UNIVERSITY OF INDONESIA

ANALYZING THE DEGREE OF MIXING BETWEEN INCLINE AND STRAIGHT WALL FLUIDIZED BED THROUGH IMAGE ANALYSIS

THESIS

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ENGINEERING FACULTY UNIVERSITY OF INDONESIA
BACHELOR PROGRAMME
DEPOK
JULY 2010

UNIVERSITY OF INDONESIA

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TITLE PAGE

THESIS

Proposed as one of the requirement to obtain the title

Sarjana Teknik Kimia

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STATEMENT OF ORIGINALITY

This thesis is a result of my own work,

and all the sources that has been quoted or cited

has been correctly stated.

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PREFACE

First of all I would like to say thanks to the god almighty Allah SWT due to his help this thesis could be finished on time**.** The thesis with title **Analyzing the Degree of Mixing Between Incline and Straight Wall Fluidized Bed Through Image Analysis** was made to fulfill part of the academic requirement in order to achieve the Sarjana Teknik degree in Chemical Engineering Department FTUI.

During the preparation of this thesis the writer would like to acknowledge all the help that has been given to the writer during the preparation of this thesis. Therefore the writer would like to send the deepest gratitude to:

1. Mom, dad and my sister thank you for all the support and help you have given me all this time this one is for you.
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The writer realize that this thesis is far from perfect and therefore would kindly accept any critics or suggestion to improve the writing for the near future.

Depok, July 2010

Aditya Satrio Prabowo

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ABSTRACT

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Fluidized bed is used for many purposes in the industry such as for reactor, drying or mixing. In the pharmaceutical manufacture, drying in fluidized bed is an essential manufacturing step because during the drying process the resulting moisture content should be uniform.

It was often founded that batch of the pharmaceutical products has a wide range of moisture content which therefore leads to a termination of the whole batch of product. To avoid different moisture content a better mixing inside the fluidized bed was then required. The effect of bed height, particle placement and geometry of fluidized bed was then analyzed.

In this experiment a mixing of two different particle size are mixed inside an adjustable wall fluidized bed. With one of the particles are colored so the mixing could be seen visually from the sides of the perplex glass. The mixing was then observed at two different flow rates which is the bubbling and twice the bubbling flow rate. The variable that will be manipulated will be the bed height, particle size and wall angle

It was then founded the higher the bed height the better the mixing will be this was shown in the result of increasing of mixing area for the 2 cm 13,8% , the 4 cm 38% and the 10 cm is 66,7%. The effect of particle placement shows that when the larger particle are placed on the bottom the mixing will increase particle placement when it is place on the bottom the mixing area is 44,2% and when it's place on the top the mixing area is just 29,1%. The effect of geometry wall was analyzed the result shows that the incline wall created better mixing for the 4 cm the mixing area is 44,2 % for straight wall and 58,2% for the angle wall although future work is still needed to strongly support the result due to possible equipment error.

keyword : Fluidized bed, Mixing, Segregation

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CHAPTER 1
INTRODUCTION

1.1. Background

Fluidized beds are commonly used in the pharmaceutical industry as a means of drying particulate material. Many solid dosage form pharmaceutical are created via wet granulation and then dried in a batch fluidized bed dryer before going to the tablet formation process. Fluidized bed drying has its own advantages because it has highly efficient thermal conditions due to the rapid mixing formed by fluidization. Fluidization is giving effective distribution of heat throughout the bed and achieving good temperature control. Another important property of the fluidization is its excellent mass transfer rates which is an important part in the drying process this is caused by the extensive mixing formed due to the fluidization. (Michael Wormsbecker, 2005)

In pharmaceutical manufacture, drying in fluidized bed is an essential manufacturing step. When the granules are feed into the dryer, it has high moisture content and has tendency to form agglomerates. At this early stage the granule will exhibit undesirable fluidization phenomena such as segregation, channelling and defluidization. This would often leads to termination of the full batch which will cost heavily. The main cause of the problem was believed to be the poor mixing effect by the fluidized bed. It was then required to investigate which type of the fluidized bed will generate a better mixing.

