The production of encapsulated holy basil oil powder via different scales of spray drying process.

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การผลิตผงห่อหุ้มน้ำมันกะเพราด้วยกระบวนการอบแห้งแบบพ่นฝอยที่มีขนาดแตกต่างกัน

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กระบวนการอบแห้งแบบพ่นฝอยเป็นกระบวนการมีการใช้งานอย่างหลากหลายทั้งในด้านของอุตสาหกรรมอาหาร ผงปรุงรส นมผง และยา ในงานวิจัยนี้จึงมีความสนใจในการศึกษาการขยายขนาดกำลังการผลิตของกระบวนการอบแห้งแบบพ่นฝอยจากระดับห้องปฏิบัติการไปยังระดับอุตสาหกรรมแต่ยังคงรักษาคุณสมบัติของผลิตภัณฑ์ที่ได้ออกมาจากระดับอุตสาหกรรมให้มีความคล้ายคลึงกับผลิตภัณฑ์ที่ได้จากห้องปฏิบัติการ สำหรับกระบวนการในการขยายขนาดในงานวิจัยนี้คือกระบวนการยืดอายุการกักเก็บของน้ำมันหอมระเหยจากต้นกะเพราด้วยเทคนิคเอนแคปซูเลชันหรือการนำสารสำคัญมาห่อหุ้มให้อยู่ในผงอนุภาคขนาดเล็กด้วยวิธีการอบแห้งแบบพ่นฝอยซึ่งเป็นการเปลี่ยนสภาวะของสารสำคัญจากของเหลว สารแขวนลอย หรืออิมัลชัน ให้เป็นสภาวะผงแห้งด้วยอากาศร้อน ซึ่งในการศึกษาการขยายขนาดจะแบ่งการศึกษาออกเป็น 2 ระดับ คือ ระดับห้องปฏิบัติการและระดับโรงงานต้นแบบ โดยจะดำเนินการจำลองกระบวนการโดยใช้โปรแกรม Aspen Plus ควบคู่กับการคำนวณจากสมการสมดุลมวลและพลังงาน ไปพร้อมกับการทำการทดลองในกระบวนการจริงเพื่อเพิ่มความแม่นยำในการดำเนินการขยายขนาด โดยการดำเนินการนี้อยู่ภายใต้สมมติฐานที่ว่า หากควบคุมให้อนุภาคพ่นฝอยที่ออกมาจากหัวฉีด, อัตราส่วนระหว่างน้ำที่ระเหยต่อปริมาณของแข็งในสายป้อนขาเข้า และ อุณหภูมิขาออกในเครื่องอบแห้งแบบพ่นฝอยขนาดห้องปฏิบัติการและขนาดโรงงานต้นแบบให้มีความเท่ากันนั้น จะทำให้คุณสมบัติของผงเก็บสารสำคัญที่ได้มีความใกล้เคียงกัน ซึ่งจากการจำลองกระบวนการในโปรแกรม Aspen Plus โดยเพิ่มอัตราการไหลของสายป้อน และเพิ่มขนาดของถังอบแห้ง พบว่าผลของคุณสมบัติที่ได้จากการคำนวณ, จากโปรแกรม Aspen Plus และจากผลการทดลองจริง มีค่าใกล้เคียงกันทั้งในด้านของความชื้นของผงเก็บสารสำคัญ, ปริมาณสารสำคัญที่คงเหลือในผงแห้ง และ ขนาดของผงเก็บสารสำคัญ

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The spray drying process is widely utilized in food manufacturing and pharmaceuticals for products like seasoning powder, milk powder, and medicine. This research investigates the scale-up of the spray drying process from laboratory to commercial scale with maintaining the product characteristic. A key aspect of the study is the encapsulation technique applied to prolong the release of essential oils from holy basil, encapsulating the essential substances (liquid, suspension, or emulsion) in nanoparticle form with hot air flow. The study utilizes the Aspen program, incorporating mass and energy balance calculations for simulation. The results, assuming similar particle size, water evaporation per solid content in feed, and outlet temperature in both laboratory and commercial spray drying scales, demonstrate a similarity in product characteristics across both scales Furthermore, A key finding is that increasing the input flow rate and reactor size results in similar outcomes at both scales, in terms of humidity of essential particles, the quantity of essential particles remaining in dried particles, and essential particle size. This successful scale-up process not only preserves the quality and characteristics of the encapsulated products but also opens avenues for industrial applications in maintaining the integrity of essential oils in various products.

Field of Study:	Chemical Engineering	Student's Signature ....
Academic Year:	2023	Advisor's Signature ....
		Co-advisor's Signature ....

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Chanakarn Chungmarisakul

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CHAPTER I Introduction and Background

1.1 Introduction

Most encapsulation research in Thailand generally begins with experiments on the laboratory scale, in order to save the raw material usage and limit the size of experimental instruments involved. However, if those lab-scale experiments are deemed successful, with the end product being favorable enough that the demand of it has also increased, the production rate of the process shall also be escalated in accord. For the encapsulation process in particular, in order to satisfy the elevated demand, it is necessary to increase the feed rate of the process. That being said, since the variation of a single parameter does affect the entire operating condition as a whole, it is important to carefully curate the method of scaling up, in ways that the end product shall be kept desirable, this can also translate to a further development in the industrial and commercial levels for the parties involved. In this study, the process of scaling up i.e., from the laboratory to the pilot scale, and the industrial scale, shall be focused on the encapsulation of holy basil essential oils by using spray drying which is a common process that has been widely adopted in the food industry[1, 2].

Encapsulation is a process in which active agents, essential oils in this case, are surrounded by a coating called wall materials and collected in the form of capsules [3]. The wall materials will prevent the essential oils from unwanted environmental conditions, as mentioned above, which is an effective way of prolonging the agent's lifespan[3, 4]. The methodology of encapsulating a substance in this research is called "spray drying". Spray drying is a process which turns substances in liquid phase i.e., a solution, a colloid, or a suspension/emulsion, to a solid phase, in the form of powder, using hot air to evaporate the water from the solution and encapsulate the substance inside[5]. The spray drying process is a common process that has been widely adopted in the food and agricultural industries - milk powder, baby foods, and medicine production - due to its eco-friendly characteristics and zero toxic by-product. The process also allows for an adjustable size of microcapsules and improving the solubility, dispersibility, and stability of food product afterward [2].

There is a substantial amount of research involved in scaling up a spray drying process from small scale to large scale. For instance, the study by Thybo et al. focused on the encapsulation process utilizing acetaminophen as an active ingredient and PVP-K30 as an excipient in Ethanol. This research aimed to investigate the feasibility of scaling up the pilot scale to the production scale by attempting to match the droplet size using two different-sized atomizers [6]. Although the particle size from the pilot scale is similar to that of the production scale, there were disparities in the particle morphology and the water residual in the particle between the two scales. Consequently, scaling up based solely on matching droplet size was not viable. Another example of research on scaling up the spray drying process is the production of H1N1 influenza hemagglutinin vaccine by Zhu et al. The objective of this research was to vary the air flow ate in an atomizer and match the inlet and outlet temperature of two different spray dryer scales. The particle from the laboratory scale spray dryer is comparable to the particle from the pilot scale spray dryer [7]. However, maintaining both inlet and outlet temperatures from the laboratory and pilot scale equally, the drying air flow rate of the large-scale process must be fine-tuned.

Most scale-up process research is driven by trial and error in the operating conditions and checking the outgoing product quality. Therefore, this research aims to utilize thermodynamic and simulation programs to determine the optimal operating conditions for the larger scale spray drying process instead of trial-and-error-based approaches.

The study on the scaling-up process of essential oil encapsulation via spray drying can be approached by examining two key components: the atomizer and the temperature of the hot air inlet [7, 8]. The atomizer is evaluated in terms of particle size, while the temperature of the hot air inlet is evaluated in terms of the heat required to evaporate water from the emulsion droplets sprayed from the nozzle. This methodology was proposed by Dobry et al. in their research, which stated that "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." [8]. In this thesis, the presumption posited by Dobry et al will be employed to scale up the spray drying process. This entails incorporating the concept derived from practical processes, emphasizing the imperative of sustaining both inlet and outlet temperatures. To test this assumption, the feeding rate of emulsion shall be varied.

1.2 Research objectives

1.2.1. To scale up the production of holy basil oil encapsulated powder via spray drying.

1.3 Research scope

1.3.1. Determine the specification of laboratory and pilot scale spray dryer.

The spray dryer model of laboratory scale is mini spray dryer B-290:

- Nozzle type : two-fluid nozzle
- Evaporation capacity 1 kg/h
- Drying air flow rate 40 kg/h
- Achieved particle size 2-25 µm

The spray dryer model of pilot scale is MOBILE MINOR™:

- Nozzle type : two-fluid nozzle, Rotary atomizer
- Evaporation capacity 0.5 - 6.0 kg/h
- Drying air flow rate 80 kg/h
- Achieved particle size 2-80 µm

1.3.2. Determine the ratio of emulsion substance sprayed in spray dryer at different scales.

- Core material : Holy basil oil
- Wall Material : Maltodextrin

The ratio between holy basil oil and modified starch is 1:4

1.3.3. Determine the operating conditions for the pilot scale. The parameters that vary for pilot scale are shown below:

- Feed flow rate 0.8 – 2 kg/h
- Drying air flow rate 50-80 kg/h
- Pressure of flow rate air in atomizer 5-6 bar

1.3.4. Study the chemical and physical characteristic of encapsulated powder after scaling up the process.

The physical characteristic of encapsulated:

- Encapsulated powder size should be in range 10-12 micron.
- Moisture content of encapsulated powder should not more than 5 percent weight by weight.
- Microcapsule structure observed by SEM.
- Bulk density

The chemical characteristic of encapsulated:

- Component retention in the encapsulated powder.

1.4 Expected benefits

1.4.1. The properties of encapsulated powder from the actual scaled-up is as same to the laboratory scale result method as possible.

1.4.2. Utilizing the Aspen Plus program to simulate the large-scale spray drying process, as part of the investigation into determining the proper drying air flow rate.

1.5 Flow chart

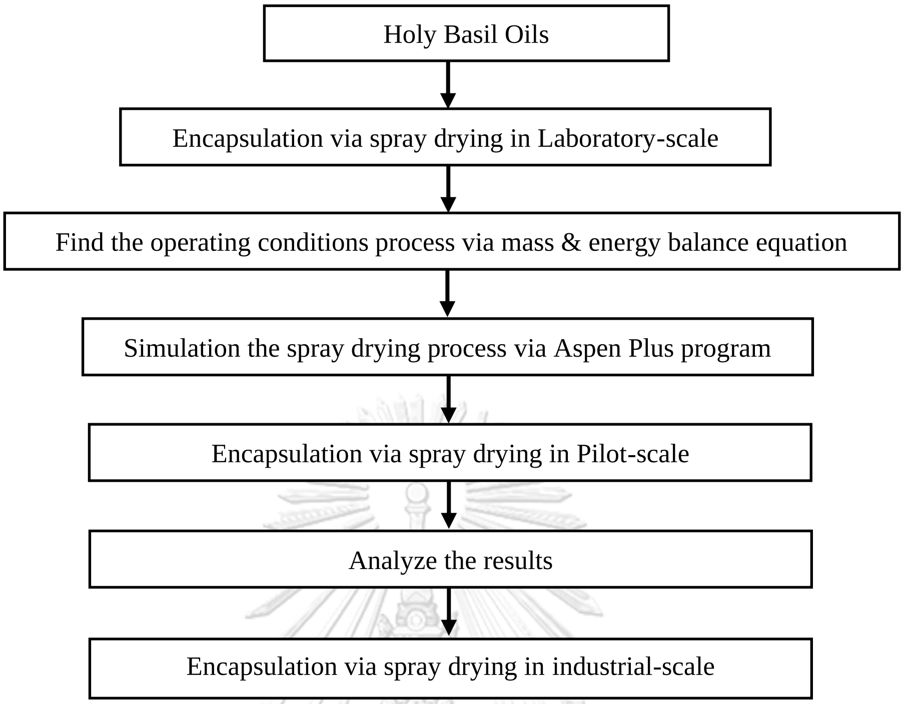


Figure 1 Work flow diagram

CHAPTER II Theory & Literature Review

2.1 Holy basil oil

Holy basil is an herb that has a unique smell and many beneficial medicinal properties, such as lower the body's level of cholesterol, ease inflammation, reduce bloating and painful bowel movements, etc. With those benefits, people commonly consume holy basil in several ways, whether it be as food or medicine. Many chemical substances can be found in holy basil, and one of the most important ones is a volatile oil, for instance, Eugenol, a versatile substance naturally found in essential oils. There are numerous pharmacological properties in eugenol, i.e., it could be used as a treatment for asthma, and it is also an antioxidant [1]. However, due to the in-stabilized nature of essential oils that can easily evaporate and degenerate in oxidation-prone conditions or various environmental factors, including sunlight, oxygen level, and more. Therefore, this project aims to figure out a way of collecting holy basil extract, in the form of encapsulated powder, using encapsulation technology to prevent degeneration and extend the lifespan of the holy basil essential oils [9].

2.1.1 Eugenol

Eugenol (4-allyl-2-methoxyphenol) is a natural aromatic compound belongs to the class of phenylpropenes as shown the molecular structure in Figure 2. It is a colorless to pale yellow, oily liquid that is extracted from certain essential oils such as clove oil, nutmeg, cinnamon, and holy basil. Eugenol has a spicy, clove-like aroma and taste, and it is widely used as a flavoring agent in foods and beverages [2].

In medical term, eugenol also has several medicinal properties and has been used for centuries as an analgesic, antiseptic, and anti-inflammatory agent. It is often used in dentistry as a local anesthetic and is also used as a natural remedy for toothache, sore throat, and other oral health problems [2]. In addition, eugenol has been found to have antioxidant and antimicrobial properties, making it a popular ingredient in cosmetic and personal care products such as soaps, lotions, and perfumes. It has also been studied for its potential anti-cancer effects.

Regarding encapsulation process, there is research related to encapsulating the eugenol via spray drying method and utilizing the mixed whey protein or soy lecithin with maltodextrin as the wall materials. This result is indicated the higher encapsulation around 95-98 % when the ratio of maltodextrin and whey protein or soy lecithin is equal to 1:42 weight by weight [10].

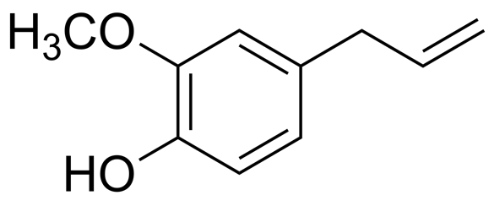


Figure 2 molecular structure of eugenol [2].

2.1.2 Linalool

Linalool is a terpene alcohol that occurs naturally in various essential oils, including but not limited to lavender, coriander, and holy basil. This colorless to pale yellow liquid has a floral aroma and a sweet, spicy taste, and is widely used in the perfume and fragrance industry, as well as in the production of personal care products and cosmetics[11]. The molecular structure of linalool is demonstrated in Figure 3.

Linalool has also been found to possess medicinal properties, including anti-inflammatory, analgesic, and sedative effects [11]. It has been utilized as a natural remedy for stress, anxiety, and sleep disorders, and has been incorporated into the practice of aromatherapy and massage therapy. Furthermore, linalool displays antioxidant and antimicrobial characteristics, and has been investigated for its potential anti-cancer properties. It is also used as a flavoring agent in the food industry and in the production of alcoholic beverages [12].

In term of encapsulation, there is research related to encapsulate the linalool of bergamot oil by spray drying method and using HI-CAP®100 which have the high retention to keep the core materials as a wall materials of the encapsulated powder [12].

Figure 3 molecular structure of linalool [13].

2.1.3 Methyl eugenol

Methyl eugenol is a naturally occurring organic compound that is found in a variety of plants, such as nutmeg, clove, holy basil, and other spices. It is a colorless to pale yellow liquid with a sweet, spicy odor that is commonly used as a flavoring agent in foods, beverages, and fragrances. Methyl eugenol is also used as a pesticide and pheromone in agriculture, particularly in the control of fruit flies. However, it has been found to be toxic to some animal species, including humans, when consumed in large quantities. For this reason, regulatory agencies such as the United States Environmental Protection Agency have placed restrictions on its use in certain applications[14]. The molecular structure of methyl eugenol is shown in Figure 4.

