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#### ENERGY SAVING IN SPRAY DRYING PROCESS

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A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Engineering Program in Chemical Engineering

Department of Chemical Engineering

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การอบแห้งแบบพ่นฝอยถือเป็นกระบวนการหลักในการผลิตผงซักฟอก ในกระบวนการนี้ ใช้พลังงานความร้อนปริมาณมากในการระเหยน้ำ การประหยัดพลังงานความร้อนโดยที่ยัง สามารถรักษาสมบัติต่างๆของผลิตภัณฑ์ได้นั้น เป็นปัจจัยสำคัญต่อผลสัมฤทธิ์ในการดำเนินงาน ของกระบวนการ จุดประสงค์ของงานวิจัยนี้เพื่อที่จะลดการใช้พลังงานในกระบวนการผลิตและ เพิ่มผลผลิตของผลิ<mark>ตภั</mark>ณฑ์โดยที่สมบัติของผลิตภัณฑ์ยังอยู่ในค่าที่กำหนด โดยสนใจศึกษาค่า ความหนาแน่นโดยรวมและพลังที่ใช้ในกระบวนการผลิตผงขักฟอกซึ่งใช้ Sodium Tri Poly Phosphate (STPP) และ Zeolite เป็นสารโครงสร้าง การออกแบบการทดลอง (Design Of Experiment DOE) ถูกนำมาใช้ในงานวิจัยนี้เพื่อศึกษาตัวแปรต่างๆได้แก่ ปริมาณลมร้อนที่เข้าใน กระบวนการผลิต ตั้งแต่ 65,000.00 ถึง 72,000.00 กิโลกรัมต่อชั่วโมง ความดันขณะพ่นฝอย ตั้งแต่ 50.00 ถึง 55.00 bars และปริมาณของ slurry ที่เข้าไปในกระบวนการผลิต ตั้งแต่ 25.50 ถึง 31.00 ตันต่อชั่วโมง โดยควบคุมปริมาณความขึ้นของผลิตภัณฑ์และปริมาณความขึ้นใน slurry ให้คงที่ จากการทดลองพบว่าค่า ความหนาแน่นโดยรวมของสารโครงสร้าง STPP และ Zeolite จะ เพิ่มขึ้นอย่างมีนัยสำคัญ ( p value < 0.05) เมื่อลดปริมาณของ slurry ที่เข้าสู่กระบวนการผลิต และเมื่อเพิ่มความดันขณะพ่นฝอย และเมื่อเพิ่มปริมาณลมร้อนเข้าสู่กระบวนการผลิตพบว่าค่า ความหนาแน่นโดยรวมของผงซักฟอกในสารโครงสร้าง Zeolite จะมีค่าเพิ่มขึ้น ในส่วนของการ ประหยัดพลังงานในกระบวนการผลิตนั้นสามารถดูได้จากอัตราส่วนระหว่างปริมาณของพลังงาน ที่ใช้ในกระบวนการผลิตต่อตันของผงซักฟอก จากการทดลองพบว่าทั้งสารโครงสร้าง STPP และ Zeolite มีแนวโน้มที่เหมือนกันคือ เมื่อเพิ่มปริมาณลมร้อน ปริมาณรlurryที่เข้าสู่กระบวนการผลิต และความดันขณะที่พ่นฝอย จะส่งผลให้ค่าอัตราส่วนระหว่างปริมาณของพลังงานที่ใช้ กระบวนการผลิตต่อต้นของผงชักฟอก ลดลงอย่างมีนัยสำคัญ ( p value < 0.05) และเมื่อนำ สภาวะที่ได้จากการทดลองมาประยุกต์ใช้จริงพบว่า สามารถลดต้นทุนในการผลิตได้ถึง 2 ล้าน บาทต่อปี

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Spray drying is the key process in the manufacturing of detergent powder. This process needs a large amount of heat for water evaporation. Saving heat while maintaining product quality is an important factor for the successful functioning of the process. The objective of this study is to minimize energy consumption and to maximize production yield while maintaining product quality. We are interested in bulk density and energy consumption in the production of detergent powder which use Sodium Tri Poly Phosphate (STPP) and Zeolite as component materials. Design of Experiment (DOE) is used in this work in order to optimize the influencing parameters. Experiments are performed with the hot air flow rate varied from 65,000.00 to 72,000.00 kg/hr, slurry feed rate ranged from 25.50 to 31.00 ton/hr and atomized pressure ranged from 50.00 to 55.00 bars. The results of both components show that the bulk density increases with a corresponding decrease in slurry feed rate and an increase in atomized pressure. An increase in hot air flow rate also increases bulk density of Zeolite based detergent powder. The ratio between energy consumption and powder output indicates energy saving. Both STPP based and Zeolite based detergent powder have similar trends. Energy consumption to powder output ratio decreases with an increasing of the slurry feed rate, hot air flow rate and atomized pressure. The optimal condition obtained is applied and can reduce cost 2 million baht a year.

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# ศูนย์วิทยทรัพยากร จุฬาลงกรณ์มหาวิทยาลัย

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#### CHAPTER I

#### INTRODUCTION

#### Definition

Drying process has been using for thousands years to reduce transport weight and to increase the storage life of numerous products and materials. It commonly descries the process to removed volatile substance (moisture) to yield a solid. Moisture in a solid presents as a liquid solution within the solid or even trapped in a micro structure of the solid product, which exerts a vapor pressure less than that of pure liquid, it is called *bound moisture*. Moisture in excess of bound moister is called unbound moisture. When removed moisture from materials two processes occur simultaneously:

- 1. Removed of moisture as vapor from the material surface, depends on the external conditions of temperature, air humid and flow, area of exposed surface, and pressure.
- 2. Movement of moisture internally within the solid, in a function of physical nature of material, the temperature, and its moisture content.

Generally drying process can be classified by method of heat transfer into two types.

- 1. Indirect or non contact Dryers.
- 2. Direct or contact Dryers.

#### Indirect dryer

It can be called non adiabatic unit, separate the heat transfer medium from the product to be dried by a metal wall This dryers are subdivided on the basis applied by radiation or though heat transfer surface and also by the method in which volatile vapors are removed .Heat transfer fluid may be of either condensing type (e.g. steam and diphenyl fluid such as downstream) or liquid (hot water and glycol solution).Indirect dryer have distinctive operating features; (1) risk of cross contamination is avoided since product does not contact the heating medium; (2) since a limited amount of gas is

encountered , solvent recovery is easier than with an adiabatic dryers; (3) dusting is minimized because of the small volume of vapors involved in indirect drying; (4) dryers allow operation under vacuum or in closely controlled atmospheres that can avoid product degradation ; and (5) explosion hazards are easier to control. Typically, indirect dryers are used for small or medium size production .The product from such a unit has a higher bulk density than the same as material process in direct dryers. Particle size degradation usually can be minimized by proper selection of agitator speed or design. The common indirect heated dryers are tubular dryers (with or without vacuum), drum dryers (atmospheric, vacuum, horizontal, or rotary vacuum, and other), hollow disk dryers, paddle dryers, mechanically fluid bed dryers, pneumatically conveyed dryers, cone and twin shell dryers and others.

#### Direct dryers

Direct dryers or adiabatic or convective dryers transfer heat by direct contact of the product by the hot gases. The gases transfer sensible heat to provide the heat of vaporization of the liquid present in the solid .Direct dryers may use air, inert gas, superheated vapor, or product of combustion as the heating medium. Generally used direct dryer in the industrial plant are rotary worm air, fluidized bed, flash dryer, spray dryer. All this have common disadvantage .The amount of air or hot gases required is fairly large, which cause the auxiliary equipment needed (e.g., air heater, blower, and dust corrector) to be sized accordingly; the thermal efficiency is also lower than that of indirect dryers.Generally spray drying had been using direct dryer and it is in the family of suspended particle process (SPP) systems together with fluid-bed drying, flash drying, spray granulation, spray agglomeration, spray reaction, spray cooling, and spray absorption. This is because drying is accomplished while particles are suspended in the air. The main differences between spray, fluid bed and flash drying are shown as:

	Spray dryer	Fluid bed	Flash dryer
Feed	fluid	solid	solid
Residence time in dryer	5-100 s	1-300 min	1-5 s
Particle size (µm)	10-5000*	10-30000	10-300

\*Prime individual particles (agglomerated and granulated particles can be larger).







Figture 1.2 flash dryer and spray dryer

Spray drying is a unique drying process, since it involves both particle formulation and drying. Powder characteristic can be controlled, and powder properties can be maintained constant throughout a continuous operation. With the design of spray dryers available, it is possible to select a dryer layout to produce either fine or coarse particle powders, agglomerates or granulates. Spray drying is by definition the transformation of feed from a fluid state into a dried particulate from by spraying the feed into a hot drying medium. It is a one step, continuous particle processing operation involving drying. The feed can either be a solution, suspension, emulsion or paste. The resulting dried product conforms to powders, granules or agglomerates, which depends upon the physical and chemical properties of the feed and the dryer design and operation. Spray drying is a procedure which in many industries meets dried product specifications most desirable for subsequent processing or consumer usage. Intensive research and development during the last few decades has resulted in spray drying becoming a highly competitive means of drying a wide variety of products. The range of product applications continues to expand, so that today spray drying has connections with many things we use daily.

Generally, the processing step of dry particles from a liquid feed d is the important step for energy consumption. Simply stated, the operating conditions in this step must be the most suitable for energy consumption while maintaining product quality. Widely varying drying characteristics and quality requirements of the thousands of products spray dried determine the selection of the atomizer, the most suitable airflow pattern, and atomization pressure.

Optimization of a spray dryer aims at greater capacity, greater energy efficiency. The most commonly quoted specifications of a powder involve moisture content, bulk density and Particle size. The Design of Experiment (DOE) is used in this work to optimize the influence of hot air flow rate; Slurry feed rate and vacuum pressure on the Bulk density, Particle size and Heat consumption of detergent powder. These key parameters and their influential laws which can give preference to industrial production are investigated.

In this work, we focus at a counter current spray drying tower to investigate the effect of the operating conditions for minimize energy consumption and maximize production yield while maintaining product quality.

#### 1.1 Research Objective

The aim of the study is to determine the optimal operating condition in minimizing energy consumption and maximizing production yield while maintaining product quality.

#### 1.2 Scope of research

The scope of this research is presented as following:

- 1. In this research investigate in 2 formulas on Zeolite and Sodium tripoly phosphate builder.
- 2. Each experiment of each builder is conducted with constant slurry moisture content, slurry temp and powder moisture content.
- 3. The tower inlet and outlet temperatures are investigated in the range of safety limits.
- 4. The variable operating conditions are within the following ranges: Slurry feed rate 25.5 31 ton / hour, Atomize pressure 50 55 bars and Hot air flow rate 65,000 -72,000 kg/hr.
- 5. MINITAB program is used to perform the Design Of Experiments

#### 1.3 Contributions of research

The contributions of this research are as following:

- 1. The optimum operating condition while maintaining product quality.
- 2. The improvement in operating cost.
- 3. The empirical model to predict the effect of each process parameter on product quality and operating cost.

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#### CHAPTER II

#### THEORY

The development of spray drying equipment and techniques evolved over a period of several decades from the 1870s through the early 1900s. The first known spray dryers used nozzle atomizers, with rotary atomizers introduced several decades later. Because of the relatively unsophisticated designs of the early spray dryers and practical difficulties in operating them continuously, very little commercial use of the process was made until the 1920s. By the second decade of the twentieth century, the evolution of spray dryer design made commercial operations practical. Milk drying was the first major commercial application of the technology. During the next 20 years, manufacturers developed designs to accommodate heat-sensitive products, emulsions and mixtures. Spray drying came of age during World War II, with the sudden need to reduce the transport weight of foods and other materials. This surge in interest led to developments in the technology that greatly expanded the range of products that could be successfully spray dried.

#### 1.1 Process stages and layout of spray dryers

Figure 2.1 is shown the example of counter current layout. Generally, spray drying consists of four process stages



Figure 2.1 spray dryer process layout

#### 1.1.1 Atomization of feed into a spray tower

This is the process which turns a liquid feed or slurry feed into small droplets immediately prior to drying. The size of the droplet formed controls the size of the powder particles coming out of a single stage spray dryer. The two principle means of atomization are centrifugal (Rotary atomizer) and pressure nozzle. Both of the atomization have their advantages and disadvantages and the choice is dependent on both feed and powder properties of the specific application.

Generally of spray drying process rotary atomizer and nozzle are used to from sprays. With rotary atomizers centrifugal energy is utilized. There are two categories of rotary atomizer;

- a. Atomizer wheels
- b. Atomizer discs

With nozzle atomization, pressure, kinetic or sonic energy is utilized. There is a wide range of nozzle sizes and designs to meet spray drying needs. Feed capacities per nozzle are lower than per rotary atomizer, leading to nozzle duplication to meet high feed rate requirements.