There are basically two types of fluidized bed that is present. They are the cylindrical and conical fluidized bed. However there is little work done on the investigation of mixing comparison between the two types of fluidized bed. Based on the explanation above it is important to know what parameter will improve the mixing inside the fluidized bed. Moreover in this paper multiple variables inside the fluidized bed from the wall geometry to bed height will be analyzed to get the best possible mixing.

1.2. Problem Statement

The problem statement in this research will be to examine

- How will the wall angle effect the mixing inside the fluidized bed?
- How does particle placement will have effect on degree of mixing?
- Does the mixing factor will be affected by bed height?

1.3. Aim of Research

The aim of the proposed research is:

- To see the effect of wall angle on mixing inside the fluidized bed through image analysis
- To get the understanding of particle placement inside the fluidized bed by using image analysis
- To see the effect of bed height on mixing through image analysis

1.4. Scope of Work

In this research project there will be limitation of work. The scope of work will only be covering the area:

- Ballotini glass bead is used as the main particle
- The fluidization media will be using dry air
- Image analysis will be based on the image taken from the side of the fluidized bed
- Since the image analysis is based on the side image it is being assumed that the same image is happening throughout the whole bed.

1.5. Report Format

The report format of this research report will be as follow:

Chapter I: Introduction

Basic background of problem, problem statement, aim of research, scope of work and report format

Chapter II: Literature review

Chapter II will be consisting of basic concept of fluidized bed theory. With a summary of previous work that has been done on the fluidized bed that is correlated with this research.

Chapter III: Research Method

In this chapter the explanation regarding how the experiment was conducted will be discussed thoroughly from the beginning of the experiment until the end of the research which also lists the equipment and materials being used.

Chapter IV: Results and Discussion

This chapter contains the result that is obtained from the experiment based on Chapter III. The corresponding results will then be discussed and analyze inside this chapter.

Chapter V: Conclusion and Suggestion

The conclusion and main findings from the whole research project is written in this chapter. This chapter also contains suggestion that is regarded as necessary to improve future findings.

CHAPTER 2
LITERATURE REVIEW

2.1 Basic Theory of Fluidization

The mechanism of fluidization is when a fluid is passed upwards through a bed of particles the pressure loss in the fluid due to frictional resistance increases with increasing fluid flow. It will eventually reach a point when the upward drag force exerted by the fluid on the particles is equal to the apparent weight of particles in the bed. At this point the particles are lifted by the fluid, the particles are separated from each other, and the bed becomes fluidized. A uniform fluidization which is the most desirable regime of operation of industrial fluidized beds is prone to instabilities. When the fluid flow increases, bubbles of clear fluid are formed at the bottom of the bed and these bubbles travel to the surface. (Rhodes, 2008) At a critical value where upward drag force is counterbalance with gravitational force the pressure loss will be constant. This is where the following equation is applicable

$$\Delta p = \frac{HA(1-\varepsilon)(\rho_p - \rho_f)g}{A} \qquad (1)$$

And simplified the equation become

$$\Delta p = H(1-\varepsilon)(\rho_p - \rho_f)g \qquad (2)$$

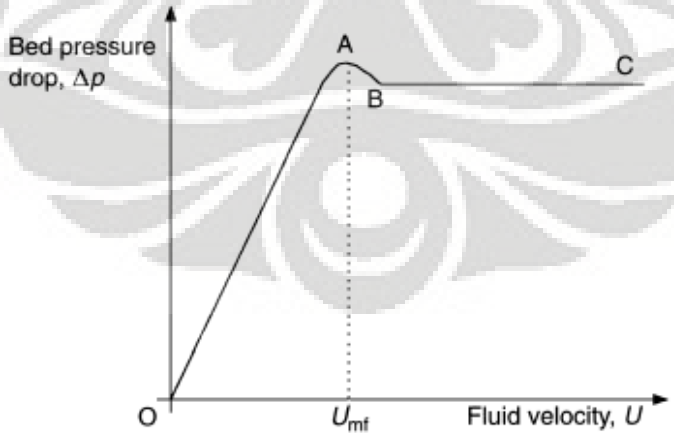


Figure 2.1. Pressure drop versus fluid velocity for packed and fluidized beds (Rhodes, Introduction to Particle Technology, 2008)