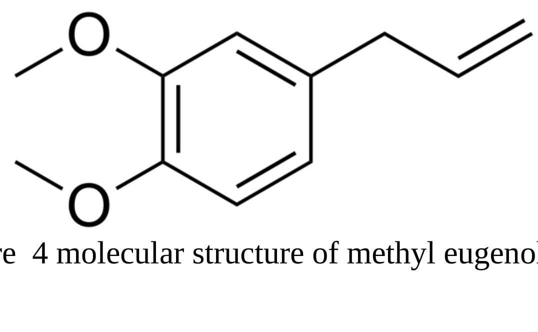


Figure 4 molecular structure of methyl eugenol [15].

2.1.4 Beta-caryophyllene

Beta-caryophyllene is a sesquiterpene, a naturally occurring organic compound that is found in various plants, including black pepper, cloves, hops, and holy basil. It possesses a distinct spicy and peppery aroma, which is responsible for the flavor and fragrance of black pepper and cloves. One of the most notable features of beta-caryophyllene is its affinity to the CB2 receptor in the human body[14]. As a result, it exhibits pharmacological activity similar to that of cannabinoids, including reducing inflammation and pain. The potential use of beta-caryophyllene in treating anxiety, depression, and addiction is also under investigation [15]. The molecular structure of linalool is demonstrated in Figure 5.

In the food and beverage industry, beta-caryophyllene is widely used as a flavoring agent. It is also utilized as a natural insecticide and food additive. Furthermore, it has applications in the cosmetics industry, where it is incorporated into perfumes and other cosmetic products for its fragrance properties [14].

Regarding the encapsulation research of beta-caryophyllene, there is studied about the encapsulation of essential oil from pterodon emarginatus via spray drying. Utilizing the Gum Arabic and Maltodextrin as a wall materials to encapsulated essential oils which have the beta-caryophyllene as a main substance. The results of this research found that the ratio between the emulsion to Gum Arabic to Maltodextrin is 1 to 3 to 3.6 with the operating conditions; inlet of hot air temperature is 160 °C, and flow rate of emulsion is 4 milliliter per minute gave the most encapsulation efficiency which equal to 98.63% [3].

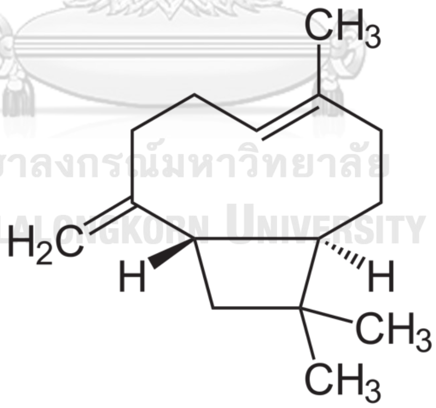


Figure 5 molecular structure of Beta-caryophyllene [16].

2.2 Encapsulation techniques

Encapsulation is a process in which active agents, holy basil essential oils in this case which is called core materials, are surrounded by a coating called wall materials or shell and collected in the form of encapsulated powder as shown in Figure 6 [16, 17]. The wall materials will prevent the essential oils or core materials from degradation and unwanted environmental conditions, such as oxygen, sun light, heat, as mentioned above, which is an effective way of prolonging the agent’s lifespan, enhance its stability, control its release and protection during storage and transportation [1, 17]. And for the methodology of encapsulating a substance is called “spray drying”.

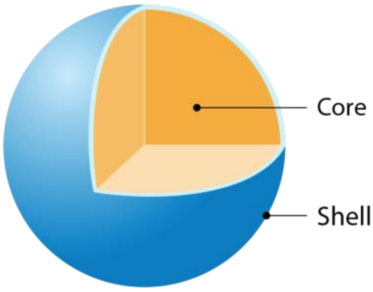


Figure 6 Encapsulated powder structure [18].

The methodology of producing encapsulated powder could be divided into two main steps:

1. Creating a thin film around the particle or the process of emulsification.

This could be achieved by using a Homogenizer to continuously stir the coating substance and the holy basil essential oil until the stratified mixture is into an emulsion [17], which is then measured for size before going to step 2.

2. Drying out the emulsion using the spray drying method.

The drying out emulsion method, where the emulsion is put through a drying chamber while being blown out by an atomizer. Turning it into minuscule particles coming in contact with the chamber’s hot dry air, resulting in rapid water evaporate from the emulsion droplet, and finally giving out a completely dried-out powder.

2.3 Spray drying

Technique of the encapsulation can be divided into 2 type; the physical method and the chemical method, each of type can provide different shape and size of the encapsulated powder. Regarding the methodology of encapsulating a substance in this case is called “spray drying”. Spray drying is a process which turns substances in liquid phase i.e., a solution, a colloid, or a suspension/emulsion, to a solid phase, in the form of powder, using heat from hot air to evaporate the water from the solution droplet and encapsulate the substance inside [9, 17]. The spray drying process is a common process that has been widely adopted in the food, pharmaceuticals and agricultural industries - milk powder, baby food, and medicine production - due to its eco-friendly characteristics and zero toxic by-product [9, 17, 18]. The process also allows for an adjustable size of microcapsules and improving the solubility, extended shelf-life, dispersibility, and stability of food product afterward [5].

Figure 7 demonstrated the formation of encapsulated powder inside the spray drying chamber. Starting from the emulsion being fed into the atomizer spray, blow out in form of emulsion droplets. The moisture content of the droplets will evaporates soon after due to the heat coming into the chamber via another feed line, which causes the rapid formation of the shell encapsulated powder after the spraying [8, 19].

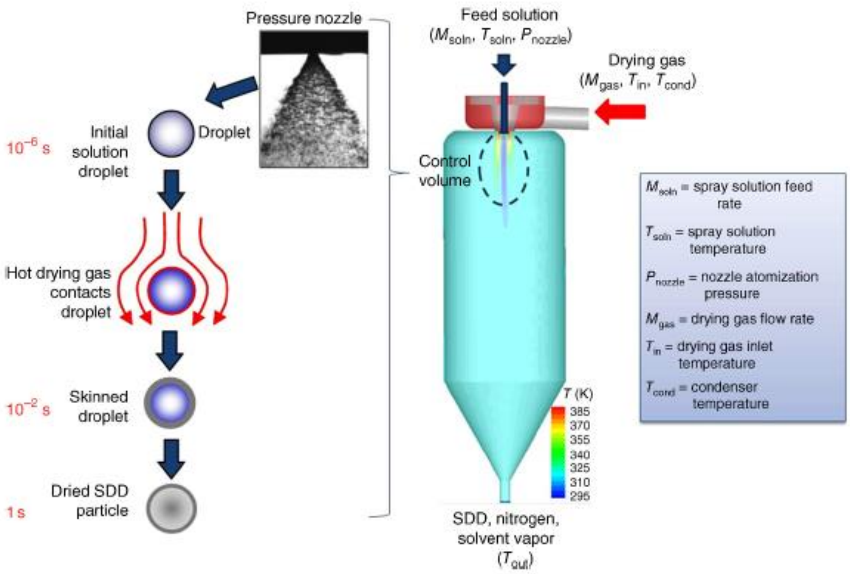


Figure 7 the formation of encapsulated powder inside the spray drying chamber [8].

There are 4 simple step method of the encapsulation via spray drying, the number of each step is demonstrated in Figure 8.

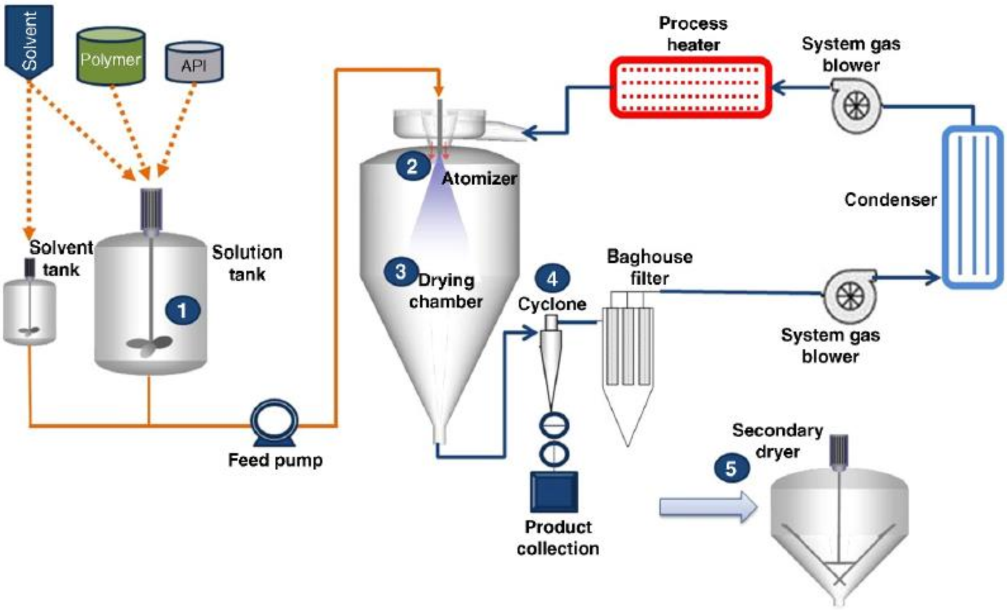


Figure 8 The diagram of spray drying method [6].

2.3.1 Preparing the emulsion

The emulsion could be achieved by using a Homogenizer to continuously stir the wall material materials and the core materials which is the holy basil essential oil in this case until the stratified mixture is into an emulsion [20].

2.3.1.1 wall materials

For the core materials of encapsulation technique in this project is holy basil essential oil, and for the wall materials, it is depend on the encapsulation process. The wall materials act as a protective barrier around the core material to preventing its degradation and controlling its release [9, 20, 21]. The following are some of the important properties of the wall materials in encapsulation technique [4, 22]:

1. Biocompatibility: If the wall encapsulated material is intended for use in foods, pharmaceuticals, or medical applications, the wall material must be biocompatible and non-toxic.
2. Permeability: The wall material should have the appropriate permeability to allow the encapsulated material to be released at the desired rate. The permeability can be controlled by adjusting the thickness of the wall or using different types of polymers.
3. Mechanical stability: The wall material should have sufficient mechanical stability to protect the core material from degradation during processing and storage. The stability of the wall material can be enhanced by cross-linking or by using more rigid polymers.
4. Adhesion: The wall material should adhere well to the core material to prevent the core from leaking out or reacting with the environment.
5. Solubility: The solubility of the wall material is important for the release of the core material. If the wall material is water-soluble, the encapsulated material will be released when exposed to water, whereas if it is insoluble, the release will be slower.
6. Chemical and thermal stability: The wall material should be chemically and thermally stable to prevent any unwanted reactions or changes in the properties of the encapsulated material.
7. Compatibility with processing methods: The wall material should be compatible with the processing methods used for encapsulation, such as spray drying or coacervation.

The wall materials which used to coat the core materials in this project is HI-CAP®100, which a modified-starch is used in various food and medication industries because its properties in term of the encapsulated substances are almost 80% and improve the protection feature at high temperature. In 2011, there are research about encapsulation the volatile compounds of bergamot via spray drying method using HI-CAP®100 as a wall material. The result shown that in suitable operating conditions of spray drying, it was confirmed that the HI-CAP®100 gave the high retention of core materials, have a high encapsulation efficiency approximately 93.05% , and gave a great protective oxidation from oxygen [23].

2.3.2 Spray the emulsion via atomizer

The emulsion is blown out in from of droplet via atomizer in form of emulsion droplet to increase the heat transfer surface area for evaporate the water from the emulsion droplet [8].

2.3.3 Evaporated the water remaining in the droplet

The moisture content of the droplets from the atomizer spray will evaporates via heat from hot air feed line to format the shell encapsulated powder after the spraying.

2.3.4 collect the encapsulated powder

The encapsulated will be separated from the leaving hot air line by a cyclone before being dropped into the glass jar.

2.4 Scaling up the encapsulation process via spray drying

In 2015, to achieve the operating conditions when scaling up the encapsulation process. The assumption from a research by Dobry et al. will be used. It was stated in the research that, "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 in both a lab-setting and a scale-up setting are kept consistent, the encapsulated power of either scales will also turn out similar [8]." Meaning that if we want the scale up process to give out a similar product as the laboratory one, the right value of the 2 parameters: atomizer's blown-out rate and the evaporated water to solid ratio, must be achieved. Therefore, the methodology for scaling up the encapsulation process from the lab-scale to the pilot-scale and industrial-scale can be divided into two parts. The first part is the size of the powder which involves examination on various factors, such as the speed of the nozzle, the type of atomizer used, and the size of the nozzle. The second part involves the amount of water evaporated from the emulsion droplets from the emulsion droplets, which is accomplished by determining the temperature of the hot air outlet. Moreover, the amount of water evaporated from the emulsion droplets can only be used to determine the moisture content of the dry powder at the outlet [6, 8].

2.4.1 Specify the encapsulated powder size via atomizer

With the outlet powder size taken into consideration, the nozzle head is one of the vital factors that need to be adjusted to scale up from the laboratory scale to the larger scale [8].

2.4.1.1 The correlation between droplet size and atomizer air flow rate

Regarding to determine the suitable nozzle head size of the pilot-scale, the operation shall start from finding out the air flow rate in the pilot plant nozzle that is able to produce the desired laboratory sized powder via vary the flowrate of nozzle air with the particle size and generate the correlation between droplet size and atomizer air flow rate of each atomizer types. The graph shown in

Figure 9 is demonstrated the correlation between atomization air flow rate and droplet size of two-fluid nozzle atomizer which is atomizer of laboratory scale and rotary atomizer which is the atomizer of pilot scale. It was found that the atomizer air flow rate is affect to the particle size. When the atomizer air flow rate is increased, it also increase shear force between air flow and the emulsion droplet. Therefore, the encapsulated powder size will be decreased [24].

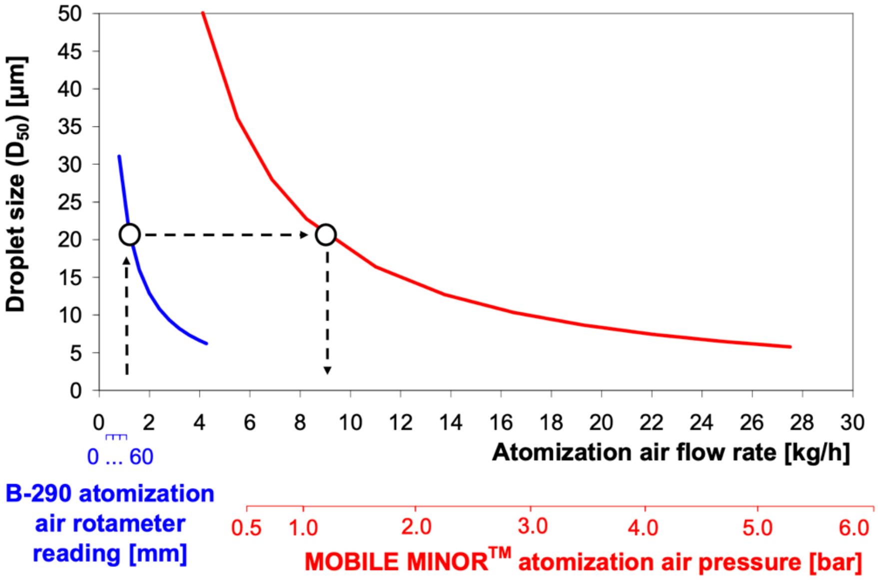


Figure 9 correlation between droplet size and atomizer air flow rate [19].

2.4.1.2 Rosin-Rammler-Sperling-Bennet (RRSB) equation

Equations for powder particle size distribution estimation from RRSB distribution model as shown in equation (1) are also used together with Aspen plus program for size prediction of the output particles. The data from the calculations and Aspen simulation will then be used as input in the spray dryer machine, to verify both the calculated and simulated results [25].

$$Q(d) = 1 - e^{-\left(\frac{d}{d_{63}}\right)^{n}} \qquad (1)$$

Where n is dispersion parameter, d is particle size, and d_{63} is particle size parameter of the RRSB distribution.

2.4.2 Evaporation the remain water from the emulsion droplet

When the size of the dry powder from the spray nozzle is identical between the laboratory scale and the pilot scale, the next step is to determine the appropriate operating conditions in the pilot plant spray dryer via mass and energy balance equation around the dryer by using assumption a constant absolute humidity of inlet and outlet hot air.