#### Rotary Atomizer

Atomization is achieved by feeding liquid onto a high speed wheel or disc rotating. The feed is centrifugally accelerated to high velocity before being discharged into an air – gas atmosphere. The liquid is distributed centrally on the wheel or disc. The liquid extends over the rotating surface as a thin film. Rotary atomization is often termed centrifugal atomization, but this can be a little misleading as centrifugal energy is also utilized to a certain degree in centrifugal pressure nozzle, where liquid is given rotation motion. The particle size of the powder produced is controlled through change of wheel speed. The rotary atomization shown in figure 2.1



#### Figure 2.2 Rotary atomization

Designs of atomizer wheel have vanes, spacers or bushing. Vanes are high, wide, straight or cured; spacers cylindrical or formed; bushing circular or square. Vaned atomizer wheels are used in many industries to produced spray of high homogeneity. Atomizer wheels with bushing are used in more specialized product.

#### Pressure Nozzles

The function of the nozzle atomizer is the acceleration and disintegration of bulk liquid flow, terminating with the dispersion of the resulting droplets to form a spray. The feed concentrate is fed to the nozzle under pressure. Pressure energy is converted to kinetic energy, and feed issues from the nozzle orifice as a high speed film that readily disintegration into a spray as the film is unstable. The feed is made to rotate with the nozzle, resulting in cone shaped spray patterns emerging from the nozzle orifice. Sprays from pressure nozzles handling high feed rate are generally less homogeneous and coarser than sprays from vaned wheels. Duplication of nozzles is required to atomize high feed rate successfully. Nozzles are generally for coarse particle and for good free flow ability. The spray from leaving a pressure nozzle is shown in figure 2.3



Figure 2.3 pressure nozzle

Variation of pressure gives control over feed rate and spray characteristic. Mean size of spray is directly proportional to feed rate and spray and viscosity, and inversely proportional to pressure. The comparison between pressure nozzle and rotary atomizer are shown in table 2.1.

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#### Table 2.1 Atomizer Features

Rotary atomizer	Pressure nozzle
Easy control of particle size	Less easy control of particle size
Large flow areas	Small flow areas
Single atomizer for low and high capacities	Nozzle duplication for high capacities
handles slurries and crystalline feedstocks	Fine feed filtering require
Particle size virtually independent of feed rate	Narrow operating feed rate range
Capacity indepentdent of fedd pressure	Cap <mark>acity propotional to square root of pressure</mark>
Large particles dried only in large diameter drying chamber	Large particles dried in smaller diameter drying chambe
unit cost with pump comparable	unit cost with pump comparable
Low pressure feed system	Hgh pressure feed system
Fine - medium size paraticl <mark>es in</mark> dividual	Coarse free -flowing particles
Deposit tendencies on wall at wheel level	Less t <mark>endency to deposit on w</mark> all

#### Two fluid Nozzle

The two fluid nozzles are shown in figure 2.4. The feed and atomizing gas are passed separately to the nozzle head. High air velocities are generated within the nozzle for effective feed contact which breaks up the feed into spray of fine droplet. Sonic velocity is generated. The air steam may be rotated within the nozzle, and feed is contacted either within the nozzle or as the liquid emerges from the orifice. The nozzles operate successfully at low pressure. The feed system includes low pressure pumps, although at low feed rates the suction cause by the air ejector effect will be sufficient to draw liquid into the nozzle. Adjustment of the air flows controls the degree of atomization at contact feed rate. Two fluid nozzles have the advantage of handling highly viscous feeds producing sprays of medium coarseness, but of poor homogeneity. For low viscosity feeds, fine particles can be produced, although the resulting dried powder may be agglomerate. There is much likelihood of high occluded air content within the particles.



Figure2.4 Two fluid atomization

#### 1.1.2 Spray – air contact

The manner in which spray contacts the drying air is an important factor in spray dryer design, as this has great bearing on dried product properties by influencing droplet behavior during drying. There are three modes of contact

#### Co-current

With a co-current dryer (Fig. 2.5), the spray is directed into the hot air entering the dryer and both pass through the chamber in the same direction. Co-current dryers are the preferred design for heat sensitive products do to the hottest drying air contacts the droplets at their maximum moisture content. Spray evaporation is rapid, and the temperature of the drying air is quickly reduced by the vaporization of water. The product does not suffer from heat degradation because the droplet temperature is low during most of the evaporation time. Once the moisture content reaches the target level, the temperature of the particle does not increase greatly because the surrounding air is now much cooler. Dairy and other heat-sensitive food products are usually dried in cocurrent dryers.



Figure 2.5 Co-current Flow Dryer

#### Counter-Current flow

In this dryer design (Fig. 2.6), the spray and the air are introduced at opposite ends of the dryer, with the atomizer positioned at the top and the air entering at the bottom. A counter-current dryer offers more rapid evaporation and higher energy efficiency than a co-current design. Do to driest particles are in contact with hottest air, this design is not suitable for heat-sensitive products. Counter-current dryers normally use nozzles for atomization because the energy of the spray can be directed against the air movement. Soaps and detergents are commonly dried in counter-current dryers.



Figure 2.6 Counter-current Flow Dryer

From Unilever research department investigated that with design of spray tower which on concurrent or countercurrent operation, the majority of systems being of the former type. Little if any work has been published comparing the design, operation and product particle morphology of systems which can be operated in either mode. The Residence Time Distribution results are shown graphically in figure 2.7 where the tracer concentration C (t) is plotted as a function of time.





versus time

Concurrent process has a reduction in mean particle residence time and an increased in the variance of the residence time distribution when compared with counter current spray drying. They presented the concurrently spray dried powder had a lower mean particle size and slightly broader particle size distribution. The bulk density was lower and the flow properties were worse than the counter currently dried powder. The carryover of cyclone fines was also increased by approximately 10%. The reason is probably that the concurrently spray dried powder had a faster initial drying rate which resulted in less agglomeration during drying and a higher particle porosity. The lower agglomeration resulted in a reduced mean size. The particle morphology of the same feedstock dried in the same tower geometry was quite different, depending upon the airflow being con- or counter-current to the spray. They show that the concurrently

spray-dried particles were lower in bulk density and had a reduced mean particle size. The particle size distribution was similar indicating that the differences were because of the different particle morphology. The particles from concurrent drying were of higher porosity and less agglomerated. This was indeed confirmed when examined under the scanning electron microscope.

#### Mixed flow dryer

Dryers of this type (Fig. 2.8) combined both co current and counter current flow. In a mixed flow dryer, the air enters at the top and the atomizer is located at the bottom. Like the counter-current design, a mixed flow dryer exposes the driest particles to the hottest air, so this design is not used with heat-sensitive products.



#### 1.1.3 Process layout

The co current, counter current and mixed flow dryers can be adopted in numerous different process layouts

#### Open cycle dryer

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In an open cycle dryer (Fig. 2.9) drying air is drawn from the atmosphere, heated, conveyed through the chamber and then exhausted to the atmosphere. This is by far the most commonly used



Figure 2.9 open cycle co-current flow layout

Closed cycle dryer

A closed cycle dryer (figure 2.10) recycles the drying gas, which may be air or more commonly, an inert gas such as nitrogen. Closed cycle units are the dryers of choice when:

- Feedstock consists of solids mixed with flammable organic solvents.
- Complete recovery of solvent is required.
- The products are toxic
- Pollution due to vapor, particulate emissions or odor is not permitted.
- Explosion risks must be eliminated.
- The powder will degrade by oxidation during drying.



Figure 2.10 closed cycle co-current flow layout

#### Semi-closed cycle dryer

This design is a cross between open and closed cycle dryers (Fig 2.11) and it is not gas tight. There are many variations on this design, with the most important being the "direct heated" or "self-inertizing" system. In the self-inertizing design, a direct-fired heater is used and the air entering the system is limited to that required for combustion. An amount of air equal to the combustion air is bled from the system at the other end of the process. The gas (mainly products of combustion) is recycled through the dryer. The recycled gas has very low oxygen content, making it suitable for materials that cannot be exposed to oxygen, due to explosive hazard or product degradation.



Figure 2.11 Semi Closed Cycle Co-current Flow Layout

#### 1.1.4 Drying of spray

When the droplet of spray comes into contact with the hot air, evaporation takes place from the vapour film which is quickly established at the droplet surface. The temperature at the droplet surface approximates to the wet bulb temperature of the hot air. Evaporation takes place in two stages. The first stage is sufficient moisture within the droplet to the replenish that lost at the surface. Diffusion of the moisture within the droplet maintains saturated surface conditions and as long as this lasts, evaporation takes place at a constant rate. This is termed the constant rate period or first period of drying. When the moisture content becomes too low to maintain saturated conditions, the so called critical point is reached and a dried shell from at the droplet surface. Evaporation is now dependent upon the rate of moisture diffusion through the dried surface shell. The thickness of the dried shell increases with time, causing a decrease in the rate of evaporation. This is termed the falling rate period or second period drying. Thus a substantial part of the droplet evaporation takes place when the droplet surfaces are saturate and cool. Drying chamber design and air flow rate provide the droplet residence time in the chamber, so that the desired droplet moisture removal is completed and product removed from the dryer before product temperatures can rise to the outlet drying air temperature of the chamber. Hence, there is a little likelihood of heat damage to the product. During evaporation, the atomized spray distribution undergoes change. Different products exhibit different evaporation characteristics. Some tend to expand, other collapse, fracture or disintegrate, leading to porous, irregularly shaped particle. The characteristic of particle shown in figure 2.12

# ศูนยวทยทรพยากร จุฬาลงกรณ์มหาวิทยาลัย



Figure 2.12 mechanism of droplet drying illustrated in simplified manner.

#### 2.1.5 Separation of dried product from the air

Separation is carried out partly within the drying chamber itself and partly in secondary separation equipment. In general, it is easy to remove 90% or more of the powder, but removal of the remainder becomes problematic. Cyclone separators(figure 2.13) operate on the 'momentum separation' principle (centrifugal action) and are extensively used in large scale dryers for removal of fines.

Charm (1971) has given an equation which relates the dimensions of a cyclone to the smallest particle  $(D_p)$  which can be separated:

 $D_p^2 = (3.6 A_i D_0 \mu)/(^{1}ZDV_0 d_s),$ 

where:  $D_p$  = diameter of particle;  $A_i$  = inlet cross sectional area of cyclone;  $D_0$  = diameter of outlet of cyclone;  $\mu$  = viscosity of the fluid; Z = depth of the separator; D = diameter of the separator;

 $V_0$  = velocity of air/powder mixture entering the cyclone; and  $d_s$  = density of the particle.



Figure 2.13 Cyclone Separation of dried product from the air

From the equation it appears that in designing a cyclone the depth and diameter should be as large as possible. Increasing the air velocity is also important. Industrial experience has shown that efficiency is also affected by the powder concentration in the air stream. For this reason, it is better to use several cyclones in parallel than just one single separator. Since cyclones do not always allow complete separation of 'fines' other systems are also in use, including filters, scrubbers, or electrostatic precipitation equipment. Cloth-bag filters are very effective systems but are expensive in labor cost to maintain. Besides, the fabric is weakened by high temperatures. C. G. J. Baker and K.A. McKenzie survey on the UK companies in the chemicals, ceramics and food industries. This sought information relating to the nature of the feedstock and product, the type of dryer employed, and its operating conditions. Usable returns were received for 32 dryers. These are summarized in table2.2.

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Industry Sector	Ceramics	Chemicals	Food
No. of Dryers	7	17	8
Atomizers	Rotary – 3	Rotary – 10	Rotary - 5
	Nozzle – 4	Nozzle – 6	Nozzle – 3
	2-fluid Nozzle - 0	2-fluid Nozzle – 1	2-fluid Nozzle - 0
Flow	Cocurrent – 4	Cocurrent – 11	Cocurrent – 6
	Countercurrent - 0	Countercurrent - 5	Countercurrent – 0
	Mixed - 3	Mixed - 1	Mixed - 2
Fuel	Steam - 0	Steam - 0	Steam – 4
	Gas – 7	Gas - 16	Gas – 1
	Oil – 0	Oil – 1	Oil - 3
No. of Stages	1-7	1-15	1-0
	2-0	2-2	2-7
	3-0	3-0	3-1
Range of Evaporation Rates, t/h	0.3-6.2	0.1-12.3	0.6-4.1
Range of Total Heat Inputs, GJ/h	1-23.1	1-58.4	3.8-13.4

Table 2.2: Summary of the characteristics of the dryers covered in the survey

#### 2.2 Advantages and disadvantages of spray drying

With spray dryer operating in industries that range from aseptic pharmaceutical processing through to mining operations and handling feed rates that range from a few kilograms per hour to well over 100 tonnes per hour per dryer, it is obvious there are many positive aspects that have led to establishing spray drying as a most important industrial drying system today. For example:

- The specification or powder quality remains constant throughout the entire dryer operation irrespective of the length of the dryer run when drying conditions are held constant.
- 2. Spray dryer operation is continuous and easy, operation is adaptable to full automatically control, response times fast. One operator can handle more than one automatically controlled spray dryer if located together in one complex.
- 3. A wide range of dryer designs are available. Product specifications are readily met through selection of the appropriate spray dryer design and its operation.
- 4. Spray drying is applicable to both heat sensitive and heat resistant material.

