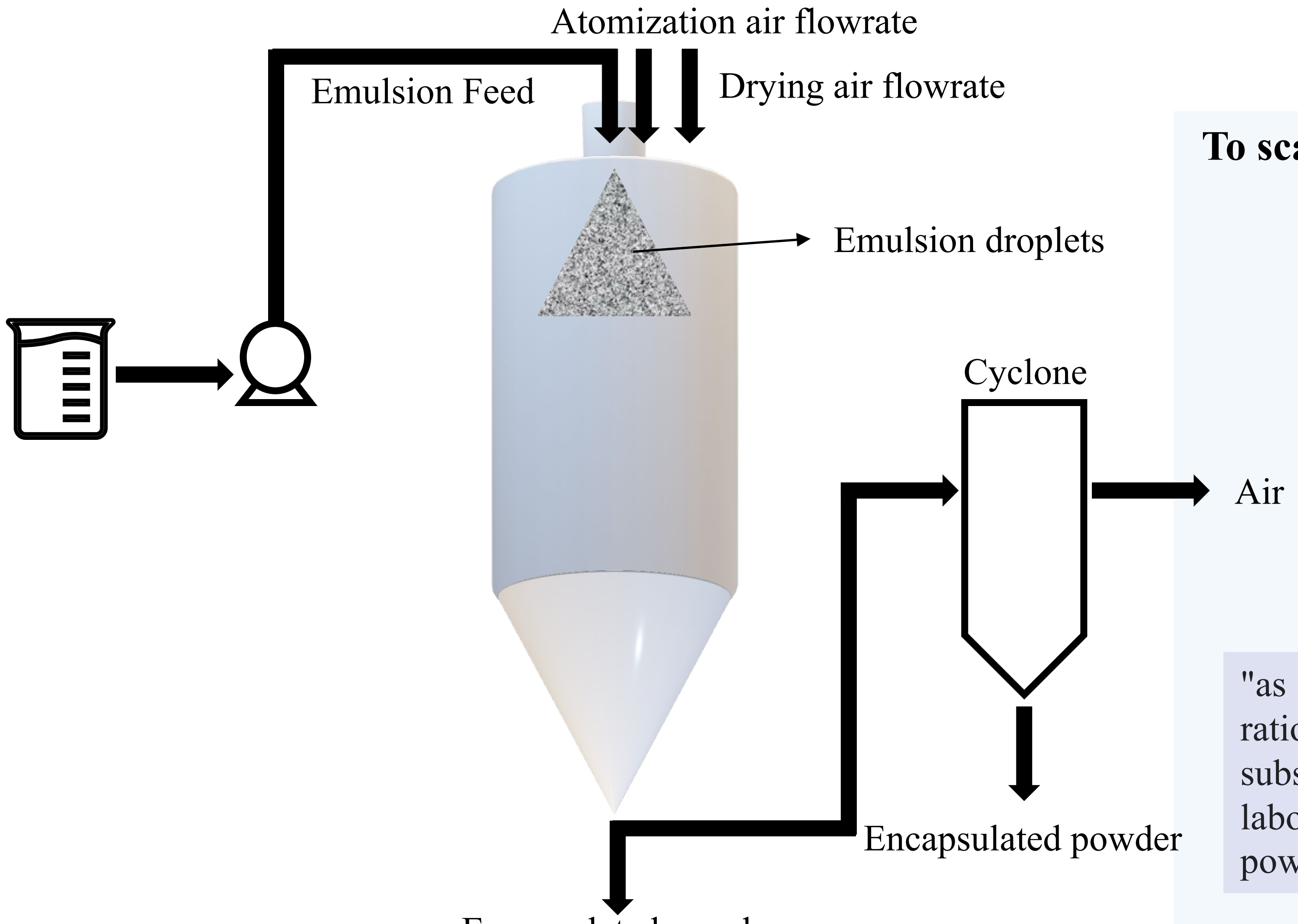


Figure 10 Spray dryer process diagram

Calculation part

To scaling up the spray drying

1. Spray emulsion by atomizer
   - Using air flow rate to specify size of encapsulated powder.
2. Drying emulsion by hot air temperature
   - Using inlet hot air temperature to evaporate the residual water from encapsulated powder.