2.4.2.1 Mass and Energy balance

In this study, attention of the operating conditions must be given to the temperature of the hot air inlet, to control the output encapsulated powder humidity as well as ensuring that it closely resembles the moisture leaving the laboratory settings. Figure 10 demonstrated the schematic diagram of spray dryer system. It was found that The spray dryer machine consists of 3 incoming lines: the feed entering the dryer, the hot air entering the nozzle, and the line for hot air intake to the chamber. There is only one outlet line, which is the product outgoing line where air and dry powder are mixed.

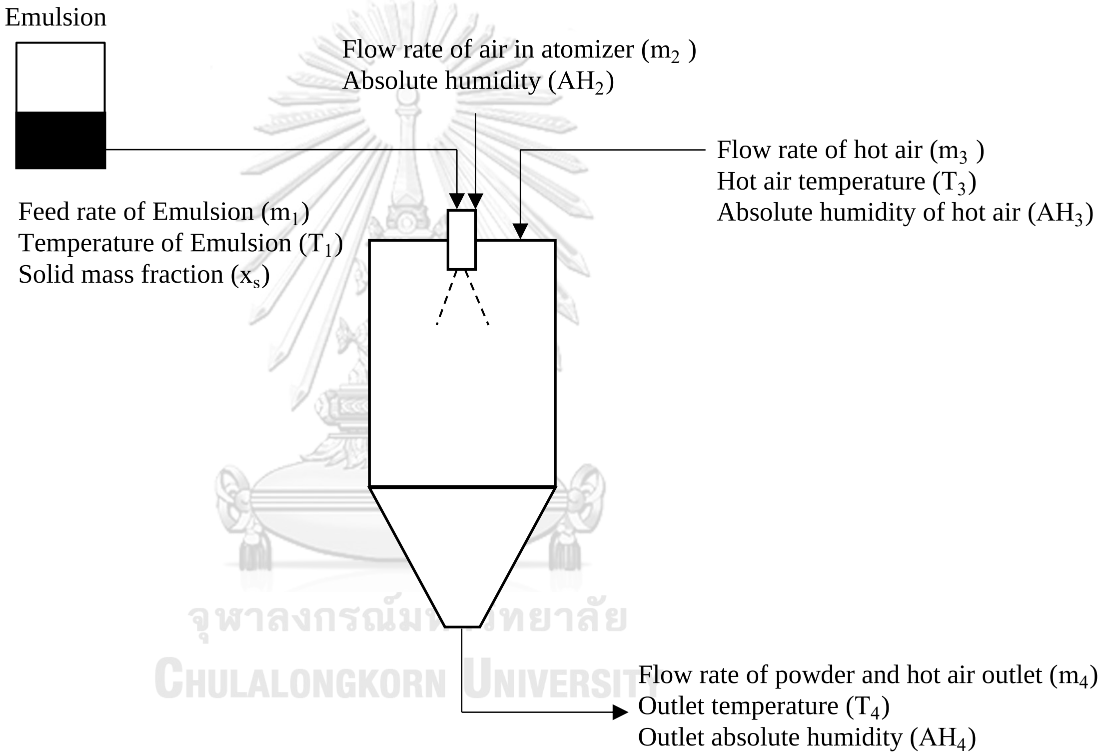


Figure 10 schematic diagram of spray dryer.

The relevant variables used in the calculations are shown in the schematic diagram of the spray dryer in Figure 10. Where m is flow rate in each stream (kg/hr), x_s is solid mass fraction, AH is absolute humidity (kg water/kg dry powder), T is temperature (°C), ΔH_v is latent heat of vaporization, C_p is specific heat capacity(kJ/kg.K), U is overall heat coefficient(W/m^2°C), A is heat transfer area (m^2) and RW is water remain in encapsulated powder (kg water/kg powder).

Mass balance equation is consist of mass input: feed rate of emulsion, flow rate of air in atomizer, and flow rate of hot air and mass output: flow rate of encapsulated powder [26]. Therefore, the mass balance equation can be written in form of equation (2).

$$\frac{AH_4}{1-AH_3} m_3 + \frac{AH_4}{1-AH_2} m_2 = \frac{AH_3}{1-AH_3} m_3 + \frac{AH_2}{1-AH_2} m_2 + m_1 (1 - x_s) - m_1 x_s \left(\frac{RW}{1-RW} \right) \quad (2)$$

Energy balance equation is derived from the law of energy conservation which consist of 3 part: the energy incoming to the dryer, the energy leaving to the dryer, and the energy loss of the dryer [26].

Entering energy:

- Energy of air stream: $m_3 (Cpa\, T_3 + AH_3 (\lambda + Cpv\, T_3))$
- Energy of water stream: $m_1 (1-x_s)\, Cpw\, T_1$
- Energy of emulsion: $m_1 x_s\, Cps\, T_1$

Leaving energy:

- Energy of air stream: $m_3 (Cpa\, T_4 + AH_4 ((\lambda + Cpv\, T_4))$
- Energy of emulsion: $m_1 (Cps\, T_4 + (1- x_s)\, Cpw\, T_4)$

Therefore, the energy balance of spray dryer is demonstrated in (3)

$$m_3 (Cpa\, T_3 + AH_3 (\lambda + Cpv\, T_3)) + m_1 (1-x_s)\, Cpw\, T_1 + m_1 x_s\, Cps\, T_1 = m_3 (Cpa\, T_4 + AH_4 ((\lambda + Cpv\, T_4)) + m_1 (Cps\, T_4 + (1- x_s)\, Cpw\, T_4) + UA(T_4 - T_1) \quad (3)$$

For the evaluation of the required pilot plant operating conditions, the mass and energy balance equations shall be used. This calculation is based on the assumption that the ratio of the evaporated water to solids in the laboratory scale and the pilot plant scale feed lines are similar.

2.5 Aspen Plus Version 11

Aspen Plus is a renowned process simulation software utilized in a diverse range of industries, such as oil and gas, chemical engineering, and pharmaceuticals. Its primary function is to simulate and model chemical processes virtually, enabling engineers to analyze various scenarios, optimize processes, and troubleshoot issues before implementation in the real world. The simulation process in Aspen Plus involves constructing a process flow diagram (PFD) that outlines the process components, including equipment, streams, and chemical reactions. The PFD is then complemented by the input of physical and thermodynamic properties of the process components, and process conditions such as temperature, pressure, and flow rate are defined. Aspen Plus offers an extensive array of analysis tools that facilitate the evaluation of the process model's performance. These tools can identify potential bottlenecks, optimize process conditions, and evaluate the economic feasibility of the process [27, 28].

In 2020, there research stating about simulation the spray process via Aspen Plus program. Aspen Plus software was used to simulate the dairy plant process and to evaluate the potential of using biogas as an alternative energy source. its allows the user to create process flow diagrams, specify process conditions, and simulate the behavior of the system under various scenarios. In this paper, the authors used Aspen Plus to simulate the milk drying process, both with natural gas and with biogas. The simulation results were then compared to determine the feasibility and benefits of using biogas. Aspen Plus was also used to perform an economic analysis of the plant, which included the costs of implementing biogas infrastructure and the potential savings from using biogas instead of natural gas. The economic analysis showed that although the initial investment in biogas infrastructure is higher, the long-term savings from using biogas outweigh the costs. Overall, Aspen Plus was an essential tool in the paper, enabling the authors to create a detailed simulation of the dairy plant process and to evaluate the economic and environmental impacts of using biogas [28].

In addition, there are further references to Aspen plus simulation in the literature. The authors identify the challenges associated with modeling the properties of liquid foods, such as their non-Newtonian behavior and sensitivity to temperature and concentration and propose a method for addressing these challenges. The method involves measuring the physical and chemical properties of the liquid food and using these data to develop mathematical models that can be integrated into process simulation software. Aspen Plus, a process simulation software, is utilized by the authors to simulate the concentration of milk by reverse osmosis. The simulation results are then compared to experimental data to evaluate the accuracy of the model. The paper emphasizes the importance of accurate property modeling for process simulation in the food industry, and highlights the potential of Aspen Plus as a tool for such simulations. The simulation results show that the proposed method is effective in accurately predicting the behavior of liquid foods during concentration, which can be used to optimize the design and operation of food processing equipment. Overall, the paper presents a comprehensive approach for modeling the properties of liquid foods and demonstrates the usefulness of Aspen Plus in simulating food processing operations[27].

CHAPTER III Experiments

3.1 Materials

Materials	Company
White Holy basil oil	Thai - China Flavours and Fragrances Industry Co., Ltd. (Thailand).
HI-CAP®100	Ingredion (Thailand) Co., Ltd.
Hexane (Analytical grade)	Fisher chemical (Belgium)
Deionized water	Ultrafiltration module UV-modules, ELGA PURELAB Ultra Genetic
Ethanol (Analytical grade)	Sigma-Aldrich, Singapore
Benzyl alcohol	S.M. Chemical Supplies Co., Ltd.
Linalool	Tokyo Chemical Industry, Japan
Eugenol	RCI Labscan, Thailand
Methyl eugenol	Aldrich, Singapore
Beta-caryophyllene	Tokyo Chemical Industry, Japan

3.2 Apparatus

3.2.1 High speed homogenizer

Company: IKA® Works (Thailand) Co. Ltd.

Model: S25N-25G

Application: Preparing the emulsion

Figure 11 Homogenizer model S25N-25G, IKA® Works (Thailand) Co. Ltd.

3.2.2 Spray dryer in Laboratory-scale

Company: BUCHI (Thailand) Ltd.

Model: Mini spray dryer B-290

Application: Transformed the emulsion consisted of substance and wall materials in to the powder.

Figure 12 Mini spray dryer B-290, BUCHI (Thailand) Ltd.

3.2.3 Spray dryer in Pilot-scale

Company:	GEA Niro Engineering for a better world.
Model:	MOBILE MINOR™ (NIRO)
Application:	Drying the suspensions or emulsions into representative powder samples.

Figure 13 MOBILE MINOR™ (NIRO), GEA.

3.2.4 Laser scattering particle size distribution analyzer

Company:	HORIBA, Ltd.
Model:	Partica LA-950V2
Dynamic range:	0.01 - 3000 µm
Application:	Analyze the re-dispersed particle and encapsulated powder size

Figure 14 Laser Scattering Particle Size Distribution Analyzer (HORIBA, Ltd.)

3.2.5 Gas chromatograph

Company: Agilent Technologies Co.,Ltd.

Model: Agilent 7890A

Application: Analyzed the amount of components

Figure 15 Gas chromatography (Agilent Technologies (Thailand) Co.,Ltd.)

3.2.6 Moisture analyzer

Company: Mettler-Toledo (Thailand) Ltd.

Model: HR83 Halogen

Application: Measured the humidity of encapsulated powder

Figure 16 Moisture analyzer (Mettler-Toledo (Thailand) Ltd.)

3.2.7 Scanning electron microscope

Company: JEOL Ltd.

Model: HR83 Halogen

Application: To generate high-resolution, three-dimensional surface images of specimens at a minute scale.

Figure 17 Scanning Electron Microscope

3.2.7 Viscometer

Company: Brookfield engineering Ltd.

Model: DV2T

Application: To measure the viscosity of fluids by assessing their resistance to flow, providing crucial information about the fluid's thickness or resistance to deformation.

Figure 18 Viscometer

3.3 Methodology

3.3.1 Spray drying the encapsulated powder via laboratory-scale

Spray drying process schematic of the Mini Spray Dryer B-290 is demonstrated in. The laboratory-scale nozzle type is two-fluid nozzle with the following technical feature; the evaporation capacity 1 kg/hr, the drying air flowrate is 40 kg/hr, and achieved particle size is 2 - 25 micron. The streams between hot air stream and emulsion stream are co-current stream, the emulsion will be sprayed in form of droplet through a drying chamber while being blown out by a two fluid nozzle atomizer. After that, the emulsion droplet turn into minuscule particles coming in contact with the dry air, resulting in rapid water evaporation, and finally giving out a completely dried-out powder.

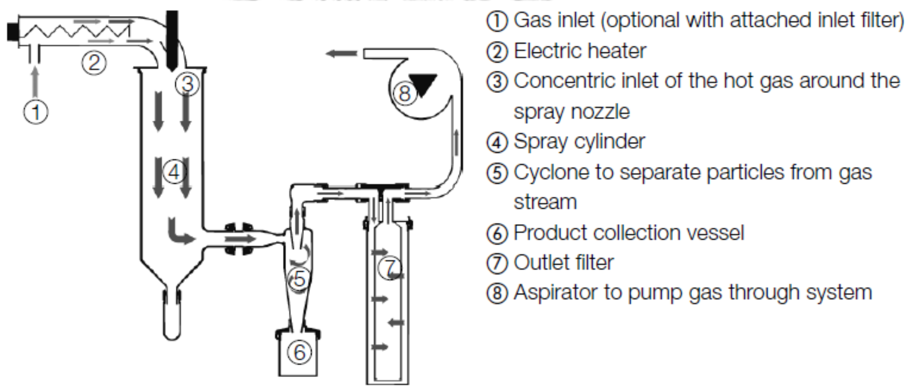


Figure 19 Direction of the feed and air stream [5].

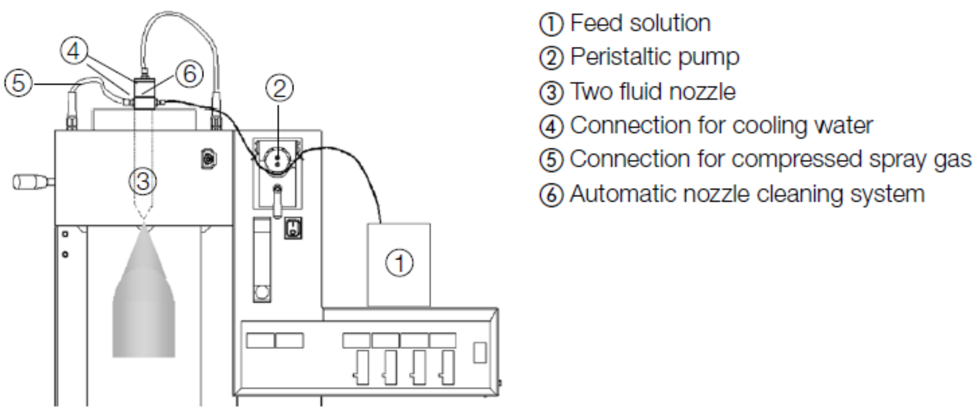


Figure 20 The operation of the two fluid nozzle [5].

3.3.1.1 Preparation of holy basil oil emulsion

Preparation of emulsion can be done via the following step, to start with dissolving the wall materials, which is HI-CAP®100 and deionized water with solid content 40% w/w to prepare the wall materials solution via stirring overnight. Secondly, add the essential oil and using a homogenizer to produce the emulsions at a rotation speed of homogenizer 8,000 rpm and time using for 3 minutes.

Table 1 Composition of spraying emulsion.

Solid content (%w/w)	Ratio of oil to wall materials (w/w)	Wall material solution (g)	Essential oil (g)
40	1:4	120	12

3.3.1.2 Preparation of the encapsulation powder

Utilizing the preparing emulsion from the previous step, which consists of holy basil essential oils and wall material; HI-CAP®100 and deionized water, as a initial substance. Then, spraying the initial substance via Mini Spray Dryer B-290 which is the laboratory-scale spray dryer model with the operating conditions of laboratory-scale spray dryer as shown in Table 2.

Table 2 Operating conditions of Mini Spray Dryer B-290 in laboratory scale

Variables		
Atomizer type	Two fluid nozzle	
Feed rate (mL/min)	11	
Total solid in feed (kg solid/ kg feed)	0.36	
Hot air inlet temperature (°C)	140	160
Hot air outlet temperature (°C)	76	87

3.3.2 Spray drying the encapsulated powder via Pilot-scale

MOBILE MINOR™ spray dryer from GEA Niro A/s company is used as a spray dryer in pilot scale. The method of work is similar to the laboratory-scale spray dryer. The streams of emulsion and air are co-current stream. The rotary atomizer and two-fluid nozzle are the nozzle types of pilot scale with the following technical feature; the evaporation capacity 0.5 - 6 kg/hr, the maximum drying air flowrate is 80 kg/hr, and achieved particle size is 2 - 80 micron. The schematic of pilot-scale spray dryer is demonstrated in Figure 21.