- 5. Feedstocks in solution, slurry, emulsion, paste or melt from can be handled if pumable, whether or not they be corrosive or abrasive.
- Spray dryers can be designed to any individual capacity requirement. The largest spray dryer absorber complex in operation today handles over five million cubic metres of fuel gas per hour.
- 7. There is extensive flexibility in spray dryer designs. Designs are available to handle:
  - Evaporation of organic solvent based feed stocks without explosion and fire risks;
  - b. Evaporation of aqueous feedstocks that from powders that are potentially explosive when mixed in air ;
  - c. Evaporation of aqueous feedstocks where the drying process odour discharge;
  - d. Drying of toxic materials;
  - e. Drying of feedstocks that require handling in aseptic/ hygienetic drying condition;
  - f. Drying of feedstocks to granular, agglomerated and non agglomerated powder.

Spray drying is disadvantage by high installation cost. Industrial units are physically larger per unit powder output than other dryer types. This makes spray dryers expensive to fabricate. Furthermore their large diameter or tall drying chambers require expensive building and/or supporting structures. However, the latest designs with integrated fluid beds in drying chambers have decreased space requirements significantly. The spray dryer, begin a convection dryer, has a poor thermal efficiency unless very high inlet drying temperatures can be used. This is possible only in the minority of case due to product heat degradation effects by high temperature spray drying. Drying air exhausted from spray dryers contains large amounts of low grade waste heat. It is expensive to remove this heat in heat exchange equipment since such equipment must handle powder laden air at saturated or near saturated conditions, and this leads to the need for sophisticated heat exchanger design. Development towards more compact lower energy consuming dyer designs is prime activity of spray dryer manufacturers. The current development within integrated fluid bed spray dryer concept has done much to counter the most often cited disadvantage of spray drying, i.e. its relatively poor thermal efficiencies at inlet temperature below 350 °C : the inlet temperature range that covers the vast majority of industrial spray drying operation

#### 2.3 Everyday applications

Spray drying has moved into all major industries ranging from production in the most delicate of conditions laid down in food and pharmaceutical manufacture right through to the high tonnage outputs within such heavy chemical fields as mineral ores and clays. There are many products and articles in use around us each day to exemplify the extensive usage of spray drying. This is apparent if we consider just one aspect of common interest to us all, namely our domestic life.

### Spray Dryers for Industrial Production Pharmaceutical Industry

Generally in pharmaceutical Industry used spray drying process for many objectives such as used for agglomerate powder for: Analgesics Antibiotics, Enzymes, Plasma /plasma substitutes, Vaccines, Vitamins and Yeasts. Spray dryers designed specialty for integration into batch or continuous operations under sanitary or aseptic conditions. The systems are also available for taste masking and encapsulate powder. Dryers with integrated fluid beds are ideal for producing non-dusty powders for perfect tablet ting. Other equipment supplied to the pharmaceutical processing industries includes fluid bed tablet coaters, granulators and dryers, microwave dryers, concentrators (thermal/freeze), homogenizers and powder blenders.

#### Food and Dairy Industry

Foodstuffs and dairy products in powder used spray drying process for many objectives, agglomerate powder is the one for: Baby food, Cheese/ whey products,

Coconut milk, Coffee/ coffee substitutes, Coffee whitener, Eggs, Flavours, Maltodextrine, Mild, Soup mixes, Soy- based food, Spices/ herb extracts, Sugar-based food, Tea, tomato, Vegetable protein. Spray drying is ideal for these heat sensitive products, where selection of system an operation is the key to high nutritive and quality powders of precise specification."Instant", highly soluble powders are a specialty of spray dryers featuring integrated belts or fluid beds. All components in contact with product comply with hygienic processing standards. Today's plants have special sanitary features, automatic cleaning (CIP) and bagging-off systems. Associated equipment includes agglomerators, evaporators, freeze concentrators, homogenizers, lecithination units and powder blenders.

#### Chemical Industry

Many Specialty chemicals used spray drying process such as: Catalysts, Detergents, Dyestuffs, Fine organic/inorganic chemicals, Agro- chemicals, Chelates, Fungicides, Herbicides and Insecticides. Spray dryers for the chemical industries produce a variety of powdered, granulated and agglomerated products in systems that minimize formation of gaseous, particulate and liquid effluents. High efficiency scrubber systems and high performance bag filters prevent powder emission, while recycle systems eliminate problems of handling solvents, product toxicity, and fire explosion risks. Special component designs (e.g. atomizers) are available for abrasive and corrosive feed stocks. High thermal efficiencies, low maintenance costs, full environmental protection, computerized control, dust-free working area are some of the features of today's spray dryers.

#### Polymer Industry

Many of Polymers production plant used spray drying to produced products such as : ABS, PVC, PMMA,UF/MF resins, Polymer dispersions and solutions in water or organic solvents are spray dried under closely controlled operating conditions, producing powders to precise particle size, heat treatment, and redispersibility specifications. Low softening point products are produced continuously in plants with air-brooms, air-sweeps or integrated fluid beds. For drying moist polymer powders, NIEO offers fluid beds.

#### Ceramic Industry

In ceramic industry production used spray drying process for advanced ceramic formulations, Carbides, Ferrites, Nitrides, Oxides, Silicates, Steatites and Titanates. Spray drying is applicable to tile and electronic press powders, and plays an important role in the industrial development of high performance (advanced) ceramics. The ability to meet particle size distribution requirements, produce a spherical particle form and handle abrasive feed stocks is an important reason for the widespread use of spray dryers in the ceramic industries.

### 2.4 Heat and mass balance in spray dryers The Drying Curve

For each and every product, there is a representative curve that describes the drying characteristics for that product at specific temperature, velocity and pressure conditions. This curve is referred to as the drying curve for a specific product. Fig 2.14 shows a typical drying curve. Variations in the curve will occur principally in rate relative to carrier velocity and temperature.



Fig 2.14 drying curve

Drying occurs in three different periods, or phases, which can be clearly defined. **The first phase**, or *initial period*, is where sensible heat is transferred to the product and the contained moisture. This is the heating up of the product from the inlet condition to the process condition, which enables the subsequent processes to take place. The rate of evaporation increases dramatically during this period with mostly free moisture being removed. In some instances, pre-processing can reduce or eliminate this phase. For example, if the feed material is coming from a reactor or if the feed is preheated by a source of waste energy, the inlet condition of the material will already be at a raised temperature.

The second phase, or *constant rate period*, is when the free moisture persists on the surfaces and the rate of evaporation alters very little as the moisture content reduces. During this period, drying rates are high, and higher inlet air temperatures than in subsequent drying stages can be used without detrimental effect to the product. There is a gradual and relatively small increase in the product temperature during this period.

#### Heat and mass balance

Air product flow and temperature data for assessing drying performance are obtainable form heat and mass balance data. For continuous operation with negligible hold up of the product in the drying chamber, the mass input of air and feed in unit time equals the mass output of air and product. Heat input of air and feed equals heat output of air and product plus the heat losses from the drying chamber. For semi continuous operation, shut down is often due to build up of product in the dryer. The difference in product input and output equals the accumulation. Heat and mass balance are in the figure 2.15. For calculation of air feed and product enthalpies, the freezing point of water
is used as a reference temperature. It is common to base moisture balances on a unit weight of bone dry product.



Figure 2.15 Dryer data for calculation of heat and mass balance

M<sub>s</sub>: weight per units per hour of dry solid enter the dryer in feed

W<sub>s1</sub>: moisture in feed in term units of moisture per unit of dry solid in weight

W<sub>s2</sub>: moisture in product in term units of moisture per unit of dry solid in weight

Ts1: The temperature of feed when atomization

Ts2: Product temperature

Ta1: Hot air inlet temperature

Ta2: Exhaust air outlet temperature

G<sub>a</sub>: Hot air flow rate

H1: Humid in inlet air

H2: Humid in exhaust air

# Moisture balance

Moisture entering in feed =  $M_s \times W_{s1}$ 

Moisture entering in hot air =  $G_a x H_1$ .

Moisture leaving the dryer in the dried product =  $M_s \times W_{s2}$ 

Moisture leaving the dryer in the dried product =  $G_a x H_a$ 

For no production accumulation in the chamber,

Input = Output

Thus;

 $M_s \times W_{s1} + G_a \times H_1 = M_s \times W_{s2} + G_a \times H_2$ 

Or

 $M_{s}(W_{s1} - W_{s2}) = G_{a}(H_{1} - H_{2})$ 

The similar procedure for Enthalpies

Enthalpy or heat balance

Enthalpy of air entering dryer =  $G_a \times Q_{a1}$ 

Enthalpy of air entering dryer  $= M_s \times Q_{s1}$ 

Enthalpy of exhaust drying air =  $G_a \times Q_{a2}$ 

Enthalpy of dried solid =  $M_s \times Q_{s2}$ 

Heat in = heat out + Heat loss

 $G_a \times Q_{a1} + M_s \times Q_{s1} = G_a \times Q_{a2} + M_s \times Q_{s2} + Q_L$ 

Where  $\boldsymbol{Q}_{\!\scriptscriptstyle L}$  = heat losses from the dryer outer cladding and structural supports.

The heat loss is expressed by the standard heat transfer equation:

$$Q_1 = UA\Delta T$$

For well insulated drying chambers heat losses are low. For non insulate drying chambers, or chambers designed with cooling air jacket to maintain cool dryer walls, heat losses from the dryer are high, and QL becomes a significant term in the equation for heat balance. The enthalpy of the feed (Qs1) as it enters the sum of the enthalpy of the dry solid and the moisture as liquid.

Thus

$$Q_{s1} = C_{DS} (\Delta T) + W_{s1} C_{w} (\Delta T)$$

Where  $C_{DS}$  = heat capacity of dry solid

 $C_w$  = heat capacity of moisture (in liquid from)

 $\Delta T$  = difference in temperature between feed temperature and the reference

temperature level.

The enthalpy of the drying medium (Qa) whether entering or leaving the dryer is

expressed in terms of the humid heat, the absolute humidity, and the latent heat of

vaporization of water at freezing point, where

 $Q_a = C_s (\Delta T) + H\lambda$ 

 $\lambda$  = 597.3 kcal/kg at 0 °C (760 mm Hg)

Darko Veli et al. (2003) descried the drying process using thermodynamic calculations of heat and mass balance (figure 2.16). They used the program to possible to reduce operating costs and to improve capacity per unit of drying equipment. The

minimization of energy consumption during the drying operation can be achieved by recycling exhaust air



Figure 2.16 Process flow sheet for calculate heat and mass balance in spray dryer

Calculation data example of program application	
Amount of wet material (m <sub>wm</sub> )	25,000 kg/h
Moisture content (X <sub>wm</sub> )	62 %
Dry matter content (DM <sub>c</sub> )	38 %
Temperature of wet material (T <sub>wm</sub> )	80 °C
Amount of dried material (mdr)	17,613.64 kg/h
Moisture content of dried material (Xd)	12 %
Temperature of dried material (Tdm)	80 °C
Leak air (LA)	5 %
Temperature of hot inlet air (Tin)	50 °C
Temperature of outlet air (Tout)	110 °C
Temperature of ambiental air (Ta)	20 °C
Net caloric value of fuel oil (NCVF)	41,200 kJ/kg
Enthalpy of ambiental air (h3a)	20.11 kJ/kg

Enthalpy of outlet air (h2a) Enthalpy of inlet air (h1a) Enthalpy of water on Twm (h1w) Enthalpy of water on Tout (h2w) Enthalpy of water vapour on Tin (h3w) Specific heat of nitrogen (cpN2) Specific heat of CO2 (cpCO2) Specific heat of CO2 (cpCO2) Specific heat of dry material (cp (Mwet)) Specific heat of dried material (cp (Mdr)) Heat from evaporation of water (r(H2O)) Specific heat of fuel on 60 °C (cp f ) Temperature of fuel (Tf ) 110.99 kJ/kg 466.2 kJ/kg 334.9 kJ/kg 2691 kJ/kg 3383 kJ/kg 1.112 kJ/kg °C 1.187 kJ/kg °C 2.3442 kJ/kg °C 1.633 kJ/kg °C 2500 kJ/kg 2.199 kJ/kg °C