A plot of fluid pressure loss across the bed versus superficial fluid velocity through the bed is shown above. Point O is where packed bed of particle is initially fluidized by upwards fluid. As the flow rate of fluid is increased the pressure drop between will also increases. Point A is point where the bed is started to fluidized. Equation (1) is applicable at BC region. There is a slight different between point A and BC region, this rise is more noticeable in small vessels and in powders which have been compacted to some extent before the test. This point A is associated with extra force required to overcome wall friction, cohesive force between particles, and adhesive forces between bed and the distributor (Rhodes, 2008).

A critical velocity where the bed started to fluidized is known as minimum fluidization velocity, U_{mf}. U_{mf} varies for each particles and different fluid properties. This U_{mf} can be calculate using Ergun equation

$$\frac{-\Delta p}{H} = 150\frac{(1-\varepsilon)^2}{\varepsilon^3} + 1.75\frac{(1-\varepsilon)}{\varepsilon^3}\frac{\rho_f U^2}{x_{sv}} \qquad (3)$$

Substitute (2) into (3)

$$Ar = 150\frac{(1-\varepsilon)}{\varepsilon^3}Re_{mf} + 1.75\frac{1}{\varepsilon^3}Re_{mf}^2 \qquad (4)$$

Where Ar is the dimensionless Archimedes number

$$Ar = \frac{\rho_f(\rho_p-\rho_f)g x_{sv}^3}{\mu^2} \qquad (5)$$

And Re_{mf} is the Reynolds number at the incipient fluidization

$$Re_{mf} = \frac{U_{mf}x_{sv}\rho_f}{\mu} \qquad (6)$$

Wen and Yu (1966) also produced an empirical correlation for Umf with a form

$$Re_{mf} = 33{,}7\,[(1 + 3{,}59 \times 10^{-5}Ar)^{0{,}5} - 1] \qquad (7)$$

This equation is only valid between spheres in the range of $0.01 < Re_{m\,f} < 1000$. For gas fluidization the Wen and Yu (1966) correlation is suitable for particles larger than 100 microns whereas the formula for particles less than 100 microns is created by Geldart and

Bayens.

$$U_{mf} = 33{,}7\ \frac{(\rho_p - \rho_f)^{0{,}934} g^{0{,}934} x_p^{1{,}8}}{1110 \mu^{0{,}87} \rho_f^{0{,}066}} \qquad (8)$$

Geldart produces a number of powder classification according to their fluidization properties. The powder classification is divided into four groups. Group A is the group of powders when fluidized gives a non-bubbling fluidization at the beginning at Umf which followed by the bubbling fluidization as the air velocity increases. While the group B powders goes straight into the bubbling fluidization state when an air flow is introduced. The other two groups are group C and D. Group C is incapable of fluidization, mainly the typical example solids for this groups are the very fine and cohesive powder. The last group is group D this group of powder is producing spouted bed when fluidized an example of the particle for this group is rice. The cause of its spouting when fluidized is due to its large particle size. Below it's the complete table Geldart classification of powder. (Rhodes, Introduction to Particle Technology, 2008)

Table 2.1. Geldart Classification powder

	Group C	Group A	Group B	Group D
Most obvious characteristic	Cohesive, difficult to fluidize	Ideal for fluidization. Exhibits range of non-bubbling fluidization	Starts bubbling at U_{mf}	Coarse solids
Typical solids	Flour, cement	Cracking catalyst	Building sand	Gravel, coffee beans
Property				
Bed expansion	Low because of chanelling	High	Moderate	Low
De-aeration rate	Initially fast, then exponential	Slow, linear	Fast	Fast
Bubble properties	No bubbles–only channels	Bubbles split and coalesce. Maximum bubble size	No limit to size	No limit to size
Solids mixing	Very low	High	Moderate	Low
Gas backmixing	Very low	High	Moderate	Low
Spouting	No	No	Only in shallow beds	Yes, even in deep beds

Source: Introduction to particle technology, Martin Rhodes

2.2 Earlier Work Done On Mixing Investigation of Fluidized Bed

Shannon was one of the earliest who investigate about segregation by size difference. He proved the most important variables affecting mixing/segregation are the minimum fluidization velocities of the solids and the superficial gas velocity. In a equal density systems, segregation is most discrete at superficial velocities between components (Shannon, 1959).