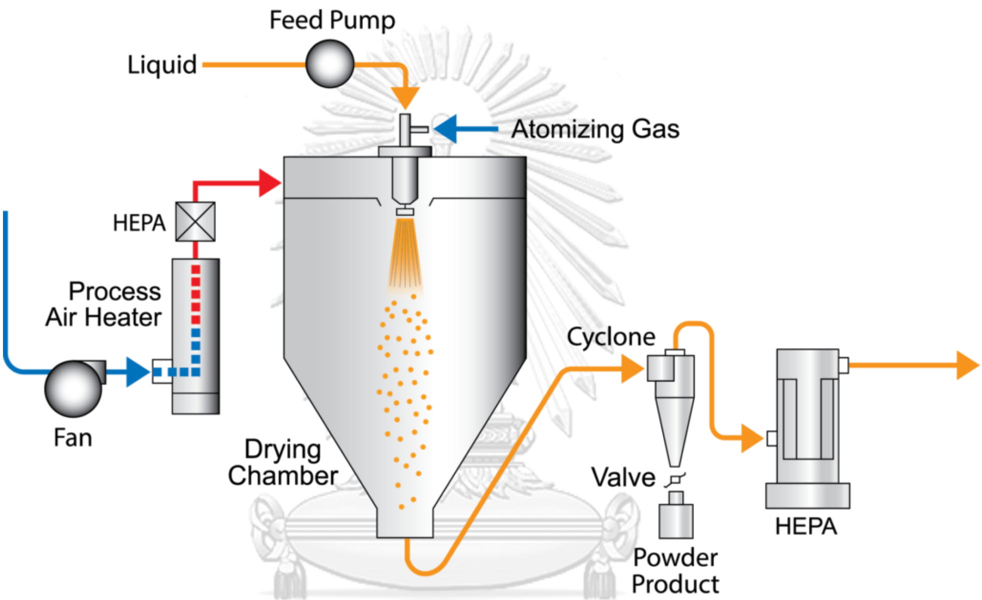


Figure 21 The schematic of MOBILE MINOR™ [6].

3.3.2.1 Scaling up the process of encapsulation of holy basil oils via spray dryer

For the scaling up process of essential oil via spray drying, this part will be implemented via examining two key components; the atomizer and the temperature of the hot air inlet. It was operated under assumption which stated that 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. And, there are 2 main part for this scaling part; the calculation mass and energy balance and simulation the process via aspen plus program.

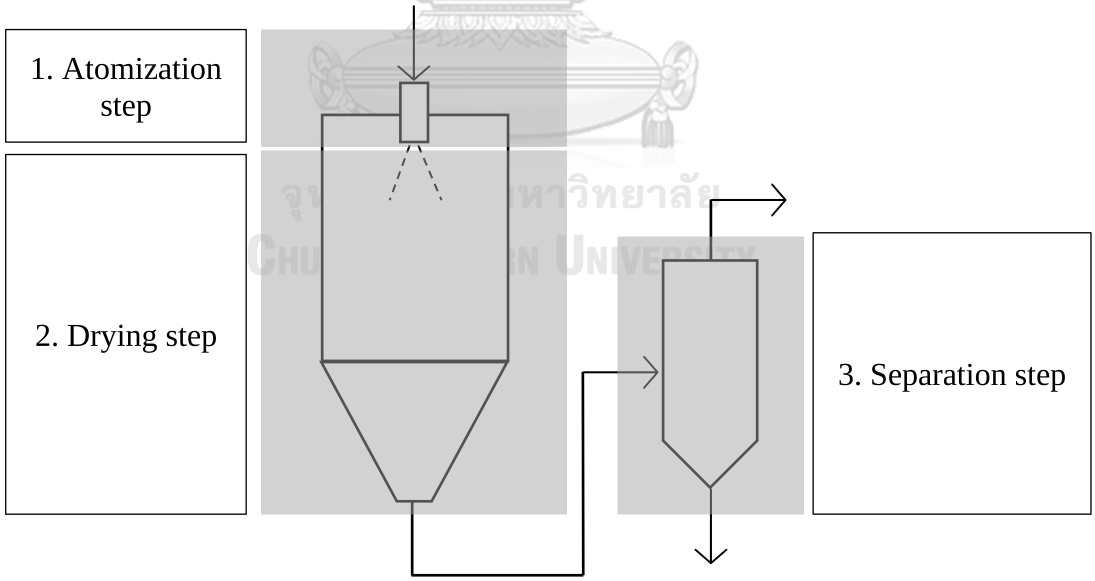
Part 1 Calculation mass and energy balance :

To determine the appropriate operating conditions in the pilot plant spray dryer. In this study, attention must be given to the temperature of the hot air inlet, to control the output moisture content as well as ensuring that it closely resembles the moisture leaving the laboratory settings. For the evaluation of the required pilot plant operating conditions, the mass and energy balance equations shall be used [7]. This calculation is based on the assumption that the ratio of the evaporated water to solids in the laboratory scale and the pilot plant scale feed lines are similar, further details on the calculation methodology shall be explained below.

Mass and energy balance analysis is crucial in understanding and optimizing the spray drying process, which is widely used in various industries to convert liquid or slurry feed into dried powder or granular products. Here's an introduction to how mass and energy balance principles apply to the spray drying process:

Spray Drying Process Overview:

Spray drying is a continuous, industrial drying process that involves the following steps:



Step 1 Atomization: A liquid or slurry feed, which contains the desired product to be dried, is pumped or atomized into small droplets using a nozzle or atomizer. This step creates a large surface area for the efficient evaporation of moisture.

Step 2 Drying: The atomized droplets are introduced into a hot air chamber, known as the drying chamber or tower. In this chamber, the liquid in the droplets quickly evaporates due to exposure to the hot air, leaving behind solid particles

Step 3 Separation: The dried particles are separated from the hot air using cyclones, bag filters, or other separation equipment. The separated product is collected for further processing or packaging.

Mass Balance in Spray Drying:
A mass balance for the spray drying process ensures that the amount of material entering the system equals the amount exiting. This balance is essential for product quality control and process efficiency. The spray dryer machine consists of 3 incoming lines: the feed entering the dryer, the hot air entering the nozzle, and the line for hot air intake to the chamber. There is only one outlet line, which is the product outgoing line where air and dry powder are mixed. The variables in each stream will be utilized in the mass and energy balance equations.

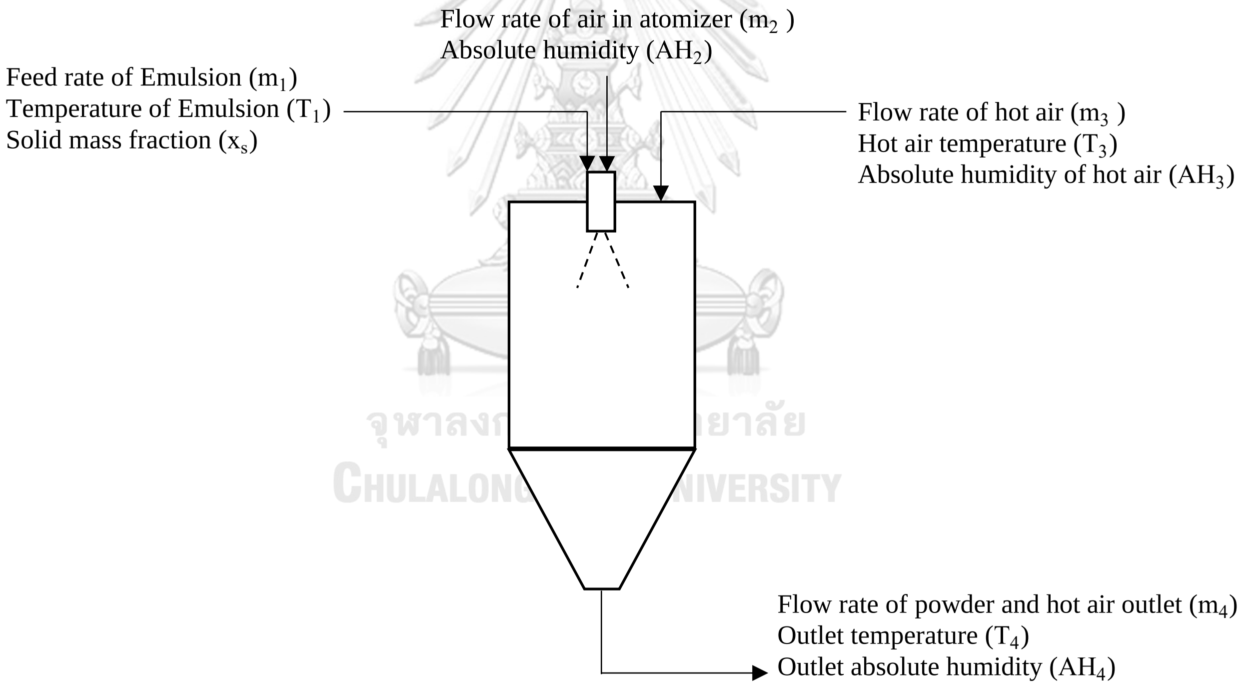


Figure 22 schematic diagram of the spray dryer

Inlet Feed: The mass of the liquid or slurry feed entering the spray drying system is a critical input parameter. It includes the mass of the drying air going to the dryer.

Outlet Products: The mass of the dried product, as well as any remaining moisture or unevaporated components, is measured as the output.

Energy Balance in Spray Drying:
Energy balance analysis in spray drying ensures that the energy input is sufficient to evaporate the liquid and achieve the desired drying effect. Key aspects of energy balance in spray drying include:

Heat Input: The energy required to heat the inlet air to the desired temperature is a crucial part of the energy balance.

Heat of Vaporization: The energy required to convert the liquid feed into vapor is a significant component of energy consumption in the process.

Sensible Heat: The energy needed to raise the temperature of the feedstock and product is also considered in the energy balance.

To scaling up the spray dry process:
By applying mass and energy balance principles can optimize the scale-up operating conditions the spray drying process for efficiency, product quality, and cost-effectiveness. This involves adjusting the inlet air temperature, feed rate, and other process parameters to achieve the desired moisture content and particle size in the final product while ensuring that the product from both scales remains similar and minimizing energy consumption.

Mass of water balance equations :

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

Energy balance equations :

$$m_1 (1- x_s) C_{pw} (T_4 - T_1) + \frac{m_2+m_3}{1+ AH_3} (AH_4 - AH_3) \Delta H_v + m_1(x_s) C_{ps}(T_4 - T_1) = \frac{m_2+m_3}{1+ AH_3} C_{p,air} (T_3 - T_4) - UA (T_4 - T_1)$$

Part 2 Simulation the spray drying process via Aspen :

The following sections will thoroughly explain the simulation via the Aspen Plus V11 program. The program settings shall begin with the selection of the spray drying chemical and the base model used in the operation of the system. For this study, SOLIDS is chosen as the base method model. After that the dryer apparatus, which is the equipment used to dry-out the designated substance via the energy brought in by the heat of the incoming hot air, is chosen. After that, the feed inlet line, the air inlet line, the feed outlet line and the air outlet line are connected as shown in Figure 23. After connecting all the lines according to Figure 23, the data acquired from the calculation from the excel file is entered. The data is divided into 2 parts: data of the streamline part and the data of the spray dryer machine. And, the heat transfer factor will be tuned to complement the output results.

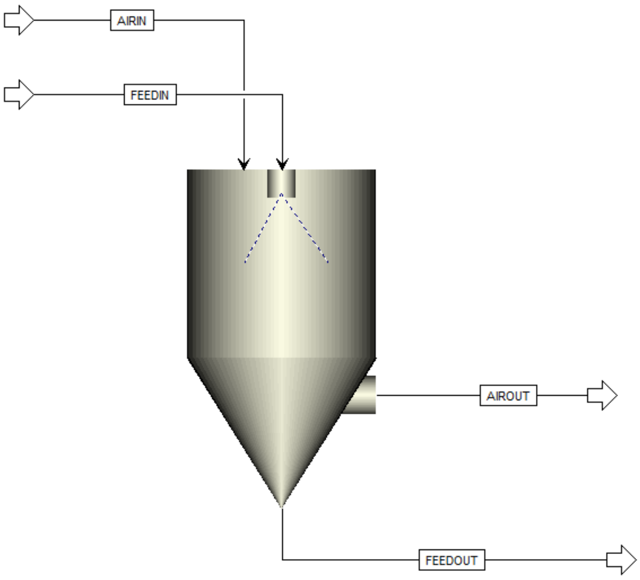


Figure 23 schematic diagram of the spray dryer in program Aspen plus.

3.3.3 Testing the characteristics of encapsulated powder

3.3.3.1 Encapsulated holy basil powder size measurement

The size of encapsulated powder can be measured via Laser Scattering Particle Size Distribution Analyzer or HORIBA model LA-950V2. The refractive index of holy basil oil was set at 1.515. The dispersion medium was set at 1.361 for ethanol which are using to measure the encapsulated powder size. The sizing machine is shown Figure 24.

Figure 24 Laser Scattering Particle Size Distribution Analyzer (HORIBA, Ltd.)

3.3.3.2 Moisture content of encapsulated holy basil powder

The humidity of holy basil oils encapsulated powder can be measured via Moisture analyzer as shown in Figure 25.

Figure 25 Moisture analyzer.

3.3.3.3 Quantitative determination of residue components in powder

The main components in the encapsulated powder; linalool, eugenol, methyl eugenol, and beta-caryophyllene will be measured via Gas Chromatography (GC) as shown in Figure 26. The Agilent GC 7890A with flame ionization detector (FID) is used with the DB-5HT column (30 m x 0.25 i.d.; film thickness 0.10 μm). The injection volume is 1 μL in a split mode (1:10). Helium was used as carrier gas at 1 mL/min flow rate. The oven temperature was set at 40 ˚C for 2 minutes, and increased by 10 ˚C/min until 250 ˚C where it was held at this temperature for 5 min.

Figure 26 Gas Chromatography (GC).

3.3.3.4 Structure of microcapsule

The structural characteristics of microcapsules both before and after the spraying process can be assessed through the utilization of a Scanning Electron Microscope, as demonstrated in Figure 27.

Figure 28 Scanning Electron Microscope

3.3.3.5 Total retention interest substance in encapsulated powder

The calculated retention of main components in encapsulated powder are divided into 3 part; the amount of main holy basil essential oils in encapsulated powder, the retention of main components in encapsulated powder, and the retention of main components at the encapsulated powder surface. The measured components were divided into three constituents: Eugenol, Methyl eugenol, and Beta-caryophyllene.

The equation for calculating the amount of main holy basil essential oil in encapsulated powder is shown in equation (4) where the unit of oil content is gram per gram.

$$\text{Oil content} = \frac{\text{The remaining oil in the powder}}{\text{Total of encapsulated powder}} \quad (4)$$

The equation for calculating the retention of main components at the encapsulated powder surface is presented in equation (5) where the unit of components in powder is gram per gram.

$$\text{Oil on surface} = \frac{\text{The components on powder surface}}{\text{Total of encapsulated powder}} \quad (5)$$

The equation for calculating the retention of the main components within the encapsulated powder is shown in equation

(6) where the unit of encapsulated is gram per gram.

Oil retention = Oil content - Oil on surface (6)

CHAPTER VI Results and Discussion

The objective of this research is to scale up the spray drying process from a small to a large scale by completing three steps in the process of scaling up spray drying. The concept of scaling up will be divided into three steps: The first part aims to maintain consistent feed properties before introducing them into the dryer across both scales. The second step involves specifying the size of the powder by studying factors affecting particle size, and the third part focuses on using the drying air flow rate to match the ratio of workers and jobs in the two scales. This chapter will present all the results obtained, including calculations, experiments, and the utilization of tools such as Mastersizer, scanning electron microscope (SEM), and gas chromatography machine (GC). In this project, two types of pilot-scale atomizers should be considered: the rotary atomizer and the two-fluid nozzle.

4.1 Emulsion preparation

This step is dedicated to emulsion preparation, during which the emulsion is introduced into the dryer. To scale up the process, the primary concern, before addressing other aspects, is to maintain the consistency of the feed properties with the laboratory scale. The emulsion ratio remains consistent with the laboratory data, maintaining a 1:4 ratio between the oil and HI-CAP 100. Wall material solution is created using deionized water with a solid content of 40% w/w, and this solution is subjected to overnight stirring. Subsequently, incorporate the essential oil into the mixture and employ a homogenizer to generate emulsions. Set the homogenizer to a rotation speed of 8,000 rpm and maintain the process for a duration of 3 minutes. The properties of the emulsion before it is sprayed are presented in Table 3.