## Calculation of heat and mass balance

Amount of dried materi <mark>a</mark> l	$m_{dm} = m_{wm} (100 - X_{wm})$	)/(100 –DM <sub>c</sub> ) m <sub>dm</sub> = 17,613	8.6364 kg
Amount of evaporate water	$m_{ev} = m_{dr} (X_{wm} - X_d)$	/(100 – X <sub>wm</sub> ) m <sub>ev</sub> = 7386.3	36364 kg
		kg/	m3/h
Required amount of hot (inlet	t) air m <sub>ha</sub> = m <sub>ev</sub> .rH2O/(h	$_{1a}$ -h <sub>2a</sub> ) m <sub>ha</sub> = 51,985.893 4	43772.12
Heat for water evaporation	$Q_1 = m_{ha} \cdot h_{1a}$	Q <sub>1</sub> = 24,235,82	3.4 kJ/h
Outlet air heat content	$Q_2 = m_{ha} (h_{2a} - h_{3a})$	$Q_2 = 4,724,477.$	97 kJ/h
Total amount of heat	$Q_{U} = Q_{1} + Q_{2}$	Q <sub>U</sub> = 28,960,301.3	3 kJ/h
		kg/h	m3/h
Required amount of fuel	$m_f = Q_U / NCV$	F m <sub>f</sub> = 702.919935	747.79
Evaporation efficiency	$C_{ev} = m_{ev}/m_{f}$	C <sub>ev</sub> = 10.5081152 kg H <sub>2</sub> O	/kg fuel
Leak air (approx. 5% of inlet a	air) m <sub>la</sub> = m <sub>ha</sub> 0.0	5 m <sub>la</sub> = 2599.29466	i kg/h
Heat from ambiental air	$Q_a = m_{ha} \cdot h_{3a}$	Q <sub>a</sub> = 1,045,436.3	1 kJ/h

Heat from fuel  $Q_f = m_f \cdot T_f \cdot cp_f$  $Q_f = 92,743.2563 \text{ kJ/h}$  $Q_{comb} = m_{f}$  . NCVF  $Q_{comb} = 28,960,301.3 \text{ kJ/h}$ Heat from combustion  $Q_{Fin} = Q_{a} + Q_{f} + Q_{comb}$   $Q_{Fin} = 30,098,480.9 \text{ kJ/h}$ Total amount of heat in furnace Fuel burning The fuel used is mixture of light and medium light fuel oil composition 87%C and 11%H The amount of oxygen and air can be calculated as follows  $m(O2) = 0.01 \cdot (2.667 \cdot C + 8 \cdot H) = 0.01 \cdot (2.667 \cdot 87 + 8 \cdot 11)$ mO2 = 3.19 kgmAIR = 0.115 .C + 0.345 .H = 0.115 .87 + 0.345 .11 mAIR = 13.79 kg1.3 coefficient of air surplus mAIR = 1.3 . 13.79 = 17. 927 kgair/kgfuel m(N2) = 0.786 . mAIR = 0.786 . 17.927 = 13.76 kg N2/kg fuel Air for combustion of 1 kg fuel 17.927 kg Total amount of air for combustion  $m_{aircomb} = mf \cdot m_{AIR}$  $m_{aircomb} = 12,601.2457 \text{ kg}$ Total amount of heat from furnace Heat from hot air Qha =  $[m_{ha} - m (air_{comb})]$ .  $h_{1a}$ Qha = 18,361,122.6 kJ/h Heat from water in combustion Q(H20)= mf.m(H2O).h1(H2O) Q(H2O)=2,461,207.38kJ/h Burning of 1 kg of fuel 1.035 kg water is produced Heat from nitrogen  $Q(N2) = mf \cdot m(N2) \cdot cp(N2)$ . Tin Q(N2)=3,746,043.09 kJ/h Burning of 1 kg fuel 10.65 kg nitrogen is produced Heat from CO2 Q(CO2) = mf. m(CO2). cp(CO2) + Tin Q(CO2) = 1,167,695.17 kJ/h Burning of 1 kg fuel 3.11 kg CO2 is produced Total heat from furnace  $Q_{Fout}$  = Qha + Q(H2O)+Q(N2)+Q(CO2)  $Q_{Fout}$  = 25,736,068.3 kJ/h Furnace losses Q<sub>FL</sub> = QFin - QFout Q<sub>FL</sub> = 4,362,412.63 kJ/h Heat entering in tower (Qt) [kJ/h] Heat from furnace  $Q_{E} = 25,736,068.3 \text{ kJ/h}$ Qsl = 4,688,400 kJ/h Heat from slurry Qsl =  $m_{wm}$  . cp S . TS Heat from leak air  $Q_{IA} = Qa$  .h3a Q<sub>LA</sub> = 52,271.8155 kJ/h

Total amount of heat in tower Qt in = QFout + Qsl + Qla $Q_{tin} = 30,476,740.1 \text{ kJ/h}$ Heat leaving tower (Qout) [kJ/h] Heat from exhaust air Qa out = (mha + mla). h2a Qa out = 6,058,409.99 kJ/h Heat from evaporation of water Q<sub>ewout</sub>=(m<sub>ev</sub>+(m(H20) .mf )).h<sub>2w</sub> Q<sub>ewout</sub>= 21,834,466.6 kJ/h Heat from N2 Q(N2)out = mf . m(N2) .cp(N2).Tout Q(N2)out = 893,465.714 kJ/hHeat from CO2 Q(CO2)out =mf.m(CO2).cp(CO2).Tout Q(CO2)out = 167,125.892 kJ/h Heat from dried material Qout = Qa out + Qew out + Q(N2)out + Q(CO2)out + Qdm Qout = 31,254,513.7 kJ/h Total amount of heat in plant Qplant =  $Q_{Fin}$  + Qla + Qs Qplant = 34,839,152.7 kJ/h Heat utilization Heat for water evaporation Qev = mev . [h2(H2O) - h1(H2O)] Qev = 17,403,011.4 kJ/h Heat from dried material Qdm= mdm.cp dm.Tdm Qdm = 2,301,045.45 kJ/hHeat on exit Qout = Qa out + Q(N2)out + Q(CO2)out + QLA + (mf. 1.035.h2w)Furnace losses QFL = QFin - QFout QFL = 4,362,412.63 kJ/hTower losses QTL = Qplant – (Qev + Qop+ Qout+ QFL) QTL = 1,643,647.79 kJ/h

### 2.5 Efficiency calculations

Spray drier design is directed towards achieving desired-product properties at the highest possible efficiency. Calculate the highest possible thermal and evaporative efficiencies obtained from the experiment. Are these acceptable? If not, discuss why.

### Thermal efficiency:

The thermal efficiency of a spray drier depends upon the operation temperatures. Overall thermal efficiency  $\eta_{\text{overall}}$  is defined as the fraction of total heat supplied to the drier used in the evaporation process. It can be approximated to the relation:

$$\eta_{\text{overall}} = \frac{T_1 - T_{2ad}}{T_1 - T_0} \times 100$$

where  $T_1$  = air temperature entering into the drier (heated).

 $T_{2ad}$  = air temperature at end of evaporation (adiabatic).

 $T_0$  = ambient temperature

### Evaporative Efficiency:

Is defined as the ratio of the actual evaporative capacity to the capacity obtained in the ideal case of exhausting air at saturation:

$$\eta_{\text{evaporative}} = \frac{T_1 - T_{2ad}}{T_1 - T_{saturation}} \times 100$$

Where  $T_{\text{saturation}}$  = the adiabatic saturation temperature corresponding to the inlet temperature  $T_1$ .

## 2.6 Factorial Design

Many experiments involve the study of the effects of two or more factors .In general , factorial designs are most efficient for this type of experiment .By a factorial design ,it mean that in each complete trial or replication of the experiment all possible combinations of the levels of the factors are investigated.

The effect of a factor is defined to be the change in response produced by a change in the level of the factor. This is frequently called **a main effect** because it refers to the primary factors of insert in the experiment.

In some experiments, it occurs something difference in response between the level of one factor is not the same at all levels of the other factors .When this occurs, there is an interaction between the factors. Generally, the factorial design is often used with; The 2 <sup>k</sup> factorial design, The 2 <sup>k-p</sup> factorial design and the General full factorial design ( where k is the total of factor in the experiment ). The 2 <sup>k</sup> factorial design and the 2 <sup>k-p</sup> factorial design will use when the factor has got 2 levels and the 2 <sup>k-p</sup> Factorial design prefers to use when the experiment has got many factors to reduction the sample size of the experiment .The General full factorial design will use when levels of factor more than 2 levels .

In this research, the 2 <sup>k</sup> Factorial design will use to study 3 factors and each factor has got 2 levels.

# The 2<sup>k</sup> factorial design

The most important of these special cases is that of k factors, each at only two levels. The 2 <sup>k</sup> factorial design is particularly useful in the early stages of experimental work ,when there are likely to be many factors to be investigated .It provides the smallest number of runs with which k factors can be studied in a complete factorial design.

# The 2<sup>2</sup> design

The first design in the 2 k (figure 2.17) series is one with only two factors, A and B, each run at two levels. This design is call a 2<sup>2</sup> factorial design the factor s may be arbitrarily called low and high

	Factors		Treatment Combination
	А	В	Treatment Combination
	-	-	A low, B low
	+	I	A high , B Low
101019	-	+	A low , B high
านยา	+	+	A high ,B high

The treatment combinations in this design are shown graphically that



Thus, A refers to the effect of factor A, B refers to effect of factor B, and AB refers to the AB interaction. In the  $2^2$  design low and high levels of A and B donated by - and +.

In a two level factorial design, it may define the average effect of a factor as the change in response produced by a change in the levels of the other factor .Also, the notations (1) ,a , b , and ab now represent the total of all n replicates taken at the treatment combination ,as illustrated in Figure 3-1 . Now the effect of A at the low level of B is [a- (1)]/n and the effect of A at the high level of B is [ab – b]/n. Averaging these two quantities yields the main effect of A:

The average main effect of B is found from the effect of B at the low level of A and at the high level of A

The interaction effect AB as the average difference between the effect of A at the high level of B and the effect of A at the low level of B .Thus

$$AB = = 1/2n ([ab-b] - [a-(1)])$$

A Regression model representation of the two – factor factorial experiment could be written as

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \text{ error of experiment}$$

where , Y is the response for given levels of the <u>main effects</u>  $X_1$  and  $X_2$  and the  $X_1X_2$  term is included to account for a possible <u>interaction</u> effect between  $X_1$  and  $X_2$ . The constant  $\boldsymbol{\beta}_0$  is the response of Y when both main effects are 0.

# The 2<sup>3</sup>desig

Suppose that three factors ,A, B and C , each at two levels , are of interest .The design is call a 23 factorial design and the eight treatment combinations can now be displayed geometrically as cube , as shown as figure 2.18. Using the + and - notation to represent the low and high levels of the factor. It can write the treatment combination in standard order as (1) , a ,b , ab , c ,ac ,bc and abc .



Figure 2.18 the  $2^3$  factorial designed.

There are actually three different notations that are widely used for runs in the  $2^{k}$  design. The first is the + and - notation , often called the geometric notation .The second is the use of lowercase letter labels to identify the treatment combinations

Run	А	В	С	Labels	А	В	С
1	-	-	1	(-1)	0	0	0
2	+	1	-	а	1	0	0
3		+		٥	0	1	0
4	+	+	-	ab	1	1	0
5	-		+	С	0	0	1
6 🥌	+	-	+	ac	1	0	1
7	-/	+	+	bc	0	1	1
8	+	+ 9	+	abc	1	1	1

.The final notation uses 1 and 0 to donate high and low factor levels, respectively , instead of + and - . These different notations are illustrated below for the  $2^3$  design

There are seven degrees of freedom between the eight treatment combinations in the  $2^3$  design .Three degrees of freedom are associated with the main effects of A, B and C . Four degrees of freedom are associated with interactions; one each with AB , AC , and BC and one with ABC.