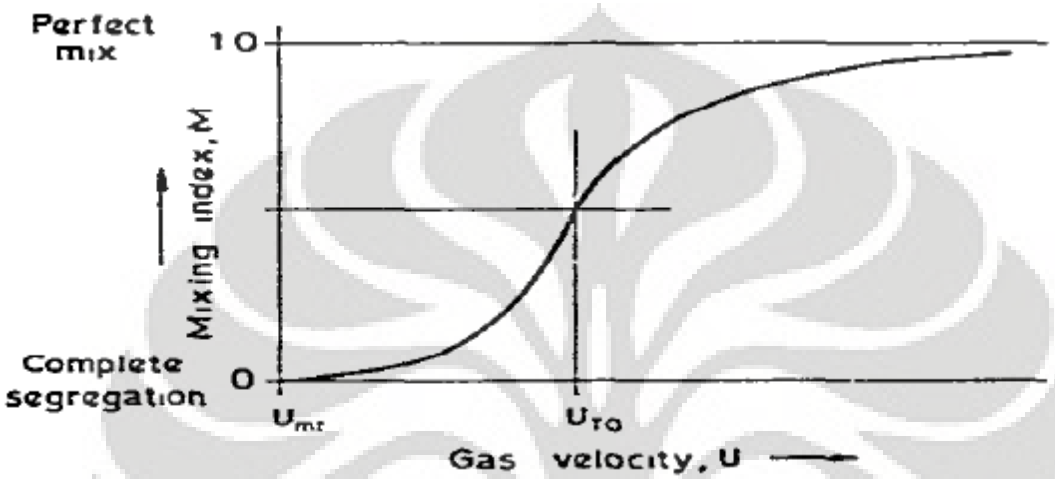


Figure 2.2. Variation of mixing index with velocity (Rowe & Nienow, 1976)

Based on experimental done by Rowe and Nienow it is clear that degree of mixing varies with gas velocity in the manner shown by *figure 2.1* It started with fully segregated system, mixing increases only slowly with increasing velocity, the mixing degree increase to maximum and then decreases again as the mixing degree near perfection (Rowe & Nienow, 1976).

This can be describe using following equation

$$M = \frac{1}{(1+e^{-Z})} \qquad (9)$$

Where

$$Z = \frac{(U-U_{TO})}{(U-U_f)} e^{U/U_{TO}} \qquad (10)$$

2.3 Previous Work Done on Comparison of Fluidized Bed

In the production of many solid-dose pharmaceutical products, drying of granule is a required manufacturing step. When wet, this granule tends to be cohesive in nature. At this early stage the granule will exhibit undesirable fluidization phenomena such as segregation, channelling and defluidization. (Wormsbecker, 2008) To overcome this problem researchers have proposed several techniques including mechanical stirring, vibration even with gas pulsation. However, the pharmaceutical industry does not use any of these techniques to help them overcome the instability during drying. Rather, the industry tries to solve this problem by changing the bed geometry design such as conical shaped bed in figure 2.4.

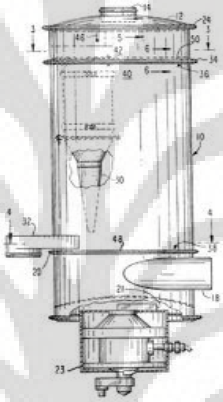


Figure 2.3. Cylindrical fluidized bed

An improvement on fluidized bed design is being invented to improve the mixing and reducing any defluidization phenomena that is happening on the previous design. A conically shaped fluidized bed design was one of the designs that are being invented. Because according to the theories the diameter of the chamber increases as it goes upwards. Therefore when the air or gas travels upwards in the chamber the air velocity will decreases. When gravity overcomes the upward forces the particles will fall back. (Sensing, 2005)