Table 3 The properties of the feed for both scales before being sprayed into the chamber.

	Laboratory scale	Pilot scale
Size of emulsion (micrometer)	$0.45 \pm 7.62\times10^{-3}$	$0.43 \pm 3.16\times10^{-4}$
Viscosity (cP)	259	260

4.2 Study the effect of parameters on the physical properties of microcapsules

The objective of this topic is to identify the conditions that affect the size of microcapsules in order to maintain the size of microcapsules consistent with the formal scale. This study examines the factors that impact the size of microcapsules in the production of microcapsules process, with a specific emphasis on the application of a spray dryer. The factors under consideration encompass the feed flow rate, and the temperature of the drying air inlet.

4.2.1 Examine the impact of the flow rate on the particle size.

The feed flow rate is one of the factors that directly affect the properties of microcapsules. In order to achieve a similar microcapsule size between the pilot scale and the laboratory scale, the study focuses on scaling up the process and analyzing the feed flow rate. The other conditions in this process are kept constant, as indicated in the Table 4.

Table 4 the operating conditions of spray drying by adjusting the feed flow rate to achieve specific microcapsule sizes.

Experiment variables	Type of atomizer
	Rotary
Feed flow rate (kg/h)	**1.6, 1.92, 2.15, 2.37, 2.84**
Drying air inlet temperature (°C)	140
Drying air Outlet temperature (°C)	77
Drying air inlet flowrate (kg/h)	86
Solid content (%by weight)	0.36
Pressure of atomizer (bar)	6
Experiment variables	Type of atomizer
	2-fluid nozzle
Feed flow rate (kg/h)	**1.36, 1.6, 1.95**
Drying air inlet temperature (°C)	140
Drying air Outlet temperature (°C)	87
Drying air inlet flowrate (kg/h)	86
Solid content (%by weight)	0.36
Pressure of atomizer (bar)	5

Table 4 demonstrate the physical properties of the microcapsules from two types of atomizers. It was observed that as the microcapsule flow rate increases, the average diameter of the microcapsules also increases. The highest recorded value for the rotary atomizer type was 13.52 micrometer at a flow rate of 2.84 kg/h, while the 2-fluid nozzle atomizer reached 11 micrometer at a flow rate of 1.95 kg/h. These experimental results are expected to be caused by the increased flow rate while keeping the air pressure in the nozzle constant. This indicates that the air pressure in the nozzle, which generates centrifugal force on the emulsion, remains unchanged. However, when the emulsion flow rate was increased, the centrifugal force acting on the larger amount of liquid remained the same. As a result, the emulsion dispersed into larger droplets.

When dealing with the moisture content in the powder, the experiment revealed that the moisture content increased with a higher feed flow rate after drying. Using high feed line flow rates will cause a large number of emulsion droplets to enter the dryer, and since these droplets are larger in number, the amount of solvent inside them increases. Consequently, drying requires a higher amount of heat to evaporate the solvent in the emulsion droplets. One way to increase the heat output is by raising the temperature of the hot air, which, in turn, decreases the feed flow rate. Without elevating the hot air temperature, there won't be enough heat to evaporate water or ethanol inside the droplets during drying. As a result, some water remains mixed with the microcapsule powder, causing the microcapsules to become excessively moist and potentially trapped inside the dryer.

The experiment revealed that, in order to consistently maintain the microcapsule size within the specified range, a feed flow rate of 1.95 kg/h at an atomizer pressure of 5 bar is necessary for the two-fluid nozzle, while a feed flow rate of 1.6 kg/h at an atomizer pressure of 6 bar is required for the rotary atomizer. The results pertaining to the size and moisture content of microcapsules from both scales are presented in Table 5.

Table 5 The size and moisture content in microcapsules' results from both scales

Feed flow rate (kg/h)	Particle size (micrometer)	Moisture content (%water/dry solid)
	Laboratory scale	
Two fluid atomizer		
0.77	11.2± 1.03	1.80
	Pilot scale	
Rotary atomizer		
1.6	$11.3\pm 1.32\times10^{-1}$	1.67
1.92	$12.3\pm 2.83\times10^{-1}$	1.73
2.15	$12.9\pm 7.57\times10^{-2}$	1.79
2.37	$13.0\pm 2.31\times10^{-1}$	1.79
2.84	$13.5\pm 3.75\times10^{-1}$	1.8
Two fluid atomizer		
1.35	$9.5\pm 5.01\times10^{-2}$	1.68
1.6	$10.0\pm 1.59\times10^{-1}$	1.73
1.95	11.0± 1.23	1.81

4.2.2 Examine the impact of the temperature of the inlet drying air on the particle size.

Examine the effect of drying air inlet temperature on the particle size. The other variables are held constant, while the drying air temperatures under investigation are 120, 140, 160, and 180 °C, as shown in Table 6.

Table 6 The conditions of the spray drying process involving various drying inlet temperatures.

Experiment variables	Type of atomizer
	Rotary
Feed flow rate (kg/h)	1.7
Drying air inlet temperature (°C)	**120, 140, 160, 180**
Drying air Outlet temperature (°C)	77
Drying air inlet flowrate (kg/h)	86
Solid content (%by weight)	0.36
Pressure of atomizer (bar)	6
Experiment variables	**Type of atomizer**
	2-fluid nozzle
Feed flow rate (kg/h)	1.95
Drying air inlet temperature (°C)	**120, 140, 160, 180**
Drying air Outlet temperature (°C)	87
Drying air inlet flowrate (kg/h)	86
Solid content (%by weight)	0.36
Pressure of atomizer (bar)	5

The experiment revealed that the alteration of the drying air's temperature did not affect the mean diameter of the microcapsules, which remained constant at 14.9 micrometer for rotary atomizer and 11.1 micrometer for two fluid nozzle. Regarding the moisture content in the microcapsules, it was found that the highest moisture content remaining in the microcapsules was 2.73% water content/dry solid for the rotary atomizer and 2.83% water content/dry solid for the two-fluid nozzle at 120°C, while the lowest was 1.02% water content/dry solid for the rotary atomizer and 1.14% water content/dry solid for the two-fluid nozzle at 180°C. Hence, as the drying air temperature increases, the moisture content decreases. Conversely, if the drying air temperature remains too low, the microcapsules may retain an excessive amount of moisture. The outcomes pertaining to the dimensions and moisture content of microcapsules subjected to various drying air inlet temperatures, from both scales, are presented in Table 7.

Table 7 The experimental results of the spray drying process encompassing different drying air inlet temperatures.

Atomizer type	inlet temperature °C	powder size micrometer	Moisture content %water content/dry solid
Rotary	120	15.2± 1.59×10^{-1}	2.73
	140	14.3± 1.20×10^{-1}	1.73
	160	14.9± 2.83×10^{-1}	1.15
	180	15.0± 3.75×10^{-1}	1.02
2-fluid nozzle	120	10.5 ± 2.90×10^{-1}	2.83
	140	11.4± 2.75×10^{-1}	1.77
	160	11.0± 1.90×10^{-1}	1.29
	180	11.3± 3.50×10^{-1}	1.14

4.3 Study the scaling-up assumption in order to achieve the scaling-up conditions.

This topic explores methods for determining the operating conditions to scale up microcapsules from the laboratory to pilot scale. To achieve the operating conditions when scaling up the encapsulation process. The worker and jobs of the spray dry process will be studied. In this research, the worker represents the mass transfer rate, while the job correspond to the volumetric system. The focus is on the microcapsule system within the spray dryer. The mass transfer rate pertains to the volume of water evaporated from the emulsion droplets produced by the atomizer, while the volume system refers to the amount of solid contained within these emulsion droplets. Therefore, the research assumption is stated that, “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 in both a lab-setting and a scale-up setting are kept consistent, the encapsulated power of either scales will also turn out similar.” Signifying that in order to attain a product consistent with the laboratory-scale production, it is imperative to achieve precise values for two critical parameters: the atomizer's blowout rate and the evaporated water-to-solid ratio. In pursuit of this objective, it is essential to establish the correlation between droplet sizes and the blowout rate for both the laboratory and scale-up processes. Moreover, other operating conditions for a scale-up process must also be found, e.g., feed rate, drying air flow rate, hot air inlet temperature etc. Then, the heat loss parameter will be fixed and the relationship between evaporation rate and hot air temperature inlet shall be found using mass and energy equations.

The main component of holy basil oil will be studied before conducting the experiment. These oil components will be analyzed using gas chromatography, and the result is presented in Figure 29.

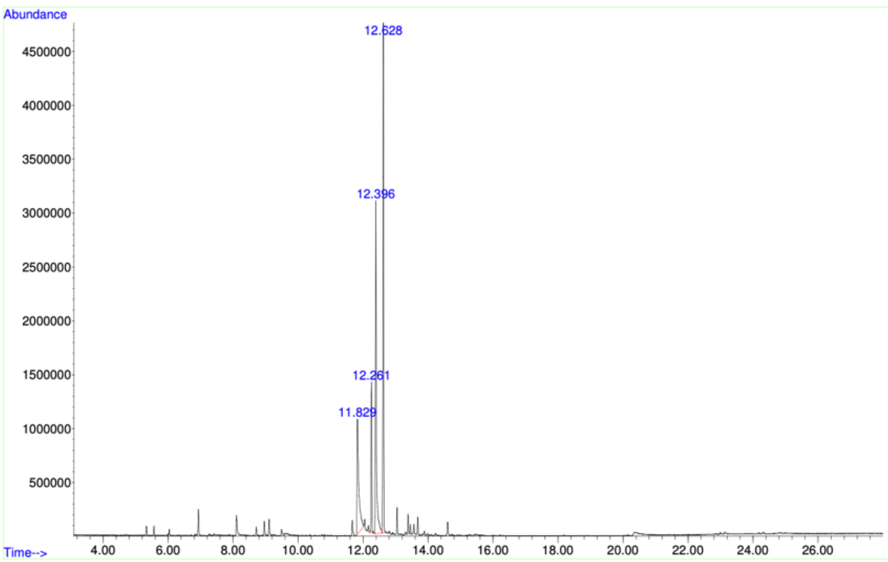


Figure 29 the graph depicting the correlation between abundance and time

Regarding the graph depicting the correlation between abundance and time, it shows four high peaks at 11.828, 12.261, 12.396, and 12.628 which is eugenol, beta-elemene, methyl eugenol and beta-caryophyllene respectively. Due to eugenol, methyl eugenol, and beta-caryophyllene being the major constituents of holy basil oil, while beta-elemene does not have a significant effect on the scent of holy basil [29]. Hence, there are three components employed in the comparative analysis are eugenol, methyl eugenol, and beta-caryophyllene.

According to the laboratory results, the optimal operating conditions for the encapsulation process of holy basil oil were ascertained. These conditions include a ratio of 1:4 between HI-CAP powder and holy basil oil, with drying air temperatures set at 140 and 160 degrees Celsius. The laboratory experiment studied four conditions of drying air inlet temperatures: 120, 140, 160, and 180 degrees Celsius, while maintaining a 1:4 ratio between HI-CAP powder and holy basil oil. Table 8 is illustrated the data experiment for lab scale.

Table 8 The experimental data obtained under varying drying air inlet temperatures in different operating conditions.

Experiment Variable	Type of Atomizer Two-fluid nozzle
Feed flowrate (kg/h)	0.77
Drying air inlet temperature (°C)	**120, 140, 160, 180**
Drying air flowrate (kg/h)	40
Solid content (%by weight)	0.36

The experimental results reveal that with respect to the percentage of component retention, as demonstrated in Figure 30. It effectively conveys that at 160 degrees Celsius of drying air inlet temperature, eugenol and methyl eugenol have the highest percentage of retention in the microcapsule, which is 89.36% and 70.95%, respectively. And at 140 degrees Celsius of drying air inlet temperature, Beta-caryophyllene have the highest percentage of retention in the microcapsule, which is 84.44%.

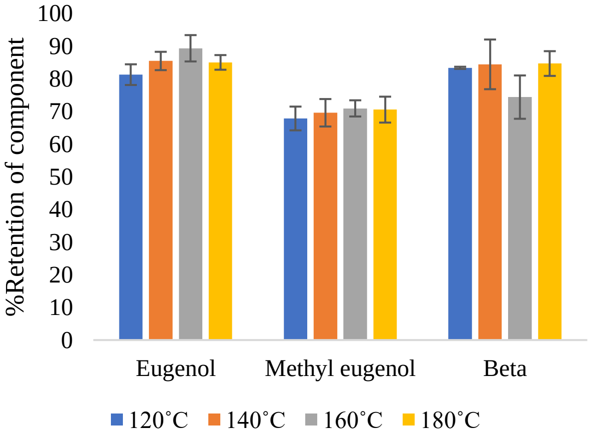


Figure 30 The graph illustrates the main components exposed on the encapsulated powder.

As a result, the selected drying air temperature conditions for scaling up section from the laboratory scale are 140 and 160 degrees Celsius.

4.3.1 Finding heat loss of the spray dryer

Heat loss constitutes a pivotal parameter within the framework of mass and energy balance, critical for ascertaining the conditions requisite for scaling up. To quantify heat loss, experiments were conducted by systematically adjusting the drying inlet temperature and gathering outlet temperature data. After obtaining the experimental data, these data were utilized to calculate the heat loss parameter using mass and energy balance equations. These measurements were utilized in the subsequent phase for the purpose of mass and energy balance calculations.

In the experimental phase, we systematically adjusted the drying temperatures to 120, 140, 160, and 180 degrees Celsius, employing two distinct atomizer types: the two-fluid nozzle and the rotary atomizer. The other conditions in this process were kept constant, as indicated in the Table 9.

Table 9 The operating conditions during experiments to find heat loss varied with changes in the drying air inlet temperature.

Experiment variables	Type of atomizer Rotary
Feed flow rate (kg/h)	1.7
Drying air inlet temperature (°C)	**120, 140, 160, 180**
Drying air inlet flowrate (kg/h)	86
Solid content (%by weight)	0.36
Pressure of atomizer (bar)	6
Experiment variables	**Type of atomizer 2-fluid nozzle**
Feed flow rate (kg/h)	1.95
Drying air inlet temperature (°C)	**120, 140, 160, 180**
Drying air inlet flowrate (kg/h)	86
Solid content (%by weight)	0.36
Pressure of atomizer (bar)	5

After acquiring the outlet temperature data, the heat loss parameter is determined as a percentage using mass and energy balance equations, as detailed in Table 10. Subsequently, these results are graphically depicted to analyze the correlation between percent heat loss and drying air inlet temperature, as showcased in Figure 31.

Table 10 The data calculated for heat loss percentages in the NIRO spray drying model.

Atomizer type	Feed rate kg/h	Inlet hor air temperature °C	Outlet hot air temperature °C	Heat loss kJ/hr	Hot air energy kJ/hr	%heat loss
Rotary	1.7	120	79	194	2872	7
	1.7	140	88	530	3609	15
	1.7	160	108	885	3643	24
	1.7	180	115	1718	4488	38
2-fluis nozzle	1.95	120	77.5	57	2912	2
	1.95	140	82.4	1100	3947	28
	1.95	160	88.5	2054	4899	42
	1.95	180	104	3058	5876	52

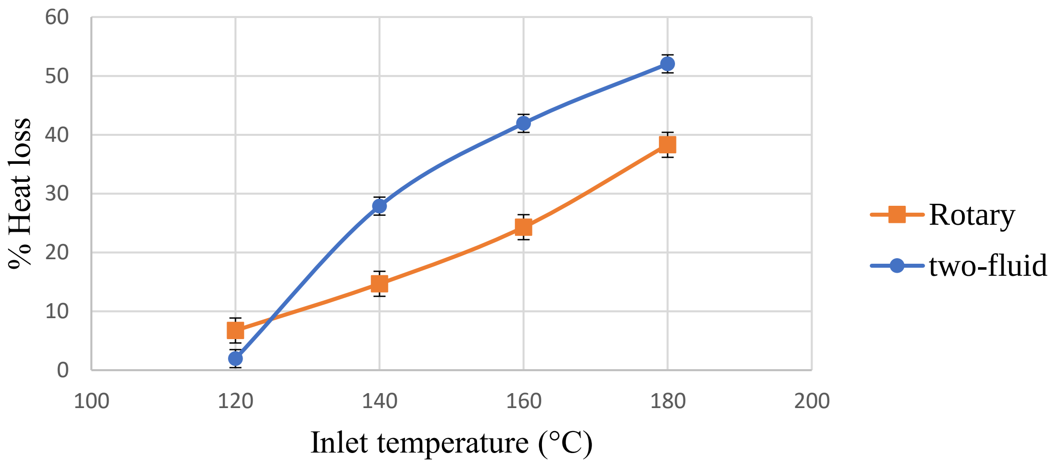


Figure 31 The graph depicting the correlation between the percentages of heat loss and drying air inlet temperature.