"as long as the blown-out rate from the atomizer and the ratio between the evaporated water and the solid substance in the drying chamber is kept consistent in both laboratory and scaled-up settings, the encapsulation power will be similar."

Reference : E. Dobry, D., D. Settell, and J. Baumann, *Spray Drying and Scale-Up*. 2015. p. 315-340.

Methodology

Atomization air flowrate

Lab-Scale : Two-fluid nozzle

Pilot-Scale : Rotary Atomizer

To scaling up the spray drying

SECTION 1 : Spray emulsion by atomizer

Finding the appropriate atomizer air flow rate

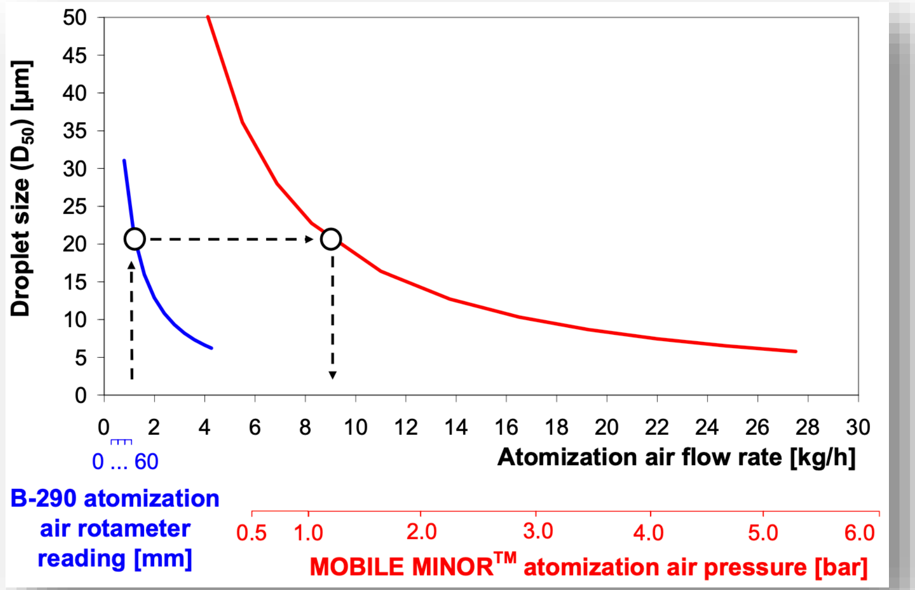


Figure 11 Correlation between particle size and atomizer air flowrate of B-290 and MOBILE MINOR

Reference : Thybo, P., et al., *Scaling up the spray drying process from pilot to production scale using an atomized droplet size criterion.* Pharm Res, 2008. **25**(7): p. 1610-20.