Consider estimating the main effects. First, consider estimating the main effect A. the effect of A when B and C are at low level is [a-1]/n. Similarly, the effect of A when C is at the high level and C is at low kevel is [ab-b]/n. The effect of A when C is at the high level and B is at the low level is [ac - c] / n. Finally, the effect of A when both B and C are at the high level is [abc - bc] / n. Thus, the average effect of A is adjust the average of these four, or

A = 1/4n [a-(1) + ab - b + ac - c + abc - bc]

A = 1/4n [a + ab + ac + abc - (1) - b - c - bc]

In similar manner, the effect of B is the difference in averages between the four treatment combinations in the front face of the cube and the four in the back. This yield

$$B = 1/4n [b + ab + bc + abc - (1) - a - c - ac]$$

The effect of C is the difference in averages between the four treatment combination in the top surface of cube and the four in the bottom, that is,

$$C = 1/4n [c + ac + bc + abc - (1) - a - b - ab]$$

The two – factor interaction effects may be computed easily. A measure of the AB interaction is the difference between the average A effects at the two levels Of B .By convection, one – half of this difference is called the AB interaction

В	Average A Effect
High +	1/ <mark>2</mark> n [(abc – bc) + (ab – b))
Low -	1/2n [(ac-c)+(a-(1)))
Difference	<mark>1/2n [</mark> abc – bc +ab –b –ac +c –a +(1) ]

Since the AB interaction is one - half of this difference,

$$AB = 1/4n [abc - bc + ab - b - ac + c - a + (1)]$$

Using similar logic and referring to the AC and BC interactions are

AC = 1/4n [(1) - a + b - ab - c + ac - bc + abc]

and

$$BC = 1/4n [(1) + a - b - ab - c + ac + bc + abc]$$

The ABC interaction is defined as the average difference between the AB interactions for the two different levels of C .Thus

ABC = 1/4n [abc - bc - ac + c - ab + b + a -(1)]

The regression model for predicting fill height deviation is

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3$$
  
+  $\beta_{123} X_1 X_2 X_3 + + \text{ error of experiment}$ 

Where the coded variables X1, X2 and X3 represent A, B, and C, respectively. The X1X2 term is the AB interaction .Residuals can be obtained as the difference between observed and predicted fill eight deviations



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# CHAPTER III

# LITERATURE REVIEWS

### 3.1 Optimization in spray dryer

For the past 10 years an intensive optimisation of spray drying operation has been performed.

Billon et al. (2000) investigated the effects of five parameters on production yields and moisture contents of spray-dried products. The aim of the study was to optimize the operating conditions to maximize production yields while minimizing moisture contents. First screening experiments consisting of fractional factorial designs revealed the most significant factors to be inlet temperature, feed rate and their interaction for both formulations containing sodium carboxymethylcellulose and feed rate and colloidal silica concentration for the formulation containing microcrystalline cellulose. Then, the optimal operating conditions were estimated by response surface methodology.

Kristina Sta°hl et al. (2002) investigated with the effect of process variables on the degradation and physical properties of spray dried insulin intended for inhalation. A design of experiment with2<sup>4</sup>full factorial was performed to investigate the influence of the following independent spray drying variables: feed flow rate, nozzle gas flow rate, inlet air temperature and aspirator capacity (drying gas flow rate). The solutions were spray dried in a Mini Spray Dryer Buchi and the dry powders produced were characterized by high performance liquid chromatography, size exclusion chromatography, laser diffraction, thermo gravimetric analysis, scanning electron microscopy and weighing. The degradation of insulin was found to be affected mainly by the process variables that determine the outlet air temperature, i.e.: inlet air temperature, aspirator capacity and feed flow rate. A statistical optimization of the spray drying variables was performed, and found to recommend an experiment. Athanasia et al. (2005) investigated on concentrate tomato powder in lab scale co current spray dyer. Sixty-four different experiments were conducted keeping constant the feed rate, the feed temperature and the atomizer pressure, and varying the compressed air flow rate, the flow rate of drying rate, and the air inlet temperature. The tomato powders were analyzed for moisture content, bulk density and solubility. Analysis of experimental data yielded correlations between the powder properties and the variable operating conditions. Regression analysis was used to fit mathematical models to the data of each of the powder properties evaluated. Comparisons between the moisture content, the bulk density, and the solubility of powders produced by the two drying systems proved that the use of dehumidified air, promoting rapid particulate skin formation, decreased powder moisture content and increased powder bulk density and solubility. The modified spray drying system proved advantageous over the standard laboratory spray dryer. Preliminary air dehumidification improved not only product recovery, but, also product properties.

Michael Maury et al. (2005) investigated the effects of process variables on the powder yield of amorphous trehalose obtained from the Buchi Model 191 laboratoryscale mini spray dryer. By using a specially made, narrow cyclone the powder yield could be greatly improved at all process temperatures examined. Calculations of the separation efficiencies of the improved cyclone and the manufacturer's standard cyclone are given, which show that the former's higher tangential particle velocity at the radius of the exit duct is responsible for the improved performance. The powder yield increases with higher process temperatures, owing to improved droplet drying and reduced droplet/particle deposition on the walls of the drying chamber. A maximum in the powder yield is reached, however, after which it decreases sharply. This is caused by heating of the cyclone wall toO10 8C above the so-called 'sticky point' of the trehalose, causing increased particle deposits on the walls of the tower and cyclone. Increasing liquid feed flow rate or decreasing atomizing air flow rate too extensively were both detrimental to powder yield. The drying air flow rate should be as high as possible to ensure sufficient enthalpy throughput to dry the trehalose adequately to give a high powder yield. The enthalpy balance calculation for drying trehalose with the new

cyclone was used successfully to interpret the results obtained. Some recommendations for optimizing powder yield of an amorphous material are given.

G.R. Chegini and B. Ghobadian (2007) investigated with different drying parameters. These parameters should be tested and determined before the design of the dryers. With laboratory spray dryer the basic parameters for drying of orange juice with 65% concentration was studied. The investigated parameters include: drying agent material, feed flow rate inlet and outlet air temperature and sticky point temperature. Tests were performed without and with agent materials and studied the different operating condition of spray drying and the sticky point temperature for orange juice powder. The results of statistical analysis of experimental data show that the parameters of inlet air temperature and feed flow rate have significant effect on the dryer yield and wall deposit of spray dryer individually and jointly. Also with the addition of liquid glucose, the optimum conditions have been obtained with feed flow rate of 15 ml minG1, inlet air temperature of 130°C and outlet air temperature of 85°C. For the orange powder containing 2% moisture, the sticky point temperature was 44°C.

Y. Gonnissen et al. (2007) investigated on co current spray dryer. It was evaluated if co processing via spray drying can be used as a formulation platform to improve the compactability of formulations containing drug substance (acetaminophen, ibuprofen, cimetidine) and excipients (carbohydrates, disintegrant, glidant, surfactant). Experimental design was applied to optimize the drug concentration and solid content of the feed suspension. In addition, scaling-up of acetaminophen- and ibuprofencontaining formulations was performed on a production-scale spray dryer. Optimized acetaminophen (drug concentration: 70% w/w), ibuprofen (drug concentration: 75% w/w) and cimetidine (drug concentration: 70% w/w) powders were obtained via co-spray drying of aqueous suspensions with a high solid content of the feed (35% w/w) and the resulting powders were directly compressed. Scaling-up of optimized acetaminophen and ibuprofen formulations was performed successfully, resulting in a robust and reproducible manufacturing process. It can be concluded that a combination of mannitol, erythritol, Glucidex 9, Kollidon CL, colloidal silicon dioxide and polyoxyethylene 20 sorbitan monooleate allowed the spray drying of highly dosed drug substances (acetaminophen, ibuprofen, cimetidine) in order to obtain 'ready-tocompress' powder mixtures on lab-scale and production-scale equipment.

L. Tajber et al. (2008) investigate the effect of changing spray drying parameters on the production of a budesonide/formoterol fumarate 100:6 (w/w) composite. The systems were spray dried as solutions from 95% ethanol/5% water (v/v) using a Buchi 191-Mini Spray Dryer. A 2<sup>5-1</sup> factorial design study was undertaken to assess the consequence of altering spray drying processing variables on particle characteristics. The processing parameters that were studied were inlet temperature, spray drier airflow rate, pump rate, aspirator setting and feed concentration. Each batch of the resulting powder was characterized in terms of thermal and micromeritic properties as well as an in vitro deposition by twin impinger analysis. Overall, the parameter that had the greatest influence on each response investigated was production yield – airflow (higher airflow giving greater yields), median particle size – airflow (higher airflow giving smaller particle sizes) and Carr's compressibility index - feed concentration (lower feed concentration gives a smaller Carr's indices). A six- to seven-fold difference in respirable fraction can be observed by changing the spray drying process parameters. The co-spray dried composite system which displayed best in vitro deposition characteristics, showed a 2.6-fold increase in respirable fraction in the twin impinge experiments and better dose uniformity compared with the physical mix of micronized powders.

# 3.2 The characteristic of powders in spray dryers

L. Alamilla-Beltran et al. (2003) described for morphological changes of particles along spray drying was carried out by means of scanning electron and light microscopy. Samples of atomized material (40% maltodextrin solution) are withdrawn during drying from a laboratory spray drier at various vertical distances from the atomizing nozzle, photographs of these materials are taken and images analyzed for detecting processes such as crust formation, inflation and shrinking. Final product was analyzed by SEM, mean size of particles determined and fractal dimension of projected perimeter was evaluated by means of the box counting method. This parameter of particles allowed for distinguishing between irregular and smooth contours of final powders. It was possible to explore qualitative relations of morphology and moisture content of particles with air drying temperatures along the actual drying operation, especially those related to breakage and inflation (intermediate and high drying temperatures) and with collapse (low-temperature drying).

S. X. Q. LIN and X. D. CHEN (2004) investigated on spray dryer of food liquid droplets is a primary means of manufacturing food powder today. Modeling this process requires the droplet size change during drying to be quantified and the effects of various initial or environmental conditions to be understood. Here, we report the experimental results of the effects of drying conditions on milk drop size change (projected area diameter) as a result of water removal and shrinkage. The experiments were conducted using the improved glass-filament technique, complimented by video imaging analysis software reported recently by the same authors. Under the constant drying air conditions (i.e. constant temperatures of 67–110°C and humidity of 0.0001– 0.072 kg kg<sup>-1</sup>), and in the range tested, a simple relationship between the drop/particle size and the water content has been established. This provides a good benchmark for further work on variable drying conditions.

Marcin Piatkowski and Ireneusz Zbicinski (2006) investigated the effect of drying and atomization parameters on counter-current spray drying are discussed. Based on 96 experimental tests, the local and global distributions of velocity, temperature, drying air humidity and moisture content of material dried in the drying tower were determined. Analysis of the results showed that the process of agglomeration during counter-current spray drying depended mainly on air temperature in the atomization zone.

### 3.3 The mathematic model in spray dryers

Vinh Truong et al. (2004) investigated with a steady state mathematical model for cocurrent spray drying was used to find the optimal spray drying process for sugar-rich foods. Maltodextrin-sucrose solution was used as a sugar-rich food model. The optimization of the spray drying process was carried out by finding a set of inlet variables to minimize the between the outlet air temperature and the glass transition temperature of the final products. The minimization was simplified by approaching the outlet drying air and average powder moisture content with the polynomial equations of the inlet variables in a range of interest. The drying condition at the lowest between outlet air temperature and product glass transition temperature provided the highest powder recovery at the cyclone obtained from experiment. This result shows that the stickiness of a particular sugar-rich food in spray drying can be minimized by choosing proper inlet variables using the glass transition temperature

concept.

X. Zhou; S. Chen and Z. Yu (2004) investigated with the effects of inlet air temperature, liquid feed rate and atomising air pressure on the yield, moisture content, crystal protein content and lethality of Bacillus thuringiensis powder during the spray drying of B. huringiensis fermentation liquor were studied by an orthogonal experiment. The results of variance analysis indicate that the extent of influence of factors on the yield of B. thuringiensis is in the order: atomising air pressure greater than inlet air temperature greater than liquid feed rate; and on the moisture content, crystal protein content and lethality of B. thuringiensis is in the order: inlet air temperature greater than liquid feed rate; and on the moisture content, crystal protein content and lethality of B. thuringiensis air pressure. The mathematical models describing the relationship between spray drying parameters and the yield, moisture content, crystal protein content and lethality of B. thuringiensis were established. The optimum spray drying conditions determined by mathematical models .The results of the validation experiment show that these mathematical models established by the orthogonal experiment are reliable.

### 3.4 Energy saving in spray dryer

C. G. J. Baker and K.A. McKenzie (2002) surveys in 2000, the UK Government's Energy Efficiency Best Practice Program commissioned a survey to determine the energy consumption of typical spray dryers within the chemicals, foods and ceramics industries. The results from this survey, which included dryers having evaporation rates ranging from 0.1-12 t/h, are described in this paper. Values of the specific energy consumption  $E_s$  varied widely, ranging from less than 3 to around 20 GJ/t water evaporated; the average for all dryers included in the survey was 4.87 GJ/t. As might be anticipated there was a wide scatter in the results. A preliminary analysis of the data indicated that  $E_s$  showed a small but significant decrease with evaporation rate over most of the range. A small number of dryers operating at very low throughputs (<1 t/h evaporation rates) exhibited very high values of  $E_s$ , often as much as 4-5 times the norm. Further statistical analysis is required to evaluate other possible trends.