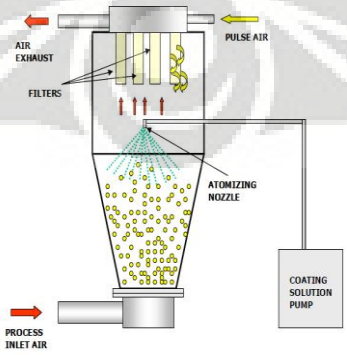


Figure 2.4. Conical fluidized bed

The hydrodynamic behaviour associated with conical fluidized beds is a cross between conventional fluidization and spouted bed behaviour. Conical fluidized beds experience gross

circulation of particles much like a spouted bed due to the geometry, but the intimate gas-solids contacting associated with conventional fluidization is still maintained. This distinct circulation pattern is a defining characteristic of conical fluidized beds which has been quantified by many researchers such as Schaafsma et al. used positron emission particle tracking (PEPT) to quantify this circulation pattern in a shallow bed of Geldart B powders. They found that the particles rose quickly through the fluidized core region of the bed and back downward through the slower moving annular region. (Wormsbecker, 2008)

Because there are two design of fluidized bed it is still not known which of these has a better hydrodynamics. Michael Wormsbecker was the first to study the effect of the conical geometry versus that of a cylindrical bed on fluidized bed drying hydrodynamics behaviour. The previous work that was done was concentrating on the impact of vessel geometry on the hydrodynamic behaviour during the drying of placebo pharmaceutical granule. The experiment was studying the hydrodynamics resulting from cylindrical and conical laboratory scale fluidized beds that will be compared using pressure fluctuation analysis. Wormsbecker found out that even though both of the conical and cylindrical fluidized bed has the same drying times the cylindrical fluidized bed develops undesirable fluidization phenomena such as channelling, defluidization and segregation when the granule is wet. These undesirable fluidization phenomena couldn't be witnessed in the conical fluidized bed. Worsmbecker explanation for this was that the tapered walls of the conical fluidized bed is creating superior particle circulation at the bed inlet while maintaining it's bubbling hydrodynamics in the core region of the bed. Wormsbecker sketch a figure that explained the flow inside the fluidized bed with different wall angle that is shown in figure 2.5 (Wormsbecker, 2008)

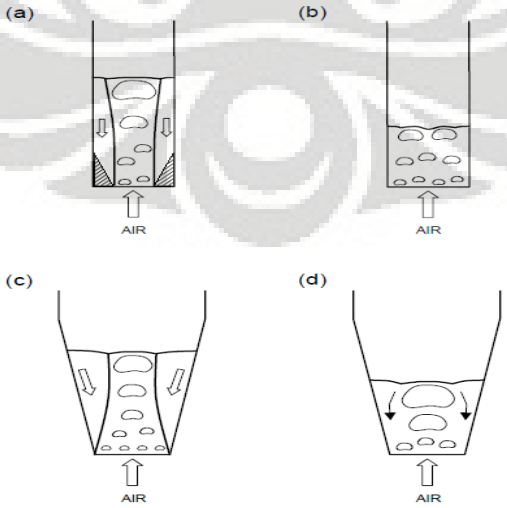


Figure 2.5. Michael Wormsbecker illustration of the flow inside each type of fluidized bed (a, b:flow inside straight wall;c,d: flow inside incline wall)

2.4 Methods for Measuring Segregation

Previous work of Michael wormsbecker et al shows that particle size distribution can be measured with the Mastersizer S-Series Long Bench particle size analyzer (Malvern, Worcestershire, UK). The sampling method of Michael Wormsbecker et al as followed:

A novel core sampling technique was employed to measured degree of segregation for a given experimental condition. Initially the bed was fluidised to ensure the different particle sizes are well mixed, and then the speed was lowered. At this point segregation was occurred. Followed by instantly shut off the gas flow valve. After the fluidizing gas had been shut off, core samples were extracted from the bed using five core sampling probes. The samples were taken from five different radial positions in the bed using the template shown in fig 1.