Based on the data from the experimental Table 10 and Figure 31 depicting the relationship between the percentage of heat loss and the type of atomizer above, it is evident that as the drying air inlet temperature increases, the tendency of heat loss also increases. The aforementioned heat loss variables were incorporated into the mass and energy equations to ascertain the optimal operational parameters for the subsequent section, which addresses the scaling-up process.

4.3.2 Study the optimal operating conditions for scaling up the spray drying process.

The objective of this topic is to determine the operating conditions for scaling up the spray drying process while maintaining a constant worker and job, as previously mentioned. Therefore, 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 was kept consistent in both laboratory and scaled-up settings, the encapsulation power were similar.

In the experiment, the conditions used for the scaling-up process are 140 and 160 degrees Celsius, with a HI-CAP to holy basil oil ratio of 1:4, as determined from the optimal operating conditions at the laboratory scale. For the pilot scale, two types of atomizers were used in this part: Rotary atomizer, and two-fluids nozzle. The operating conditions data for the feed rate are 1.6 kg/h with a rotary atomizer at a pressure of 6 bar in the atomizer, and 1.95 kg/h with the two-fluid nozzle at a pressure of 5 bar in the atomizer, in order to achieve the same particle size for each scale. The others conditions will be shown in Table 11. The objective can be achieved via adjusting the drying air flowrate parameter, which is calculated using mass and energy balance.

Table 11 The operational parameters for scaling up the NIRO spray drying model.

	Laboratory scale	Pilot scale	
Atomizer type	Two-fluid nozzle	Two-fluid nozzle	Rotary Atomizer
Drying air inlet temperature (°C)		140	
Solid contents (g solid/g feed)	0.36	0.36	0.36
Feed rate (kg/h)	0.77	1.95	1.6
Drying air flow rate (kg/h)	40	**62**	**52**
Environmental temperature (°C)	33.8	31.8	30.6
%Relative Humidity	66	67	64.4
Outlet temperature (°C)	76	77	76
%Heat loss		28	15
Evaporation rate per solid content	1.752	1.756	1.757
Drying air inlet temperature (°C)		160	
Solid contents (g solid/g feed)	0.36	0.36	0.36
Feed rate (kg/h)	0.77	1.95	1.6
Drying air flow rate (kg/h)	40	**77**	**59**
Environmental temperature (°C)	33.8	34.8	30.3
%Relative Humidity	66	58.7	81.3
Outlet temperature (°C)	87	87	88
%Heat loss		28	15
Evaporation rate per solid content	1.757	1.757	1.758

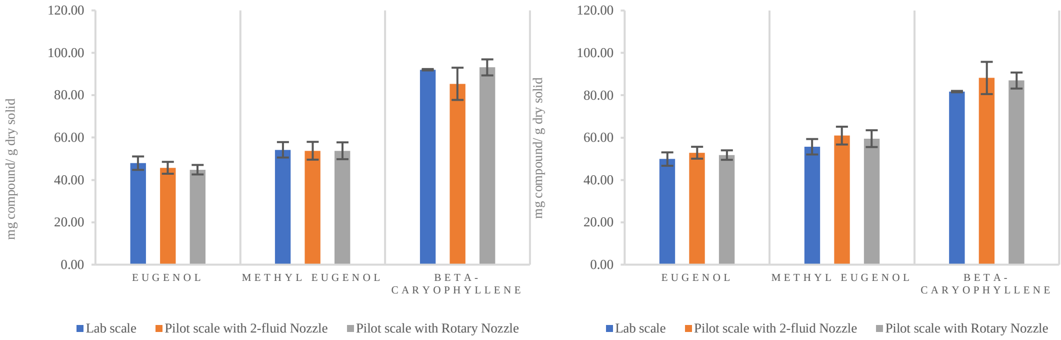
The experiment revealed that the properties of microcapsules at both the laboratory and pilot scales exhibit similarity. These properties can be categorized into two parts: chemical properties and physical properties.

The physical properties revealed that the average microcapsule size in the laboratory scale was 10.5 micrometer. Which at the pilot scale, 11.1 microns for the two-fluid nozzle and 11.3 micrometer for the rotary atomizer were found, due to variations in feed rate and atomizer pressure. The moisture content in the microcapsules is 2.49%water content per dry solid for laboratory scale, 1.99% for the two-fluid nozzle and 2.12% for the rotary atomizer in pilot scale. The bulk density is found at 0.511 g/ml for laboratory scale, 0.529 g/ml for the two-fluid nozzle and 0.530 g/ml for the rotary atomizer in pilot scale. The physical properties results are demonstrated in Table 12.

Table 12 The operational parameters of the spray dryer, at both the laboratory and pilot scales.

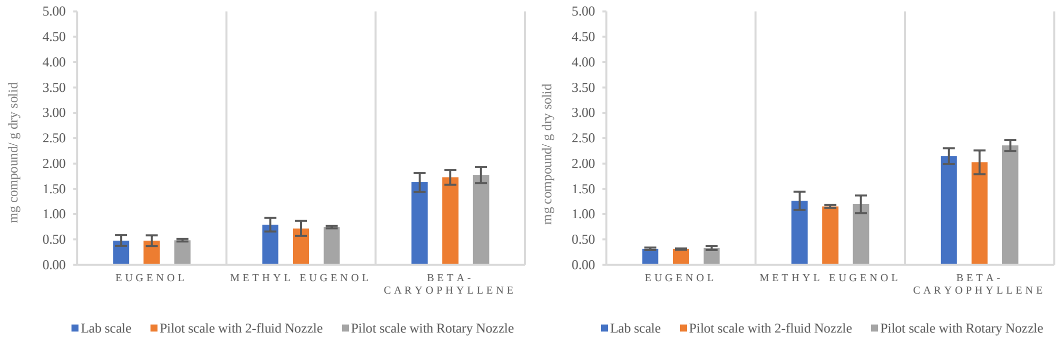
Scale	**Air flow rate**	**Drying air temperature**		**Atomizer type**	**Feed rate**	**Powder size**	**Moisture content**	**Bulk density**
	kg/h	Inlet (℃)	Outlet (℃)		kg/h	Micrometer	%water content/ dry solid	g/ml
Lab	40		76	2-fluid nozzle	0.77	10.5	2.49	0.511
	63	140	76	Rotary	1.6	11.3	2.12	0.530
Pilot	60		77	2-fluid nozzle	1.95	11.1	1.99	0.529
Lab	40		87	2-fluid nozzle	0.77	11.4	2.40	0.463
	63	160	88	Rotary	1.6	12.0	1.97	0.472
Pilot	60		88	2-fluid nozzle	1.95	11.2	2.01	0.468

For the chemical properties, The three crucial components: eugenol, methyl eugenol, and beta-caryophyllene, are used to compare between both scales. It was observed that these components remained consistent in the microcapsules across various aspects, including the total amount of main components in the microcapsules, the amount of main components on the microcapsule surface, and the amount of encapsulated substance within the microcapsules from both the two-fluid nozzle and rotary atomizer in the pilot scale as in the laboratory scale. These findings are represented in Figure 32, Figure 33,and Figure 34. The experimental data for both scales are presented in Table 11, Table 12,and Table 13.



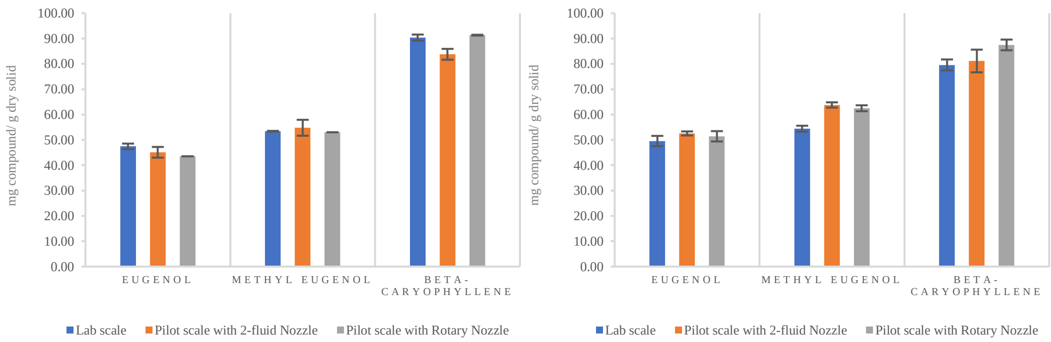
(a) (b)

Figure 32 The amount of main components in microcapsules in both scenarios: (a) components from conditions at 140 degrees Celsius and (b) components from conditions at 160 degrees Celsius.



(c) (d)

Figure 33 The amount of main components on the microcapsule surface in both scenarios: (c) components from conditions at 140 degrees Celsius and (d) components from conditions at 160 degrees Celsius.



(e) (f)

Figure 34 The amount of encapsulated substance within the microcapsule in both scenarios: (e) components from conditions at 140 degrees Celsius and (f) components from conditions at 160 degrees Celsius.

Table 13 The amount of main components in the microcapsules for two conditions: 140 and 160 degrees Celsius of drying air inlet temperature.

scale	Atomizer type	Air flow	Inlet temperature	Outlet temperature	Eugenol		Methyl eugenol		Beta-caryophyllene	
		kg/h	℃	℃	Concentration mg compound/ g dry solid	%diff	Concentration mg compound/ g dry solid	%diff	Concentration mg compound/ g dry solid	%diff
Lab	2-fluid nozzle	40	140	76	47.91±3.17		68.14±3.64		92.01±0.29	
Pilot	2-fluid nozzle	62	140	77	45.72±2.81	4.56	53.76±4.21	0.83	85.36±7.61	7.23
Pilot	Rotary	52	140	76	44.82±2.24	6.44	53.75±3.97	0.85	93.11±3.81	1.20
Lab	2-fluid nozzle	40	160	87	49.86±3.01		55.68±3.38		81.72±0.27	
Pilot	2-fluid nozzle	77	160	87	52.86±2.50	6.00	60.94±3.94	9.45	88.16±5.11	7.88
Pilot	Rotary	59	160	88	51.75±2.32	3.77	59.51±3.91	6.87	86.95±2.19	6.39

%diff = The percentage difference in composition concentration between the laboratory scale and pilot scale.

Table 14 The amount of main components on the microcapsule surface for two conditions: 140 and 160 degrees Celsius of drying air inlet temperature.

scale	Atomizer type	Air flow	Inlet temperature	Outlet temperature	Eugenol		Methyl eugenol		Beta-caryophyllene	
		kg/h	℃	℃	Concentration mg compound/ g dry solid	%diff	Concentration mg compound/ g dry solid	%diff	Concentration mg compound/ g dry solid	%diff
Lab	2-fluid nozzle	40	140	76	0.48±0.11		0.79±0.14		1.63±0.18	
Pilot	2-fluid nozzle	62	140	77	0.47±0.11	0.72	0.72±0.15	9.22	1.73±0.14	6.03
Pilot	Rotary	52	140	76	0.49±0.02	1.66	0.74±0.02	6.18	1.77±0.16	8.79
Lab	2-fluid nozzle	40	160	87	0.32±0.03		1.26±0.18		2.14±0.15	
Pilot	2-fluid nozzle	77	160	87	0.31±0.01	1.59	1.15±0.3	8.67	2.02±0.23	5.68
Pilot	Rotary	59	160	88	0.33±0.04	4.13	1.19±0.04	5.60	2.35±0.11	9.85

%diff = The percentage difference in composition concentration between the laboratory scale and pilot scale.

Table 15 The amount of encapsulated substance within the microcapsule for two conditions: 140 and 160 degrees Celsius of drying air inlet temperature.

scale	Atomizer type	Air flow	Inlet temperature	Outlet temperature	Eugenol		Methyl eugenol		Beta-caryophyllene	
		kg/h	℃	℃	Concentration mg compound/ g dry solid	%diff	Concentration mg compound/ g dry solid	%diff	Concentration mg compound/ g dry solid	%diff
Lab	2-fluid nozzle	40	140	76	47.43±1.11		53.42±0.13		90.38±1.18	
Pilot	2-fluid nozzle	62	140	77	45.11±2.11	4.89	53.04±3.14	2.55	83.78±2.14	7.30
Pilot	Rotary	52	140	76	43.52±0.02	8.24	53.01±0.02	0.77	91.34±0.16	1.06
Lab	2-fluid nozzle	40	160	87	49.55±2.03		54.41±1.18		79.58±2.15	
Pilot	2-fluid nozzle	77	160	87	52.54±0.80	6.05	59.78±1.03	9.87	81.14±3.47	1.96
Pilot	Rotary	59	160	88	51.42±2.03	3.77	58.32±1.17	7.18	84.57±2.11	6.27

%diff = The percentage difference in composition concentration between the laboratory scale and pilot scale.

4.4 Simulation the model of spray dry process via Aspen plus V11

The objective of this study is to simulate the spray drying process via Aspen plus program with the aim of predicting the operating conditions required for scaling up the process from laboratory scale to pilot scale. Another objective is to establish operating conditions for different weather conditions in Thailand, including both summer and the rainy season.

In order to simulate the spray drying process and facilitate its scaling-up, the employment of the Aspen Plus program proves to be an advantageous strategy. This approach offers enhanced convenience for data input and result analysis, substantially reducing the time invested compared to conventional Excel-based methods. Additionally, it expedites the determination of the airflow rate under varying environmental conditions, encompassing temperature and relative humidity. This method, celebrated for its efficiency, stands as one of the preeminent techniques available for the purpose of ascertaining appropriate operating conditions the scaling-up of the spray drying process.

4.4.1 Validate model of the spray dry process

To validate the simulation model in the Aspen Plus program, the drying air flow rates at 140°C and 160°C in the spray drying process with a two-fluid atomizer will be considered. After creating the process diagram in Aspen Plus V11, we complete this model using the 'design spec' function. The Aspen plus program input data is demonstrated in Table 16 for 140°C and Table 17 for 160 °C.

Table 16 The input parameters for the Aspen Plus program at a drying air inlet temperature of 140 degrees Celsius.

Feed stream	
Total flow rate (kg/h)	1.95
Solid mass fraction (kg solid/kg feed)	0.36
Feed temperature (°C)	33.2
Feed pressure (bar)	1.013
Drying air inlet stream	
Air flow rate (kg/h)	67.1
Water flow rate (kg/h)	1.28
Drying air stream temperature (°C)	140
Drying air stream pressure (bar)	1.013
Air flow in atomizer	
Air flow rate (kg/h)	5.4
Air stream temperature (°C)	140
Air stream pressure (bar)	5
Dryer	
Dryer type	Spray dryer
Spray tower height (m)	1.5
Spray tower diameter (m)	0.8
Ambient temperature (°C)	33.2
Atomizer type	Two fluid nozzle
Nozzle orifice diameter (mm)	5

Table 17 The input parameters for the Aspen Plus program at a drying air inlet temperature of 160 degrees Celsius.

Feed stream	
Total flow rate (kg/h)	1.95
Solid mass fraction (kg solid/kg feed)	0.36
Feed temperature (℃)	34.8
Feed pressure (bar)	1.013
Drying air inlet stream	
Air flow rate (kg/h)	67.1
Water flow rate (kg/h)	1.43
Drying air stream temperature (℃)	140
Drying air stream pressure (bar)	1.013
Air flow in atomizer	
Air flow rate (kg/h)	5.4
Air stream temperature (℃)	140
Air stream pressure (bar)	5
Dryer	
Dryer type	Spray dryer
Spray tower height (m)	1.5
Spray tower diameter (m)	0.8
Ambient temperature (℃)	34.8
Atomizer type	Two fluid nozzle
Nozzle orifice diameter (mm)	5

Design spec or design specification is used to define the desired behavior or properties of a chemical process and can be crucial in optimizing and fine-tuning the design of a process. The objective of this process is to achieve the target outlet temperature of actual spray dry process. To control and optimize this, we manipulate the heat transfer coefficient, which serves as a representative parameter for heat loss. Based on the utilization of the design specifications function in the Aspen plus program, the heat transfer coefficient is 1 W/m^2. K for a drying air inlet temperature of 140 ℃ and 1.1 W/m^2. K for a drying air inlet temperature of 160 ℃.