Y. Gonnissen et al. (2007) investigated on Manufacturing of 'ready-to-compress' powder mixtures for direct compression was performed by spray drying, without granulation, milling and/or blending steps in between spray drying and compaction. Powder mixtures containing acetaminophen, mannitol, erythritol, maltodextrin, crospovidone, colloidal silicon dioxide and polyoxyethylene 20 sorbitan monooleate were prepared via co-spray drying. A feed suspension having a solid content of 27.2% w/w was selected for further process optimization because of its high process yield, excellent flowability and short tablet disintegration time. Experimental design was applied to evaluate processibility, physic chemical properties and compactability of the spray dried powder mixtures. Significant and adequate regression models were developed for powder flowability, median particle size, bulk density, residual moisture content and process yield. An increasing inlet and outlet drying air temperature improved process yield. However, a higher inlet drying air temperature had a negative influence on density and moisture content, while the latter decreased at higher outlet drying air temperatures. Median particle size increased with a higher inlet temperature, while the outlet temperature had the opposite affect. Numerical optimization determined the optimal spray drying process (inlet temperature: 221°C, outlet temperature: 81 °C and atomization pressure: 6 bar) in order to produce 'ready-to-compress' powder mixtures.



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# CHAPTHER IV

# **EXPERIMENTAL**

The experimental system and procedures used in this research are divided into three parts:

- 1. Materials and equipments.
- 2. Analysis of powder
- 3. Experiment design

### 4.1 Material and equipments

### 4.1.1 Builders in Laundry Detergent Products

Early laundry detergent products were based on the surfactant 'soap' which is itself a builder. Before these products were able to clean the laundry, they had to "soften" the water, because water contains natural elements such as magnesium and calcium ions. These ions cause the hardness of the water. The process of softening the water was evident by the formation of "scum" which appeared as these ions combined with the soap to form insoluble stearates and palmitates. The harder the water the greater the amount of soap required to reduce the water hardness and get lather. The presence of a lather indicated that the wash water solution was effectively softened and was ready to clean the laundry.

With the introduction of synthetic surfactants the ability of the cleaning agent to "soften" the water was lost. However the hardness salts in the water still reduced the surfactant performance. Other agents known as 'builders' have to be included in products to provide the optimum conditions in the wash water for synthetic surfactants and other active ingredients to do their job.

Builders are basically water softeners which soften the wash water by extracting and binding the calcium and magnesium ions. In effect the availability of these ions is removed from the wash-water solution, so the "hardness" of the wash-water is reduced. Depending on the type of builder, other properties/benefits may be available such as:

- buffering of the wash water in the alkaline range (most formulations work best in alkaline conditions).
- helping to prevent redeposition of soils when they have been removed from the fabric by the action of the surfactants.
- carrying absorbed liquid components in the products, such as surfactants in order to produce a dry, free flowing powder.

Traditionally, sodium tripolyphosphate (STPP) was the preferred builder. It provides good water softening, due to its high binding capacity for calcium ions and heavy metals, good buffering properties, which contributes to detergency, and good anti-redeposition properties. In addition, the position of STPP as a preferred builder from the 1940s-1970s was further assured due to its low toxicity (combined with good formulation and in-use characteristics). Although STPP has low toxicity, it also has the potential, as a nutrient, to contribute to eutrophication (a term used to describe water which is 'over rich' in nutrients, and which can lead to an excessive growth of algae). This led to the search for alternative builders, and the development of synthetic detergent Zeolite which is today the principle alternative to phosphate builders. These two builders are now described in more detail.

### **Phosphates Builders**

Phosphates occur throughout the natural environment. They are an essential nutrient for, and occur in all living things: plants, animals and human beings. Many foodstuffs are rich in phosphates and a daily intake is important in a balanced diet - particularly for growing children where phosphate is vital in the building of strong bones and teeth. The phosphate used as a 'builder' in detergents (normally sodium tripolyphosphate) is a compound of phosphorous, oxygen and sodium with the chemical formula Na5P3O10. It has all of the functions of a 'builder' described above and has been used in domestic laundry detergents since the late 1940s. In recent years it's use

has come under scrutiny following suspicions that its nutrient value could lead to adverse changes in water quality.

### Zeolite Builders

Synthetic detergent Zeolites are today the principle alternative to phosphate type builders, softening the washing water by calcium ion exchange. Introduced into laundry detergent products in the late 1970's usage in Europe had reached 50000 tonnes by 1984 and had increased to 600000 tonnes by 1996. Although synthetic detergent Zeolites are very effective at 'tying up' or removing calcium ions, they are much less effective in removing magnesium ions. Thus, to obtain optimal water softening, manufacturers usually include a co-builder, often a polycarboxylate, to improve the water softening capacity of synthetic detergent Zeolites.

Synthetic detergent Zeolites, like their natural counterparts, are crystalline hydrated aluminosilicates based upon an infinitely extending three dimensional network of SiO<sub>4</sub> and AlO<sub>4</sub> units and have the following empirical formula:-

Na<sub>2</sub>O.XSiO<sub>2</sub> .A1<sub>2</sub>O<sub>3</sub> .YH<sub>2</sub>O, where

X = is greater than or equal to two and

Y = is the function of porosity of the framework

Comprehensive studies have shown that, like phosphates, the synthetic crystalline Zeolites used in detergents are of low toxicity and their use poses negligible risk to man

Similarly, comprehensive studies of the environmental effects of the synthetic detergent Zeolites used in detergents have shown that as a detergent builder, synthetic detergent Zeolites pose no risk to the environment. They do not contribute to eutrophication of surface water.

In general, synthetic detergents Zeolites enter the environment *via* disposal to sewer. During the wash process there is no significant structural change to the synthetic detergent Zeolite as the pH of the system tends to be alkaline. Under these alkaline

conditions synthetic detergent Zeolites are stable. However when in neutral or acidic conditions, such as usually occur in waste-water treatment plants and natural water, synthetic detergent Zeolite undergo a process of hydrolysis. This results in the loss of the synthetic detergent Zeolites crystalline structure and metal ion exchange capacity and typically, within weeks of release into the environment, synthetic detergent Zeolite is indistinguishable from other minerals common to natural waters and soils.

In reality synthetic detergent Zeolites behave like other inorganic particulate matter in the sewage treatment plant and removal during sewage treatment is principally by sedimentation processes. In the primary settlement phase the removal of synthetic detergent Zeolite from sewage is in the region of 60~75% and where secondary treatment is employed overall removal is likely to exceed 90%.

Synthetic detergent Zeolite has a negligible effect on the volume of wet sludge produced and the synthetic detergent Zeolite content of sewage waste sludge may vary in the range of 2-18% of dry matter. However, the presence of synthetic detergent Zeolite does increase the mass of sludge for disposal and if the sludge is incinerated then the presence of synthetic detergent Zeolite will lower the calorific value of the sludge and contribute to an increase in the amount of residual ash for disposal.

### 4.1.2 Counter current spray dryer

Detergent powders were prepared as described in figure 4.1. The drying tower has 28 pressure nozzles: nozzle tip 3.2 mm and swirl 4 mm for STPP builder and 3.75 mm nozzle tip and 4.68 mm swirl for Zeolite builder. The difference distance between top ring main and bottom ring main is 3 m. The hot air occurs from the direct gas into the burner and it moved to the tower to evaporate liquid from solid. The slide gate will be separated hot air to balance the hot air in the tower and the hot air will be exited at the top of the tower.



Figure 4.1 a counter current spray dryer

In this process the first one solid and liquid material will be mixed together in a mixer when it well mixed it can call slurry after that slurry will be transferred to the tower by a high pressure pump. The pressure nozzles will be sprayed it in the opposite direction of the flow of hot air. The hot air flows upwards and the product falls through increasingly hot air into the collection tray. The residual moisture is eliminated and the product becomes very hot.

## 4.2 Analysis of powder

*The moisture content*: it was determined by the moisture sensor mm 710 an it shown in figure 4.2. The infrared technology employed is based on the principle that molecular bonds such as OH and CH absorb infrared energy. The Gauge contains a quartz halogen source lamp which projects light through an optical system to provide light in the infrared spectrum at wavelengths selected to suit the particular constituents being measured. Infrared light is radiated from the Gauge on to the product where some is reflected back to the Gauge. With a given constituent in the product, at known

wavelengths the infrared light is highly absorbed (called absorption wavelengths) while at other known wavelengths there is no absorption of infrared light (called reference wavelengths). The MM710 is designed to very accurately measure changes in reflected intensity from the product and compares intensities of the absorption wavelengths with the intensities of reference wavelengths. In many cases the ratio of these intensities will provide an output proportional to the concentration level of moisture, or other constituent in the product. Mathematical functions (algorithms) are applied by the circuitry within the Gauge to convert the computed information into the measurement of interest, for example, percentage of moisture, fat or protein.



*The powder bulk density:* 500.00 g of powder was transferred to a 392.00 cm<sup>3</sup> graduated cute and it shown in figure 4.3. The bulk density was calculated by dividing the mass of the powder by the volume occupied in the cute.





Figure 4.3 the cute for find the bulk density

# 4.3 Design Of Experiment (DOE)

The data were analyzed using the statistical software MINITAB (Release 14). Full Factorial experimental designs were used to evaluate the effects of different parameters on gas consumption and bulk density. In order to limit the number of experiments, full factorial designs  $2^3$  and 5 replicates were initially built. In this experiment, the design of experiment was performed to investigate the influence of feed flow rate, hot air flow rate and atomize pressure. The variables operating conditions were within the following ranges in table 4.1 and table 4.2 along with their respective operating limits. In all experiments the powders moisture contents were conducted keeping in 7 ±1.5% powder moisture content for STPP base powder and 4 ± 1 for Zeolite base powder.

Table 4.1 p	process pa	rameters	and oper	ating limi	ts range

Parameters	Operation limits range
Atomize pressure	50.00 – 55.00 Bar
Hot air flow rate	65000.00- 72000.00 kg/hr
Slurry feed rate	25.50 – 31.00 ton/hr

The experiment design by the MINITAB is shown in table 4.2

Table 4.2 the condition by created on the design of experiment

	Factors							
Run	Atomize pressure (bars)	Slurry feed rate (ton/hr)	Hot air flow rate (kg/hr)					
1	max	max	max					
2	min	max	max					
3	max	min	max					
4	min	min	max					
5	max	max	min					
6	min	max	min					
7	max	min	min					
8	min	min	min					



# CHAPTER V

# **RESULTS AND DISCUSSION**

### 5.1 Gas consumption

The experiment's data of STPP and Zeolite has been shown in table A1 and A2 (appendix A) .Gas utilization of two different components indicate that gas consumption is varied from 530.00 to 720.00 kg/hr. The statistic analysis of STPP is shown in figure 5.1. Three parameters have affected noticeably with the gas consumption (p value < 0.05) as:

Factorial Fit: Gas consumption versus Hot air flow, Atomized pressure and Feed rate Estimated Effects and Coefficients for Gas consumption (kg/hr) (coded units)

Terms	Effect	Coef	SE Coef	Т	Ρ		
Constant		637.370	0.9796	650.66	0.000		
Hot air flow rate (kg/hr)	-19.8 <mark>33</mark>	-9.917	1.0390	<b>-</b> 9.54	0.000		
Atomize pressure (bars)	-18.917	-9.458	1.0390	-9.10	0.000		
Feed rate (ton/hr)	119.667	59.833	1.0390	57.59	0.000		
S = 7.19839 R-Sg = 98.59% R-Sg(adj) = 98.50%							

Gas consumption is related to hot air flow rate, atomized pressure and slurry feed rate. It decreases with an increase of hot air flow rate and atomized pressure, together with the decrease of slurry feed rate. For this experiment, the results of the statistic program are shown as equation A below:

Gas consumption = 637.37 + (-9.91hot air flow) + (-9.458atomize pressure) + (59.833feed rate) ......(A)

The accuracy of the equation has shown in the R square and this equation has 98.5% R square. Increased atomization resulted in particle size decrease. Reducing particle size generates more surface area which ultimately reduces the energy required for evaporation. The flow rate of hot air controlled to a certain extent the time that the product was resident in the tower. Increasing residence time resulted in a greater degree of moisture removal as long as the exhaust air humidity conditions permit the drying process to continue.



Figure 5.1 the analysis of gas consumption for STPP

For Zeolite, the statistic analytical is shown in the figure 5.2. We will see the same effect of STPP. Three parameters have effected gas consumption (p value < 0.05) as below: Factorial Fit: Gas consumption versus Hot air flow, Atomize pressure and Slurry feed rate

Estimated Effects and Coefficients for Gas consumption (kg/hr) (coded units)

Term	Effect	Coef SE Coef	Т	Р
Constant		660.289 0.9950	663.60	0.000
Atomized pressure (bars)	-11.100	-5.550 1.0554	-5.26	0.000
Hot air flow (kg/hr)	-15.200	-7.600 1.0554	-7.20	0.000
Feed rate (ton/hr)	70.100	35.050 1.0554	33.21	0.000
S = 6.67475 R-Sq = 96.65%	R-Sa (adi) = 9	6.40%		

Gas consumption is related to hot air flow rate, atomized pressure and slurry feed rate. It decreases with an increase of hot air flow rate and atomized pressure, together with the decrease of slurry feed rate. For this experiment, the results of the statistic program are shown as equation B below:

Gas consumption = 6660.289+ (-7.6 hot air flow) + (-5.55atomize pressure) + (35.05feed rate) ......(B)



The accuracy of equation has shown in 96.4% R square.