Methodology

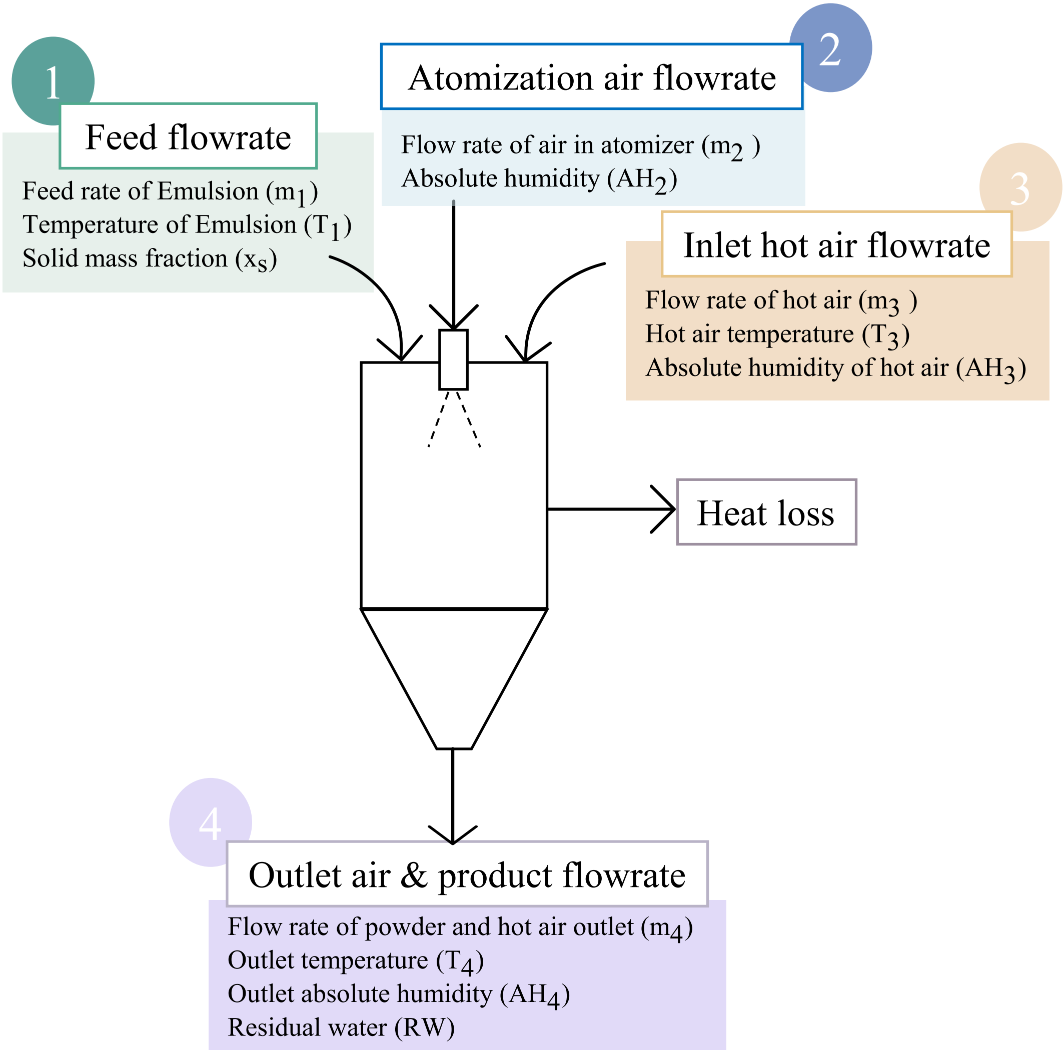


Figure 12 schematic diagram of the spray dryer

To scaling up the spray drying

SECTION 2 : Drying emulsion by hot air temperature

Mass balance

Residual water in the powder

$$\frac{m_3 AH_4}{1+AH_3} + \frac{m_2 AH_4}{1+AH_2} = \frac{m_3 AH_3}{1+AH_3} + \frac{m_2 AH_2}{1+AH_2} + m_1(1-X_s) - m_1 X_s \frac{RW}{1-RW}$$

Energy balance

Entering Energy :

Energy of inlet hot air : $m_3\,(C_{pAir}\,T_3 + AH_3(\Delta H_v + C_{pv} T_3))$

Energy of water : $m_1\,(1-X_s)\,.\,C_{pw}\,T_1$

Energy of slurry : $m_1\,X_s\,C_{ps}\,T_1$

Leaving Energy :

Energy of outlet hot air : $m_3\,(C_{pAir} T_4 + AH_4(\Delta H_v + C_{pv}\,T_4))$

Energy of product : $m_1\,X_s\,(C_{ps}\,T_4 + AH_4\,C_{pw}\,T_4)$

Energy loss :