4.4.2 Developing the scaling up conditions model using Aspen Plus V11

In this stage, we use the validated simulation model that selected the heat transfer coefficient. The design specifications were reapplied to determine the optimal drying air flow rate for the scale-up of the spray drying process. The manipulated variable is the drying air flow rate, and the target parameter to be achieved is the outlet temperature. The Aspen plus program input data is demonstrated in Table 18 for 140℃ and Table 19 for 160 ℃.

Table 18 The input parameter for scaling up in the Aspen Plus program at 140 degrees Celsius of drying air inlet temperature.

Feed stream	
Total flow rate (kg/h)	1.95
Solid mass fraction (kg solid/kg feed)	0.36
Feed temperature (°C)	31.8
Feed pressure (bar)	1.013
Drying air inlet	
Water flow rate (kg/h)	1.19
Drying air stream temperature (°C)	140
Drying air stream pressure (bar)	1.013
Air flow in atomizer	
Air flow rate (kg/h)	5.4
Air stream temperature (°C)	140
Air stream pressure (bar)	5
Dryer	
Dryer type	Spray dryer
Spray tower height (m)	1.5
Spray tower diameter (m)	0.8
Ambient temperature (°C)	31.8
Heat transfer coefficient (W/m^2. K)	1
Atomizer type	Two fluid nozzle
Nozzle orifice diameter (mm)	5

Table 19 The input parameter for scaling up in the Aspen Plus program at 160 degrees Celsius of drying air inlet temperature.

Feed stream	
Total flow rate (kg/h)	1.95
Solid mass fraction (kg solid/kg feed)	0.36
Feed temperature (°C)	34.8
Feed pressure (bar)	1.013
Drying air inlet	
Water flow rate (kg/h)	1.19
Drying air stream temperature (°C)	160
Drying air stream pressure (bar)	1.013
Air flow in atomizer	
Air flow rate (kg/h)	5.4
Air stream temperature (°C)	160
Air stream pressure (bar)	5
Dryer	
Dryer type	Spray dryer
Spray tower height (m)	1.5
Spray tower diameter (m)	0.8
Ambient temperature (°C)	34.8
Heat transfer coefficient (W/m^2. K)	1.1
Atomizer type	Two fluid nozzle
Nozzle orifice diameter (mm)	5

The drying air flow rate from the design specification was compared with the calculated data from excel. The parameter used for comparison between the two sets of results is the input data of the drying air flow rate and the output data, which includes the outlet temperature and moisture content. The result is demonstrated in Table 20. It was observed that the drying air flow rate from the Aspen Plus program and the calculated data from excel, as are the outlet parameters, which include the drying air flow rate and outlet temperature

Table 20 The operating conditions are obtained from the Aspen Plus program at drying air inlet temperatures of 140 and 160 degrees Celsius.

Drying air inlet temperature (°C)	140°C		160°C	
	Aspen Results	Excel Results	Aspen Results	Excel Results
Feed flow rate (kg/h)	1.95		1.95	
Solid content (kg solid/kg feed)	0.36		0.36	
Ambient Temperature (°C)	31.8		34.8	
%Relative humidity	67		58.7	
Drying air inlet temperature (°C)	140		160	
Drying air flow rate (kg/h)	62	63	77	77
Drying air outlet temperature (°C)	76.82	76	87.94	87
Moisture content (kg water/kg dry solid)	2.09	2.00	2.50	2.00
Evaporation rate per solid content	1.75	1.75	1.75	1.75

After concluding the comparison between the calculated drying air flow rate from Excel and the real spray drying process, these conditions were employed to compare output parameters. This comparison was specifically analyze the drying air outlet temperature, moisture content, and evaporation rate per solid content, aiming to validate the assumptions posited in this project. The result is demonstrated in Table *21*.

Table 21 The comparison data between the Aspen Plus results and the actual spray drying process.

Drying air inlet temperature (°C)	140°C		160°C	
	Aspen Results	Actual results	Actual results	Actual results
Feed flow rate (kg/h)	1.95		1.95	
Solid content (kg solid/kg feed)	0.36		0.36	
Ambient Temperature (°C)	31.8		34.8	
%Relative humidity	67		58.7	
Drying air inlet temperature (°C)	140		160	
Drying air flow rate (kg/h)	62	62	77	77
Drying air outlet temperature (°C)	76.82	76.00	87.94	88.00
Moisture content (kg water/kg dry solid)	2.09	1.99	2.50	2.01
Evaporation rate per solid content	1.75	1.75	1.75	1.75

4.4.3 Scaling up the operating conditions of the spray drying process with respect to the seasons in Thailand.

Upon achieving a validated model and successfully completing the scale-up process, the concluding section of this study seeks to utilize the model for assessing the optimal conditions when scaling up the process in various weather conditions in Thailand, including both summer and the rainy season. The environmental temperature and relative humidity percentage were modified to simulate various seasons within the spray drying process model. The corresponding input data is provided in the Table 22.

Table 22 The input data from Aspen Plus program for scaling up the spray drying process with seasonal scenarios in Thailand.

	Summer Season		Rainy Season	
Feed flow rate (kg/h)	1.95	1.95	1.95	1.95
Ambient Temperature	34.4	33.4	30.3	31.2
%Relative humidity	58.7	58.6	81.3	73.8
Drying air inlet temperature (°C)	140	140	140	140

Utilizing the data presented above, it was input into the Aspen Plus program. The design specification function was employed to ascertain the optimal drying air flow rate necessary to attain the desired outlet temperature for the scale-up of the spray drying process. The drying air flow rate obtained from the Aspen Plus program were compared with two key parameters from the actual process data: the drying air outlet temperature and moisture content as shown in Table 23 and Table 24.

Table 23 The obtained data results from both the Aspen Plus program and the practical spray drying process during the summer season.

	Summer Season			
Drying air inlet temperature (℃)	140			
Feed flow rate (kg/h)	1.95		1.95	
Ambient temperature (℃)	34.4		33.4	
%Relative humidity	58.7		58.6	
	Aspen	Actual	Aspen	Actual
Drying air flow rate (kg/h)	59.9	60	59.9	60
Drying air outlet temperature (℃)	76.5	77.7	76.5	76.9
%Moisture content (kg water/kg dry solid)	2.00	2.18	2.10	1.82
Evaporation per solid content	1.75	1.75	1.76	1.76

*Aspen = Aspen plus V11 program data results.
*Actual = Actual spray dry process data results.

Table 24 The obtained data results from both the Aspen Plus program and the practical spray drying process during the rainy season.

	Rainy season			
Drying air inlet temperature (°C)	140			
Feed flow rate (kg/h)	1.95		1.95	
Ambient temperature (°C)	30.3		31.2	
%Relative humidity	81.3		73.8	
	Aspen	Actual	Aspen	Actual
Drying air flow rate (kg/h)	60.6	61	62	62
Drying air outlet temperature (°C)	75.9	77	76.4	76.8
%Moisture content (kg water/kg dry solid)	2.30	2.42	2.10	1.80
Evaporation per solid content	1.75	1.75	1.75	1.76

*Aspen = Aspen plus V11 program data results.
*Actual = Actual spray dry process data results.

The experimental results indicated that by using the drying air flow rate obtained from the Aspen Plus program for the actual spray drying process consistently yielded similar outcomes in terms of the drying air outlet temperature and microcapsule moisture content, despite variations in environmental temperature and relative humidity.

CHAPTER V Conclusions

This research focused on the scale-up process for the spray drying of holy basil oil. The research comprised three key stages: to maintain consistent feed properties before introducing them into the dryer, an examination of parameters influencing microcapsule size, and the scaling-up of the spray drying process. After that, simulation of the spray drying process using the Aspen Plus program is considered. The results of each stage are detailed below.

5.1 Conclusions

5.1.1 Emulsion preparation

Cognizant of the need for emulsion preparation, the emulsion ratio will adhere to the same ratio of deionized water, powder, and oils as stipulated in the laboratory data. The mixture of water and powder will be stirred overnight and homogenized with oils at a speed of 8,000 rpm for a duration of 3 minutes until it transforms into an emulsion. Following the measurement of emulsion properties, the results revealed that the emulsion's characteristics prior to the drying process within the chamber closely resemble those observed in the laboratory-scale setting.

5.1.2 An examination of parameters influencing microcapsule size

In this section, the aim is to determine the size of microcapsules by observing two parameters that affect their size: feed flow rate and drying air inlet temperature.

5.1.2.1 The effect of microcapsule size on feed flow rate parameters

In experiments involving the feed flow rate parameter, it was observed that an increase in the feed flow rate, while maintaining the same atomizer pressure, resulted in an increase in the size of microcapsules. As the atomizer pressure, which determines the level of centrifugal force acting on the emulsion, remains constant, an increase in the feed flow rate has a direct impact on the size of the emulsion droplets. At the laboratory scale, the microcapsules are approximately 10-12 microns in size. In the pilot scale, a feed flow rate of 1.95 kg/h with an atomizer pressure of 5 bar using a two-fluid nozzle results in microcapsules with a size of 11 micrometer, while a feed flow rate of 1.6 kg/h with an atomizer pressure of 6 bar using a rotary atomizer yields microcapsules measuring 11.84 micrometer. Moreover, a higher feed flow rate corresponds to an increased moisture content within the microcapsules.

5.1.2.2 The effect of microcapsule size on drying air inlet temperature parameters

Regarding the drying air inlet temperature parameter, it was observed that variations in drying air inlet temperature did not have a significant impact on the size of the microcapsules. The average microcapsule diameter is 12.6 micrometer for two fluid nozzle and 10.2 micrometer for rotary atomizer. The predominant parameter influenced by variations in the drying air inlet temperature is moisture content. It is evident that a decrease in the drying air inlet temperature results in an increase in moisture content.

5.1.3 The scaling-up of the spray drying process

In the scale-up section, the process is predicated on the assumption that maintaining consistent particle size from the atomizer and a constant ratio between evaporated water and solid substance in the drying chamber, both in the laboratory and scaled-up settings, will yield similar encapsulation results. The experimental results corroborated this assumption by demonstrating that the properties of the microcapsules were akin to those of the original scale.

5.1.4 To conduct a simulation of the spray drying process using the Aspen Plus program.

To simulate the spray drying process, this phase involves determining the appropriate heat transfer coefficient to match the outlet temperature observed in the actual process. Through experimentation, it was determined that the heat transfer coefficient is 1 W/m^2. K for a drying air inlet temperature of 140 °C and 1.1 W/m^2. K for a drying air inlet temperature of 160 °C. The validated model in Aspen Plus is capable of predicting the operating conditions for a different scale through the utilization of the design specification function within the Aspen Plus model.

In practical industrial settings, to scale up the spray drying process, inlet and outlet temperatures are adjusted to be similar between the two scales by setting the same inlet temperature and fine-tuning the drying air flow rate until the outlet temperature is comparable. In this thesis, determining the drying air flow rate is achieved through the application of the worker and job concept, along with utilizing mass and energy balance to scale up the spray drying process.

5.2 Recommendations

5.2.1 Determining other properties of the microcapsule

In the course of this thesis, the types of properties measured may not be sufficient. Therefore, the writer recommends assessing additional properties, such as the release of microcapsules in solvent and the stability of microcapsules over various time intervals, etc., to verify other aspects related to the scale-up of the spray drying process.

5.2.2 The limitations of the Aspen Plus program for scaling up the spray drying process.

Although the Aspen Plus program has many benefits, there are limitations concerning the size of microcapsules. The program cannot precisely detect the microcapsule size. Therefore, the use of a CFD program may be necessary to verify the size of microcapsules in the spray drying chamber.

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Appendix

Appendix A Drying air flow rate calculation sections in spray dry process

The operating conditions of spray dry process will be calculated though mass and energy balance. This section will further explain the process of calculation, which utilizes Microsoft Excel to apply and integrate the mass and energy balance equations to the spray dryer. The relevant variables used in the calculations are shown in the schematic diagram of the spray dryer in Figure 35. The spray dryer machine consists of 3 incoming lines: the feed entering the dryer, the hot air entering the nozzle, and the line for hot air intake to the chamber. There is only one outlet line, which is the product outgoing line where air and dry powder are mixed. The variables in each stream will be utilized in the mass and energy balance equations, with the assumption that the evaporation rate of water to solid from the laboratory scale and pilot scale is constant.

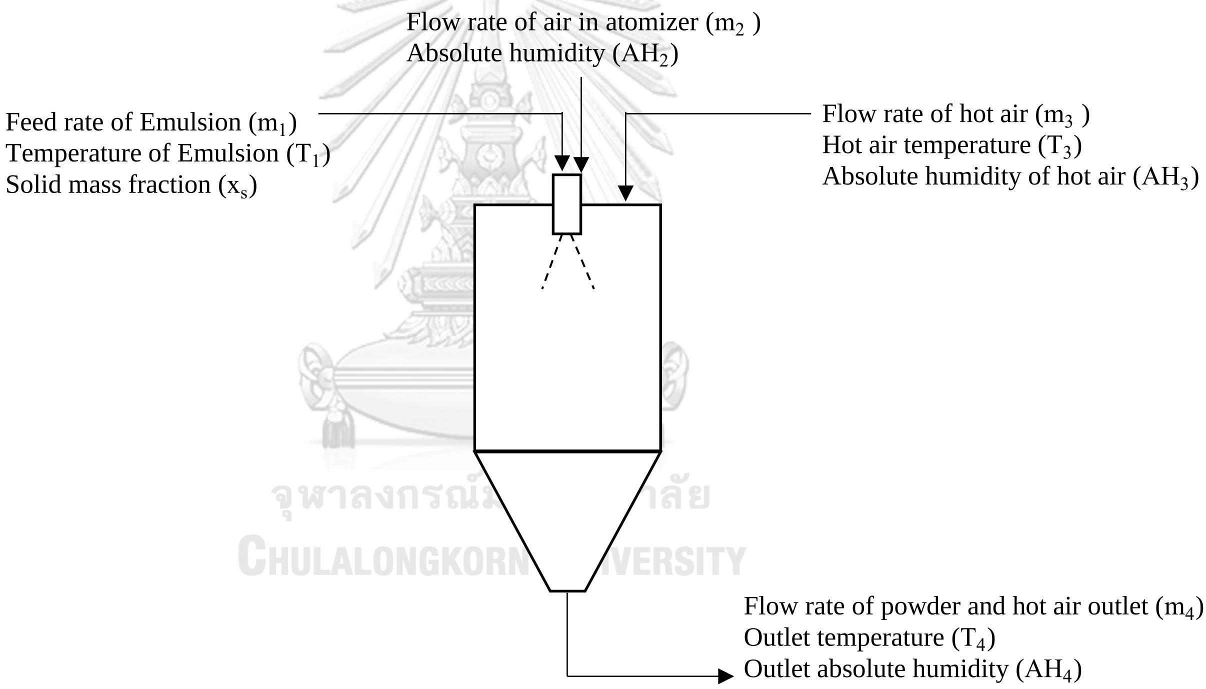


Figure 35 A schematic diagram of the spray drying process.

There are three main equations are used to find the drying air flowrate in this part.

Mass of water balance equations :

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

Energy balance equations :

$$m_1\,(1-x_s)\,C_{pw}\,(T_4 - T_1) + \frac{m_2+m_3}{1+AH_3}\,(AH_4 - AH_3)\,\Delta H_v + m_1(x_s)\,C_{ps}(T_4 - T_1)$$
$$= \frac{m_2+m_3}{1+AH_3}\,C_{p,air}\,(T_3 - T_4) - UA\,(T_4 - T_1)$$

Evaporation rate equation :

$$EVR = m_1\,(1-x_s) - m_1\,x_s\,\left(\frac{RW}{1-RW}\right)$$

; EVR is Evaporation Rate

Using the residual water in the microcapsule from the laboratory scale into the equations. In this section, the operating conditions which is used is the conditions on 140 degrees Celsius conditions. The data conditions is demonstrated in Table 25.

Table 25 The data from laboratory scale for the example calculation section.