Figure 5.2 the analysis of gas consumption for Zeolite

Generally, the ratio of gas consumption per powder (ton) and the efficiency of the spray tower indicate energy saving. The analysis of gas (kg) per powder (ton) of STPP is shown in figure 5.3. Three parameters have a dramatic effect on the gas consumption (p value < 0.05) as:

Factorial Fit: kg gas/ton powder versus Hot air flow, Atomize pressure and Slurry feed rate

Estimated Effects and Coefficients for gas (kg)/powder (ton) (coded units)

Terms	Effect Coef	SE Coef	Т Р			
Constant	32.9737	0.06185	533.11 0.000			
Hot air flow rate (kg/hr)	-1.0571 -0.5285	0.06560	-8.06 0.000			
Atomize pressure (bars)	-0.8096 -0.4048	0.06560	-6.17 0.000			
Feed rate (ton/hr)	-0.2821 -0.1410	0.06560	-2.15 0.036			
S = 0.454515 R-Sq = 68.27% R-Sq (adj) = 66.37%						

The equation *A* is directly related to the ratio of energy consumption per powder (ton). The ratio of gas (kg) consumption per powder (ton) associates with hot air flow rate, atomized pressure and slurry feed rate. It decreases with an increase of hot air flow rate and atomized pressure, and with a decrease in slurry feed rate. The results of the statistic program have been shown as an equation for gas consumption in equation C as below:

Gas(kg) / Powder(ton) = 32.9737+ (-0.5285 hot air flow) + (-0.4048atomize pressure) + (-0.141feed rate) ......(C)

This equation has 66.37% R square.



Figure 5.3 the analysis of gas(kg) / powder (ton) of STPP

The analysis of gas (kg) per powder(ton) of Zeolite is shown in figure 5.4. Like STPP, three parameters have dramatic effects on the gas consumption (p value < 0.05) as: Factorial Fit: kg gas/ton powder versus Hot air flow, Atomize pressure and Slurry feed

rate

Estimated Effects and Coefficients for kg gas/ton powder (coded units)

Terms	Effect	Coef	SE Coef	Т	Ρ	
Constant		34.695	0.04332	800.92	0.000	
Atomize pressure(bars)	-0.526	-0.263	0.04595	-5.72	0.000	
Hot air flow (kg/hr)	-0.677	-0.338	0.04595	-7.36	0.000	
Feed rate (ton/hr)	<b>-</b> 2.703	-1.351	0.04595	<b>-</b> 29.41	0.000	
S = 0.290594 R-Sq = 95.87% R-Sq(adj) = 95.57%						

The equation *B* is directly related with the ratio of energy consumption per powder (ton). The ratio of gas (kg) consumption per powder (ton) associates with hot air flow rate, atomized pressure and slurry feed rate. It decreases with an increase in hot air flow rate and atomized pressure and with a decrease in slurry feed rate. The results of the statistic program have been shown as equation D as below:

Gas(kg) / Powder(ton) = 34.695+ (-0.338 hot air flow) + (-0.263atomize pressure) + (-1.351feed rate) ......(D)

This equation has 95.57% R square.

The efficiency of the spray tower shows in the amount (kg) of energy to produce powder and the thermal efficiency. The thermal supplied to the dryer used in the evaporation process. It can be approximated to the relation efficiency defined as the fraction of total heat

 $\eta_{oevrall} = 100 (T_1 - T_2 / T_1 - T_0))$ Where  $T_1$ : Tower inlet temperature  $T_2$ : Tower outlet temperature  $T_0$ : Atmospheric temperature

The effect of thermal efficiency of STPP and Zeolite is shown in figure 5.5 and figure 5.6. It described when increased slurry feed rate of the gas (kg) per powder (ton) will be decreased.

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Figure 5.4 the analysis of gas(kg) / powder (ton)for Zeolite



Figure 5.5 the analysis of Thermal efficiency for STPP



Figure 5.6 the analysis of Thermal efficiency for Zeolite

### 5.2 Bulk Density (BD)

The results of bulk density for 2 two different components indicated that the bulk density is varied from 0.370 to 0.440 kg/Cu.cm. The statistic analytical for STPP is shown in the figure 5.7. Two parameters have affected interaction with BD (p value < 0.05) as: Factorial Fit: BD versus Hot air flow, Atomize pressure and Slurry feed rate Estimated Effects and Coefficients for BD (kg/Cu.cm) (coded units)

Terms	Effect Coef SE Coef	ТР
Constant	0.40061 0.001671	239.80 0.000
Atomize pressure(bars)	0.01225 0.00613 0.001772	3.46 0.001
Feed rate(ton/hr)	-0.04317 -0.02158 0.001772	-12.18 0.000
Atomize pressure(bars)*	-0.01642 -0.00821 0.001772	-4.63 0.000
Feed rate(ton/hr)		

S = 0.0122765 R-Sq = 78.43% R-Sq(adj) = 77.13%

Increasing slurry feed rate results in bulk density increases. The atomized pressure and the slurry feed rate have effected interaction on the bulk density which mean that the atomized pressure is significantly difference at low slurry feed rate. The bulk density increases when the atomized pressure increases .However at high slurry feed rate the atomized pressure will not be significant difference.

The results of the statistic program have been shown the equation for bulk density in equation *E*:

BD = 0.40061 + 0.00613atomize pressure – 0.02158feed rate -0.008(atomize pressure . feed rate)......(E)

In this equation has the accuracy at 78.43% R square



The hot air flow associates with the inlet temp. When hot air flow decreases, inlet temp will increase as well. However, We do not see the affection of inlet temp on bulk

density. Adem Gharsallaoui et al. investigated the affection of operation of spray dryer on encapsulate powder and found that when it has low rate evaporate it has trend to easiness of agglomeration.

The statistic analytical for Zeolite is shown in the figure 5.8 which different from STPP. The results indicate that three parameters have effected dramatically with BD (p value < 0.05) as:

Factorial Fit: BD versus Hot air flow, Atomize pressure and Slurry feed rate Estimated Effects and Coefficients for BD (kg/Cu.cm) (coded units)

Term	Effect	Coef	SE Coef	Т	Ρ				
Constant		0.410978	0.000946	434.52	2 0.000				
Atomize pressure(ba	rs) 0.019850	0.009925	0.001003	9.89	0.000				
Hot air flow (kg/hr)	0.018450	0.009225	0.001003	9.20	0.000				
Feed rate (ton/hr)	-0.016650	-0.008325	0.001003	-8.30	0.000				
S = 0.00634477									



Figure 5.8 the analysis of bulk density for Zeolite builder

The bulk density will be decreased when increased the slurry feed rate and it will be increased when increased atomize pressure and hot air flow rate. The results of the statistic program have been shown the equation for bulk density in equation *G*. The bulk density decreases if increases the slurry feed rate. However, it will increase if the atomized pressure and hot air flow rate increase. The results as the equation as below:

BD = 0.410978 + 0.009925 atomize pressure +0.009225 hot air flow rate -0.008325 feed rate ......(G)

This equation has the accuracy at 85.97% R square. Marcin Piatkowski et al. (2006) investigated the counter current spray dryer by varying the distance from the air inlet . They found that agglomeration during counter-current spray drying depended on drying air temperature in the atomization zone.

The inlet temp had effected directly with feed rate and hot air flow into tower. Inlet temp increased when increased feed rate and decrease hot air flow. Increasing feed rate along with decreasing in hot air flow rate, resulted in an increasing of Inlet temp and it is shown in figure 5.9.



Figure 5.9 the effect of hot air flow rate and feed rate on inlet temp for Zeolite builder

Esther H.-J. Kim et.al (2008) studied the effects of inlet temp at counter current flow spray dryer on milk powder. They found that higher drying rates, which leaded to rapid crust formation around the drying particles, appeared to result in less fat migration to the surface during the early stage of drying.



### CHAPTHER VI

### CONCLUSIONS AND RECOMMENDATION

### 6.1Conclusions

In this work the Design of Experiment (DOE) has been used to optimize a counter current spray dryer. Sodium tripolyphosphate (STPP) and Zeolite were used as component materials to manufacture detergent powder in order to analyze their bulk density and energy consumption. The ratio between energy consumption and powder (ton) indicates energy saving from manufacturing. The lower ratio, the smaller energy consumption. By both components we will see the same as effect of process parameter on this ratio. The results of the minimizing conditions shown that a counter current should be operate with high feed rate(31.00 ton/hr), high atomize pressure(55.00 bars) and high hot air flow rate (72,000.00 kg/hr). The results of both components showed that bulk density increases with a corresponding decrease in slurry feed rate and an increase in atomized pressure with an increase in hot air flow rate (only see this effect in Zeolite). For maximize yield a counter current should be operate as 31.00 ton/hr feed rate, 55.00 bars atomize pressure and 72,000.00 kg/hr high hot air flow rate. For minimize the energy consumption and maximize bulk density (acceptable the BD with 0.38 kg/Cu.cm up), the results of DOE shown that a counter current should be operated with 29.95 ton/hr feed rate, 55.00 bars atomize pressure and 72,000.00 kg/hr hot air flow rate and in this condition will be given the bulk density approximately 0.3883 kg/Cu.cm and 31.95 of the ratio between energy consumption and powder (ton) for STPP component. It is shown in figure 6.1. For Zeolite component, A counter current should be operated as 28.25 ton/hr feed rate, 52.50 bars atomize pressure and 68,500.00 kg/hr hot air flow rate and in this condition will be given the bulk density approximately 0.411 kg/Cu.cm and 34.69 of the ratio between energy consumption and powder (ton). It is shown in figure 6.2.







Figure 6.2 the optimization for Zeolite builder

In this experiment we will implement the conditions as the DOE recommended on Zeolite component in real production and compare it with current conditions. When we change from current conditions as 26.00 ton/hr slurry feed rate, 50.00 bars atomize pressure and 67,000.00 kg/hr hot air to new recommend conditions as 31.00 ton/hr slurry feed rate, 55.00 bars atomize pressure and 72,000.00 kg/hr hot air, the energy consumption and bulk density are significantly difference. It is shown in figure 6.3 and 6.4. The statistic analytical for energy consumption shown as:

Two-sample T for Current conditions vs Recommend conditions

	Ν	Mean	StDev	SE Mean					
Current conditions	5	29.616	0.347	0.16					
Recommend conditions	5	26.798	0.177	0.079					
Difference = mu (Current conditions) - mu (Recommend conditions)									
Estimate for difference: 2.81800									
95% CI for difference: (2.41653, 3.21947)									
T-Test of difference = 0 (vs not =): T-Value = 16.19 P-Value = 0.000 DF = 8									
Both use Pooled StDev = 0.2753									

The result of the ratio between energy consumption and powder (ton) shown that recommend conditions is lower than current conditions approximately 1.17 – 1.83 with 95% confidence interval

The statistic analytical for Bulk density shown as:

### Two-Sample T-Test and CI: Current conditions, Recommend conditions

Two-sample T for Current conditions vs Recommend conditions

	Ν	Mean	StDev	SE Mean					
Current conditions	5	0.32480	0.00415	0.0019					
Recommend conditions	5	0.34060	0.00195	0.00087					
Difference = mu (Current conditions) - mu (Recommend conditions)									
Estimate for difference: -0.015800									
95% CI for difference: (-0.020526, -0.011074)									
T-Test of difference = 0 (vs not =): T-Value = -7.71 P-Value = 0.000 DF = 8									
Both use Pooled StDev = 0.0032									

The result of the ratio between energy consumption and powder (ton) shown that bulk density of recommend conditions is higher than current conditions approximately 0.01 - 0.02 with 95% confidence interval. We expect to save conversion cost 2 million baht a year by implementing this study.



 Figure 6.3 the comparison of new recommend conditions and current conditions in

 Zeolite builder on kg gas / ton powder



Figure 6.4 the comparison of new recommend conditions and current conditions in Zeolite builder on bulk density

### 6.2 Recommendations for future studies

In this experiment can be used to the guideline of counter current spray dryers operation to minimize energy consumption and maximize yield. However the effect of each process parameter can be integrate with the Model Predictive Control to operate the counter current with the high efficiency in the future.