Heat loss : $UA\,(T_4 - T_1)$

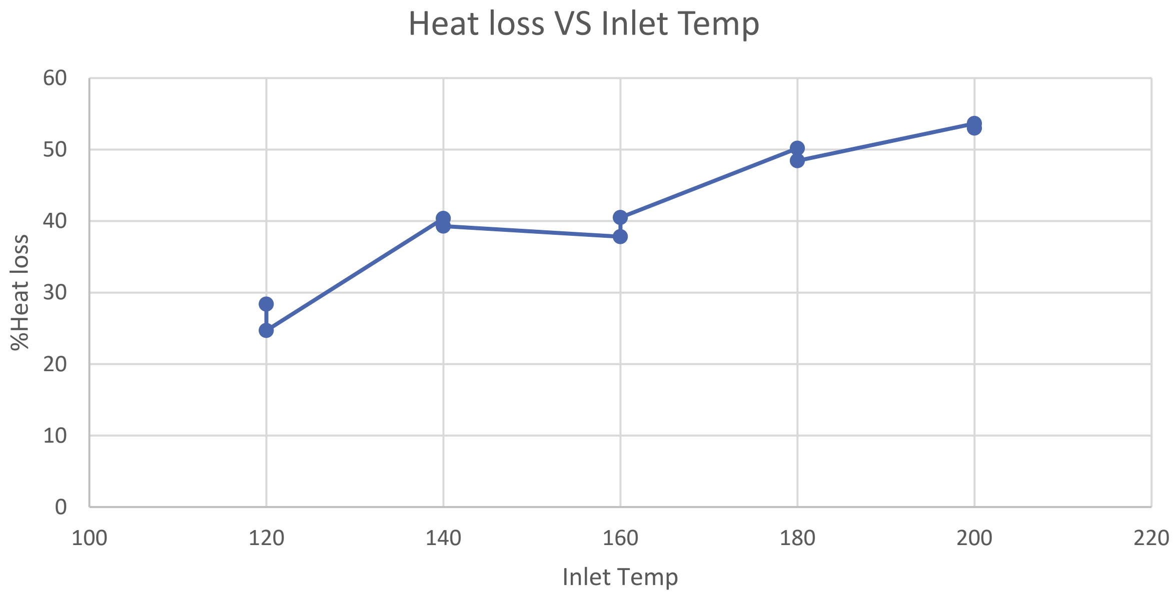
Entering Energy = Leaving Energy + Energy loss

Methodology

ChulaChE

Heat loss :

Using mass and energy balance to find heat loss.

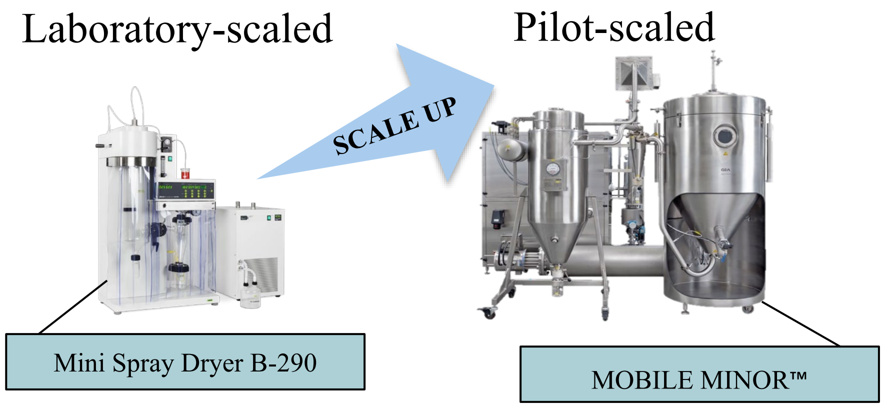


Feed rate ml/min	Inlet hor air temp °C	Outlet hot air temp °C	Heat loss kJ/hr	Hot air energy kJ/hr	%heat loss
28.2	120	78	1070	3769	28
28.2	120	80	886	3588	25
28.2	140	88	1842	4562	40
28.2	140	89	1758	4473	39
28.2	160	109	1692	4473	38
28.2	160	107	1883	4648	41
28.7	180	115	2796	5572	50
28.9	180	117	2617	5404	48
28.7	200	126	3241	6041	54
28.6	200	127	3160	5963	53

Methodology

Operating conditions for Pilot-Spray dry

		Bushi	NIRO
Feed rate	ml/min	12	35
Drying air flow rate	Kg/h	40	86
%heat loss			**40**
Tin	C	140	**140**
Tout start	C	89-91	89-91
Tout	C	73-76	84-85



Conclusion

To be done the propose of this project

Flowchart of scaling up process

Holy Basil Oils

↓

Encapsulation via spray drying in Laboratory scale

→ Collect the laboratory results for the next calculation step

↓

Finding the appropriate atomizer air flow rate

→ Get the pressure in atomizer

↓

Find the operating conditions process via mass & energy balance equation

→ Get the inlet tempurature

Scaling up the spray drying process

↓

Encapsulation via spray drying in Pilot scale

Q&A