Variables of laboratory scale	
Feed rate, m_1 (kg/h)	0.77
Total solid in feed, x_s (kg solids/kg feed)	0.36
Moisture content in microcapsule, RW (kg water/kg wet powder)	0.0225

Therefore, the evaporation rate of laboratory scale is :

$$EVR = m_1\,(1-x_s) - m_1\,x_s\,\left(\frac{RW}{1-RW}\right)$$

$$EVR = 0.77\,(1 - 0.36) - 0.77\,(0.36)\,\left(\frac{0.0225}{1-0.0225}\right)$$

$$EVR = 0.486 \text{ kg/h}$$

From mass balance and evaporation rate equations :

$$\frac{AH_4}{1-AH_3}\,m_3 + \frac{AH_4}{1-AH_2}\,m_2 = \frac{AH_3}{1-AH_3}\,m_3 + \frac{AH_2}{1-AH_2}\,m_2 + m_1\,(1-x_s) - m_1\,x_s\,\left(\frac{RW}{1-RW}\right)$$

$$EVR = m_1\,(1-x_s) - m_1\,x_s\,\left(\frac{RW}{1-RW}\right)$$

So, $\frac{AH_4}{1-AH_3}\,m_3 + \frac{AH_4}{1-AH_2}\,m_2 = \frac{AH_3}{1-AH_3}\,m_3 + \frac{AH_2}{1-AH_2}\,m_2 + EVR$

The data that will be used in the calculation part for pilot scale is demonstrated in Table 26. The evaporation rate from laboratory scale will be used in the pilot scale in the calculation part.

Table 26 The data from pilot scale for the example calculation section.

1.	Variables of laboratory scale		
2.	Environmental temperature, T_1 (°C)	3.	30.6
4.	%Relative humidity, %RH	5.	76
6.	Feed rate, m_1 (kg/h)	7.	1.95
8.	Total solid in feed, x_s (kg solids/kg feed)	9.	0.36
10.	Atomizer air flowrate, m_2 (kg/h)	11.	5.4
12.	Moisture content in microcapsule, RW (kg water/kg wet powder)	13.	0.0225
14.	Atomizer absolute humidity, AH_2	16.	0.01869
15.	(kg water/kg dry solids)		
17.	Outlet temperature, T_4 (°C)	18.	76
19.	Specific heat capacity of water, C_{pw} (kJ/kg.K)	20.	4.186
21.	Specific heat capacity of air, C_a (kJ/kg.K)	22.	1.02
23.	Specific heat capacity of solid, C_{ps} (kJ/kg.K)	24.	1.56
25.	Atmospheric pressure, P_{atm} (kPa)	26.	101.3
27.	Molecular weight of water, MW_w (g/mol)	28.	18
29.	Molecular weight of air, MW_{air} (g/mol)	30.	29
31.	%heat loss	32.	28

To determine absolute humidity of drying air stream, AH_3 (kg water/kg dry solids) :

From Antoine equation :

$$\ln(P_s) = 16.2886 - \left(\frac{3816.44}{227.02+T}\right) \quad \text{while T is environmental temperature, } T_1$$

$$P_s = 4.37 \text{ kPa}$$

From

$$\%RH = (P / P_s) \times 100$$

$$76 = (P / 4.37) \times 100$$

$$P = 2.93 \text{ kPa}$$

So,

$$AH_3 = \frac{MW_W P}{MW_{air}(P_{atm} - P)}$$

$$= 0.01847 \text{ kg water /kg dry solid}$$

To determine the drying air flow rate, m_3 (kg/h)

From energy balance equation with percent of heat loss :

$$\frac{100 - \%\text{Heat loss}}{100} \times m_3 C_{p,air} (T_3 - T_4) = m_1 (1 - x_s) C_{pw} (T_4 - T_1) + m_3 (AH_4 - AH_3) \Delta H_v$$

$$+ m_1(x_s)\, C_{ps}(T_4 - T_1)$$

While $m_3\, C_{p,air}\, (T_3 - T_4)$ = The heat from hot air inlet stream

$m_1\, (1- x_s)\, C_{pw}\, (T_4 - T_1)$ = The heat received by water

$m_3\, (AH_4 - AH_3)\, \Delta H_v$ = The heat required for evaporation in the spray drying process

$m_1(x_s)\, C_{ps}(T_4 - T_1)$ = The heat received by an emulsion droplet

Determine absolute humidity, AH_4 (kg water/kg dry solids) by reformulating the mass balance equations for water:

$$\frac{AH_4}{1-AH_3} m_3 + \frac{AH_4}{1-AH_2} m_2 = \frac{AH_3}{1-AH_3} m_3 + \frac{AH_2}{1-AH_2} m_2 + EVR$$

$$AH_4 = \frac{\frac{AH_3}{1-AH_3} m_3 + \frac{AH_2}{1-AH_2} m_2 + EVR}{\frac{1}{1-AH_3} m_3 + \frac{1}{1-AH_2} m_2}$$

so, the final equation is going to be

$$\frac{100-\%\text{Heat loss}}{100} \times m_3\, C_{p,air}\, (T_3 - T_4) = m_1\, (1- x_s)\, C_{pw}\, (T_4 - T_1) + m_3 \left(\frac{\frac{AH_3}{1-AH_3} m_3 + \frac{AH_2}{1-AH_2} m_2 + EVR}{\frac{1}{1-AH_3} m_3 + \frac{1}{1-AH_2} m_2} - AH_3 \right) \Delta H_v + m_1(x_s)\, C_{ps}(T_4 - T_1)$$

Insert the parameters from Table 1 into the concluding equation above and employ an Excel program to resolve it.

So, $m_3 = 62$ kg/h

The hot air flow rate under these specified conditions is 62 kg/h.

Appendix B The procedure involves determining the drying air flow rate through the utilization of the Aspen Plus program.

Appendix B provides a comprehensive overview of the simulation steps involved in the spray drying process of 140 ℃ drying air temperature using the Aspen Plus program. Additionally, it elucidates the procedures for scaling up the spray drying operation through the application of the design specification function, as detailed in section 4.4. The primary phase encompasses the creation of the dryer model, achieved by selecting the relevant tool from the model palette and establishing connections for the input and output streams of the dryer as demonstrated in Figure 36.

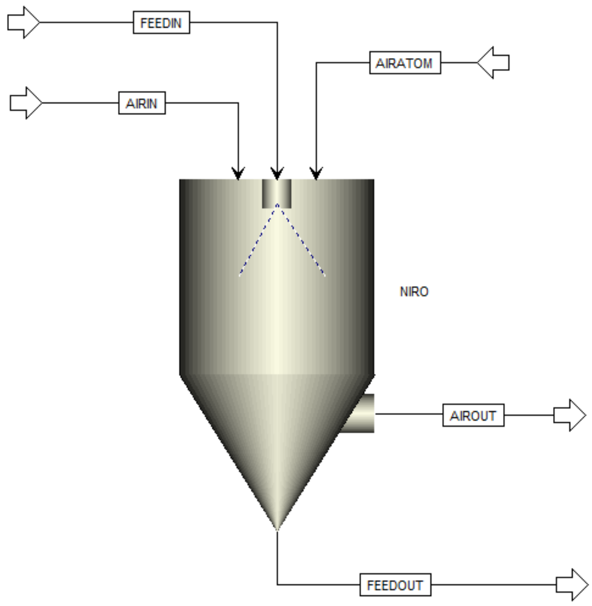


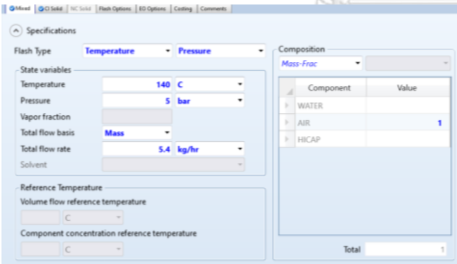
Figure 36 The spray dryer model on the main flowsheet in Aspen Plus program.

Subsequently, pertinent data is input into the program, organized into two distinct sections: the dryer section and the stream input data section. The detailed input information is illustrated in Figure 1.

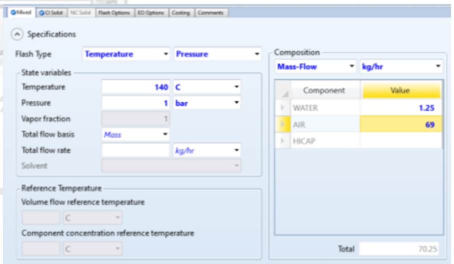
Table 27 The input data for the dryer in the Aspen Plus program.

Feed stream	
Total flow rate (kg/h)	1.95
Solid mass fraction (kg solid/kg feed)	0.36
Feed temperature (°C)	31.8
Feed pressure (bar)	1.013
Drying air inlet	
Water flow rate (kg/h)	1.19
Drying air stream temperature (°C)	140
Drying air stream pressure (bar)	1.013
Air flow in atomizer	
Air flow rate (kg/h)	5.4
Air stream temperature (°C)	140
Air stream pressure (bar)	5
Dryer	
Dryer type	Spray dryer
Spray tower height (m)	1.5
Spray tower diameter (m)	0.8
Ambient temperature (°C)	31.8
Heat transfer coefficient (W/m^2. K)	8.22×10^{-6}
Atomizer type	Two fluid nozzle
Nozzle orifice diameter (mm)	5

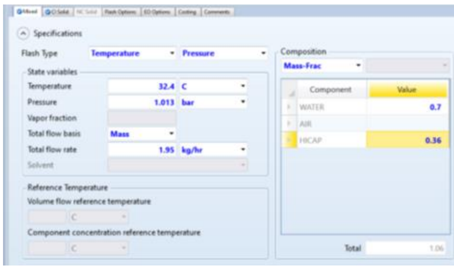
The stream information is categorized into three distinct stream lines: drying air flow rate in the atomizer, drying air flow rate into the dryer, and the feed flow rate. The input information for these three streams is delineated in Figure 37, where (a) corresponds to the drying air flow rate in the atomizer, (b)denotes the drying air flow rate into the dryer, and (c) represents the feed flow rate.



(a)



(b)



(c)

Figure 37 The input information for the three streams in the Aspen Plus program: (a) corresponds to the drying air flow rate in the atomizer, (b) denotes the drying air flow rate into the dryer, and (c) represents the feed flow rate.

Subsequently, the dryer information will be input into the respective blocks, systematically organizing the data into four sections: dryer specifications, particle formation, heat and mass transfer, and atomization model. The details of the dryer data are depicted in Figure 38.

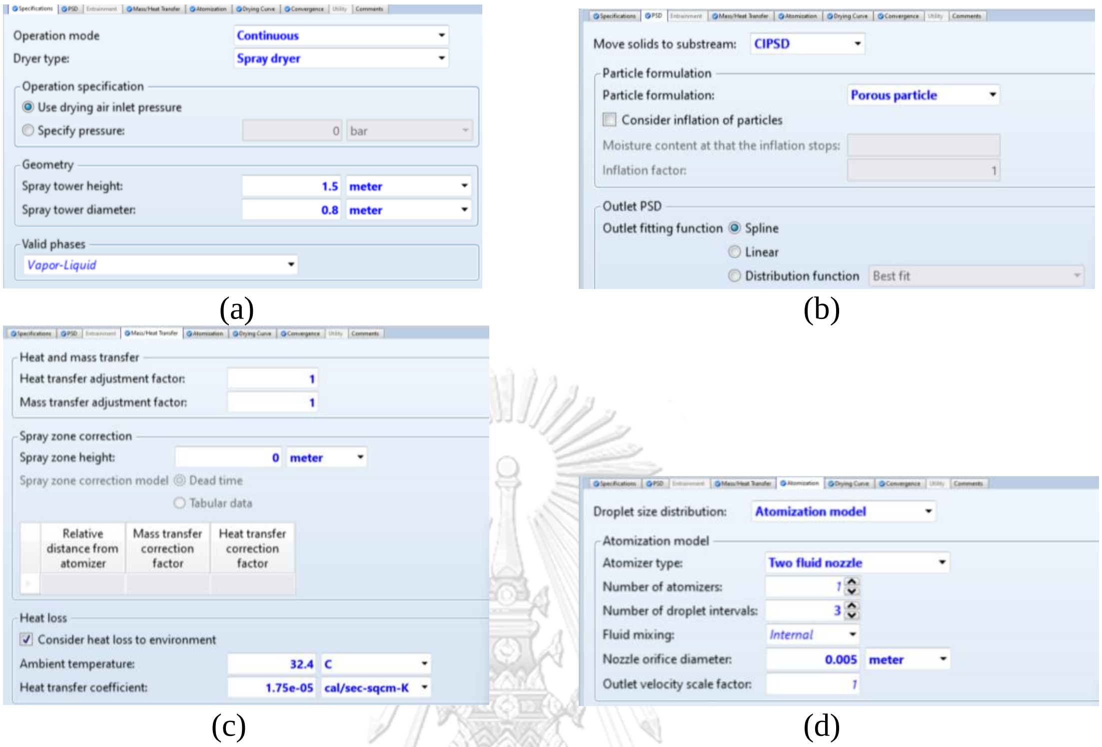


Figure 38 The input data for the dryer in the Aspen Plus program's user interface.

After configuring all input data, the subsequent step involves validating the dryer model through the utilization of the design specification. This process includes varying the heat transfer coefficient while establishing the target parameter as the outlet parameter. Due to the drying air inlet temperature in this example being 140 degrees Celsius, the corresponding drying air outlet temperature will be set at 77 degrees Celsius as shown in Figure 39.

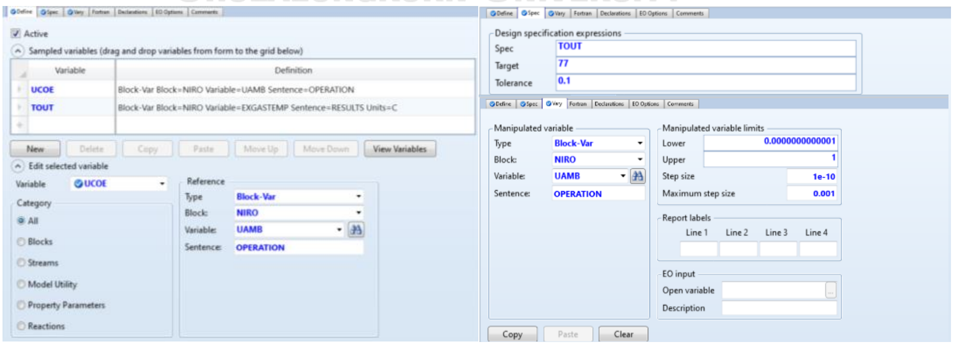


Figure 39 The functionality of the design specification in Aspen Plus's user interface which is employed for the determination of the heat transfer coefficient.

After acquiring the validated model, the design specification function will be reapplied to ascertain the drying air flow rate, utilizing the drying air outlet temperature as the target parameter. The user interface of the design specification function, designed to systematically vary the drying air flow rate to achieve alignment with the outlet temperature, is visually presented in Figure 40.

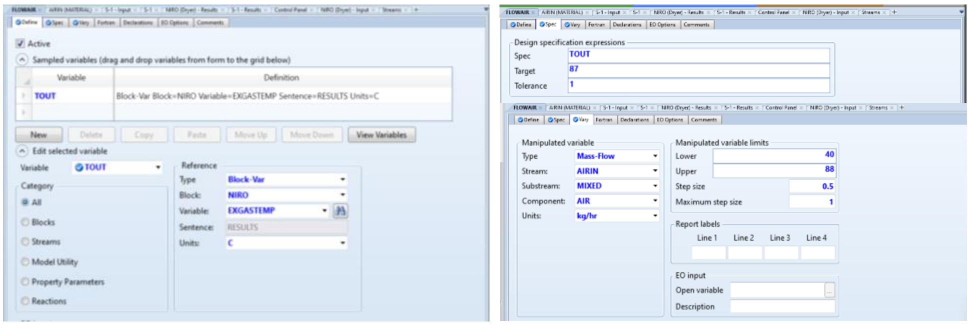


Figure 40 The design specification function for determining the drying air flow rate in the Aspen Plus program's user interface.

Hence, the results obtained from the design specification function are presented in Figure 41. These findings can be applied to upscale the spray drying process from a small to a larger scale, ensuring the constancy of properties across both scales.

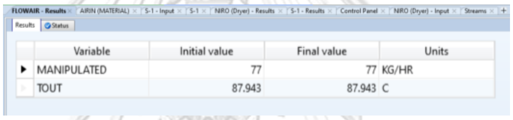


Figure 41 The result of the design specification function for determining the appropriate drying air flow rate.

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