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APPENDIX

### DATA OF EXPERIMENTS

### TableA.1the experimental data of STPP builder

			Slurry			10-				Bulk
	Hot air	Atomize	Clarry	Inlet	Outlet	Gas	Powder	kg	Thermal	Dant
No.	flow	pressure	feed	temp	temp	consumption	moisture	gas/ton	efficiency	density
	(top/br)	(horo)	rate	(°C)	(°C)	(kg/br)	(0/)	bourder	0/	(kg/cu.cm
	(lon/nr)	(bars)	(ton/hr)	(())	( ()	(kg/nr)	(%)	powder	70	)
1	65.00	55.00	31.00	463.10	103.10	725.00	8.29	33.76	83.12	0.385
2	65.00	55.00	31.00	462.50	103.20	715.00	7.06	33.99	83.08	0.385
3	65.00	55.00	31.00	462.10	103.50	717.00	7.09	34.02	82.99	0.372
4	65.00	55.00	31.00	462.60	103.00	715.00	7.40	33.81	83.13	0.354
5	65.00	55.00	31.00	463.00	102.90	717.00	7.24	33.43	83.16	0.372
6	65.00	5 <mark>5</mark> .00	31.00	463.00	103.00	707.00	6.61	33.17	83.14	0.374
7	72.00	55.00	31.00	395.40	100.00	671.00	7.36	31.56	80.84	0.395
8	72.00	55. <mark>0</mark> 0	31.00	396.30	100.00	674.00	7.45	31.86	80.89	0.395
9	72.00	55.00	3 <mark>1.0</mark> 0	395.50	100.00	679.00	7.35	32.13	80.85	0.387
10	72.00	55.00	<mark>31</mark> .00	393.70	100.00	67 <mark>6.0</mark> 0	8.34	31.46	80.75	0.359
11	72.00	55.00	31.00	394.50	100.00	671.00	7.74	31.29	80.80	0.372
12	72.00	55.00	31.00	395.30	100.00	679.00	7.73	31.67	80.84	0.372
13	72.00	50.00	31.00	409.10	98.40	717.00	7.49	33.48	81.96	0.374
14	72.00	50.00	31.00	408.60	98.40	717.00	7.25	33.80	81.93	0.377
15	72.00	50.00	31.00	409.80	99.20	715.00	7.15	33.51	81.78	0.382
16	72.00	50.00	31.00	410.60	99.60	717.00	6.90	33.90	81.71	0.357
17	72.00	50.00	31.00	409.80	99.30	715.00	7.35	33.43	81.75	0.364
18	72.00	50.00	31.00	408.70	99.40	717.00	6.55	34.02	81.67	0.387
19	65.00	50.00	31.00	461.90	103.60	720.00	6.89	34.24	82.96	0.382
20	65.00	50.00	31.00	462.90	104.20	715.00	6.93	34.12	82.86	0.390
21	65.00	50.00	31.00	462.50	104.90	717.00	6.50	34.29	82.68	0.387
22	65.00	50.00	31.00	461.00	105.20	705.00	6.96	33.65	82.55	0.405
23	65.00	50.00	31.00	460.60	105.20	705.00	6.15	33.79	82.54	0.382
24	65.00	50.00	31.00	460.50	105.10	705.00	6.67	33.60	82.56	0.385
25	72.00	50.00	25.50	338.60	98.30	540.00	6.76	30.80	77.87	0.379
26	72.00	50.00	25.50	339.70	97.40	537.00	7.39	30.75	78.24	0.392
27	72.00	50.00	25.50	339.60	96.80	547.00	7.98	30.85	78.42	0.364

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	Hot air	Atomize	Slurry	Inlet	Outlet	Gas	Powder	kg	Thermal	Bulk
No.	flow	pressure	feed	temp	temp	consumption	moisture	gas/ton	efficiency	density
	(ton/hr)	(bars)	rate	(°C)	(°C)	(kg/hr)	(%)	powder	%	(kg/cu.cm
28	72.00	50.00	25.50	339.40	96.10	547.00	8.46	30.85	78.64	0.395
29	72.00	50.00	25. <mark>50</mark>	338.20	96.00	555.00	8.48	31.13	78.59	0.39
30	72.00	50.00	25.50	343.20	95.80	556.00	8.15	31.24	78.99	0.387
31	65.00	50.00	25.50	399.80	97.60	580.00	6.34	33.25	81.72	0.377
32	65.00	50.00	25.50	397.50	98.30	582.00	6.31	33.35	81.41	0.410
33	65.00	50.00	25.50	394.90	97.30	575.00	6.82	32.61	81.56	0.402
34	65.00	50.00	25.50	395.00	98.60	575.00	6.68	32.82	81.21	0.425
35	65.00	50.00	25.50	396.60	98.70	572.00	6.76	32.69	81.26	0.407
36	65.00	50.00	25.50	<mark>394</mark> .80	98.80	572.00	6.69	32.60	81.14	0.425
37	65.00	55.00	25.50	392.40	100.80	566.00	7.87	32.18	80.46	0.447
38	65.00	55.00	25.50	392.70	101.20	566.00	6.80	32.56	80.37	0.454
39	65.00	55.00	2 <mark>5.</mark> 50	393.10	101.60	564.00	7.22	32.29	80.28	0.440
40	65.00	55.00	<mark>25.50</mark>	392.90	101.80	566.00	7.24	32.69	80.21	0.414
41	65.00	55.00	25 <mark>.5</mark> 0	393.00	101.00	569.00	7.52	32.65	80.44	0.420
42	65.00	55.00	2 <mark>5</mark> .50	393.90	102.00	56 <mark>9.</mark> 00	7.32	32.79	80.21	0.442
43	72.00	55.00	<mark>2</mark> 5.50	334.50	100.60	540.00	7.96	31.34	76.81	0.443
44	72.00	55.00	25.50	335.00	99.00	540.00	7.77	31.12	77.38	0.430
45	72.00	55.00	25.50	335.80	98.60	540.00	8.11	31.35	77.57	0.445
46	72.00	55.00	25.50	335.80	97.80	540.00	8.22	31.47	77.83	0.437
47	72.00	55.00	25.50	339.40	97.40	550.00	7.66	32.27	78.22	0.420
48	72.00	55.00	25.50	339.00	97.20	553.00	8.24	31.95	78.25	0.443
49	68.50	52.50	28.25	392.80	99.00	629.00	6.90	33.46	80.98	0.415
50	68.50	52.50	28.25	393.50	99.10	626.00	6.45	33.46	80.99	0.412
51	68.50	52.50	28.25	393.00	99.10	629.00	6.84	33.48	80.96	0.410
52	68.50	52.50	28.25	392.80	99.10	629.00	6.70	33.59	80.95	0.420
53	68.50	52.50	28.25	391.70	99.10	623.00	6.51	33.50	80.90	0.417
54	68.50	52.50	28.25	391.80	99.10	621.00	6.50	33.24	80.90	0.407
<u> </u>	- QI									

	Atomize	Hot air	Feed	Gas	kg	Bulk	Inlet	Outlet	Thermal
No	pressure	flow	rate	consumption	gas/ton	density	temp	temp	efficiency
	(bars)	(ton/hr)	(ton/hr)	(kg/hr)	powder	(kg/Cu.Cm	(°C)	(°C)	%
1	55.00	64.00	30.50	<mark>69</mark> 7.00	33.26	0.39	427.00	112.10	73.75
2	55.00	64.00	30.50	707.00	3 <mark>3.8</mark> 0	0.40	419.00	112.40	73.17
3	55.00	64.0 <mark>0</mark>	30.50	697.00	33.10	0.40	419.70	111.80	73.36
4	55.00	64.00	30.50	684.00	33.08	0.41	422.00	112.10	73.44
5	55.00	64.00	30.50	715.00	34.19	0.40	420.00	112.90	73.12
6	55.00	7 <b>1</b> .00	<mark>30</mark> .50	679.00	32.62	<mark>0.4</mark> 1	384.00	110.30	71.28
7	55.00	71.00	30.50	671.00	32.21	0.43	375.00	110.30	70.59
8	55.00	71.00	30.50	673.00	32.59	<mark>0.4</mark> 3	377.00	110.70	70.64
9	55.00	71.00	3 <mark>0</mark> .50	684.00	32.89	0.41	380.00	110.90	70.82
10	55.00	71 <mark>.0</mark> 0	30.50	681.00	32.67	0.42	378.00	110.20	70.85
11	50.00	71.00	30 <mark>.5</mark> 0	692.00	33.19	0.41	396.00	111.90	71.74
12	50.00	71.0 <mark>0</mark>	<mark>30</mark> .50	687.00	<mark>32</mark> .95	0.40	393.00	112.80	71.30
13	50.00	71.00	<b>3</b> 0.50	694.00	33.16	0.40	394.00	113.50	71.19
14	50.00	71.00	30.50	698.00	33.44	0.41	396.00	113.80	71.26
15	50.00	71.00	30.50	694.00	33.41	0.40	395.00	114.00	71.14
16	50.00	64.00	30.50	700.00	33.59	0.38	<mark>432.</mark> 00	115.40	73.29
17	50.00	64.00	30.50	710.00	33.95	0.38	429.00	116.00	72.96
18	50.00	64.00	30.50	712.00	34.09	0.39	431.80	116.40	73.04
19	50.00	6 <mark>4.0</mark> 0	30.50	715.00	34.23	0.39	431.30	116.60	72.97
20	50.00	64.00	30.50	717.00	34.32	0.39	431.60	117.00	72.89
21	50.00	64.00	25.20	635.00	36.39	0.40	379.60	116.40	69.34
22	50.00	64.00	25.20	632.00	36.29	0.40	379.80	116.70	69.27
23	50.00	64.00	25.20	625.00	36.71	0.39	381.90	116.40	69.52
24	50.00	64.00	25.20	635.00	36.28	0.40	380.00	116.40	69.37
25	50.00	64.00	25.20	635.00	36.27	0.41	380.00	116.40	69.37
26	50.00	71.00	25.20	632.00	36.05	0.41	344.60	115.00	66.63
27	50.00	71.00	25.20	626.00	36.29	0.41	345.50	114.90	66.74

	Atomize	Hot air	Feed	Gas	kg	Bulk	Inlet	Outlet	Thermal
No	pressure	flow	rate	consumption	gas/ton	density	temp	temp	efficiency
	(bars)	(ton/hr)	(ton/hr)	(kg/hr)	powder	(kg/Cu.Cm	(°C)	(°C)	%
28	50.00	71.00	25. <mark>20</mark>	621.00	35.86	0.413	344.70	114.70	66.72
29	50.00	71.00	25.20	630.00	36.26	0.409	343.20	115.00	66.49
30	50.00	71.00	25.20	627.00	36.29	0.423	344.00	114.70	66.66
31	55.00	71.00	25.20	602.00	<mark>34.92</mark>	0.44	335.00	115.20	65.61
32	55.00	71.00	25.20	609.00	35.09	0.448	336.80	114.80	65.91
33	55.00	71.00	2 <mark>5.2</mark> 0	618.00	35.78	0.446	<mark>3</mark> 35.20	114.60	65.81
34	55.00	71.00	25.20	615.00	35.43	0.438	334.80	114.10	65.92
35	55.00	71.00	25.20	621.00	35.90	0.445	336.00	113.90	66.10
36	55.00	64 <mark>.00</mark>	<mark>25</mark> .20	623.00	36 <mark>.</mark> 12	0.42	366.40	113.50	69.02
37	55.00	<mark>64</mark> .00	25 <mark>.2</mark> 0	632.00	36.33	0.425	363.40	113.90	68.66
38	55.00	64.0 <mark>0</mark>	<mark>25</mark> .20	628.00	36.07	0.41	366.00	114.20	68.80
39	55.00	6 <mark>4</mark> .00	25. <mark>2</mark> 0	630.00	36.22	0.43	365.00	114.30	68.68
40	55.00	64.00	2 <mark>5.2</mark> 0	629.00	36.24	0.417	366.00	114.40	68.74
41	52.50	67.50	<mark>27</mark> .85	663.00	34.75	0.412	385.00	113.00	70.65
42	52.50	67.50	27.85	664.00	34.78	0.42	386.00	113.00	70.73
43	52.50	67.50	27.85	659.00	34.70	0.418	386.50	112.50	70.89
44	52.50	67.50	27.85	661.00	34.80	0.41	386.70	112.40	70.93
45	52.50	67.50	27.85	654.00	34.72	0.415	386.70	112.00	71.04

### VITA

Mr. Tongkarn Keawtasi was born on May 19<sup>th</sup>, 1981 in Mahasarakham, Thailand. He received the Bachelor Degree of Chemistry from Faculty of Science, Khon Khaen University in 2003. He has work in NSD Department, Uniliver Thai Holdings Ltd. He continued study the Master of Chemical Engineering at Chulalongkorn University in June, 2006.