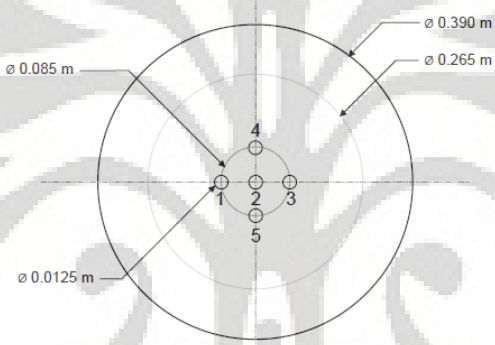


Figure 2.6. Top-view of core sampling template showing the positions of the radial sampling ports (1,3, 4, 5) and the centre sampling port (2)(Wormsbecker et al,2005).

This template ensures that the sample will be taken from the same locations within the bed. This design is based on minimum mass of particles that led to accurate and reproducible particle size distribution measurement with the Mastersizer particle size analyser. To collect the core samples each probe was connected to vacuum and successively plunged into the bed through the ports in the template. The sample probe is designed to hold 1.8 g of granule in a 4 cm segment of a core sample, the chosen length increment for analysis in this study. This design will allow measurement of particle size distribution axially and radials. Suction pressure was employed to reduce the friction between the probe wall and the particle. This aided the movement of probe when it was inserted into the bed. The rate of the probe was plunged is very important. Rapidly plunging the probe into the bed is proved to be very

efficient method. Over-pressurised the probe would cause the granule inside the probe to fluidize whilst under pressurized would not allow sample to be drawn. Optimal suction pressure was achieved by empirical trials.

A simple experiment was carried out to demonstrate the accuracy of core sampling technique. A glass beaker filled with dyed granule combined with normal white granule to create a layered packed bed in a glass beaker. This bed then sampled using the probe. Visual observation then made between the layered granule inside the probe and inside the glass beaker.it is shown in the fig 2 that the sample inside the probe closely resembled to the layered granule inside the glass beaker. Demonstration of accuracy of core sampling was conducted and it is proven to be an appropriate technique to be employed to this experiment.

Figure 2.7. Photograph showing a layered packed bed of pharmaceutical granulate in the beaker on the left and, on the right, a sample from that bed collected with the core sampling probe (Wormsbecker et al,2005).

2.5 The Effect of Bed Height on Mixing

Tanfara (Tanfara et al, 2002) did a research of the effect of particle size distribution (PSD) on local voidage has been in a conical fluidized bed containing dried placebo pharmaceutical granule.For each of the five PSDs examined, the static bed height was varied between 0.12 and 0.17m and the superficial gas velocity was varied between 0.05 and 0.75 m/s. The local voidage was sensitive to small changes in static bed height. Although the main purposes of the research wasn't to see the effect of bed height but it was founded that the gas tended to spread more uniformly over the bed crosssection as static bed height increased. The gas flow became more centralized with increasing bed height.

CHAPTER 3
EXPERIMENTAL WORK

In this chapter the diagram of the experimental stage, experimental material and apparatus, research variable, procedure and method for data analysis to obtain the results from the experiment will be thoroughly discussed.

3.1 Diagram of Research Plan

The diagram of the research plan is shown in figure 3.1. The experiment was first conducted by doing some literature review on the past work that is already done on the fluidized bed. The next step was calibrating the flow meter by using an already calibrated gas flow meter. Because the fluidized bed was just constructed shortly before the research a run out of the fluidized bed was performed to see if it's working appropriately. Due to the error design of the fluidized bed when constructed the fluidized bed was modified so it can be suitably use for research application. The experiment was basically divided into two parts. The first part is determining the minimum fluidization velocity (Umf) of each particle. This is done because the minimum fluidization velocity of each particle would then be required for the second part of the experiment.

Moreover the second part of the experiment is the binary particle mixing. In this part of the experiment the particle are mixed at two different velocities that is bubbling flow rate and twice the bubbling flow rate. The manipulated variable in this experiments are the wall angle, particle placement and bed height. The mixing was run on two different flow rate. After each experiment sieving was conducted to retrieve the particles back again due to the limitation of particles available. The resulting image from the completed experiment was then analyze using computer software. Next step will be the results comparison and discussion.

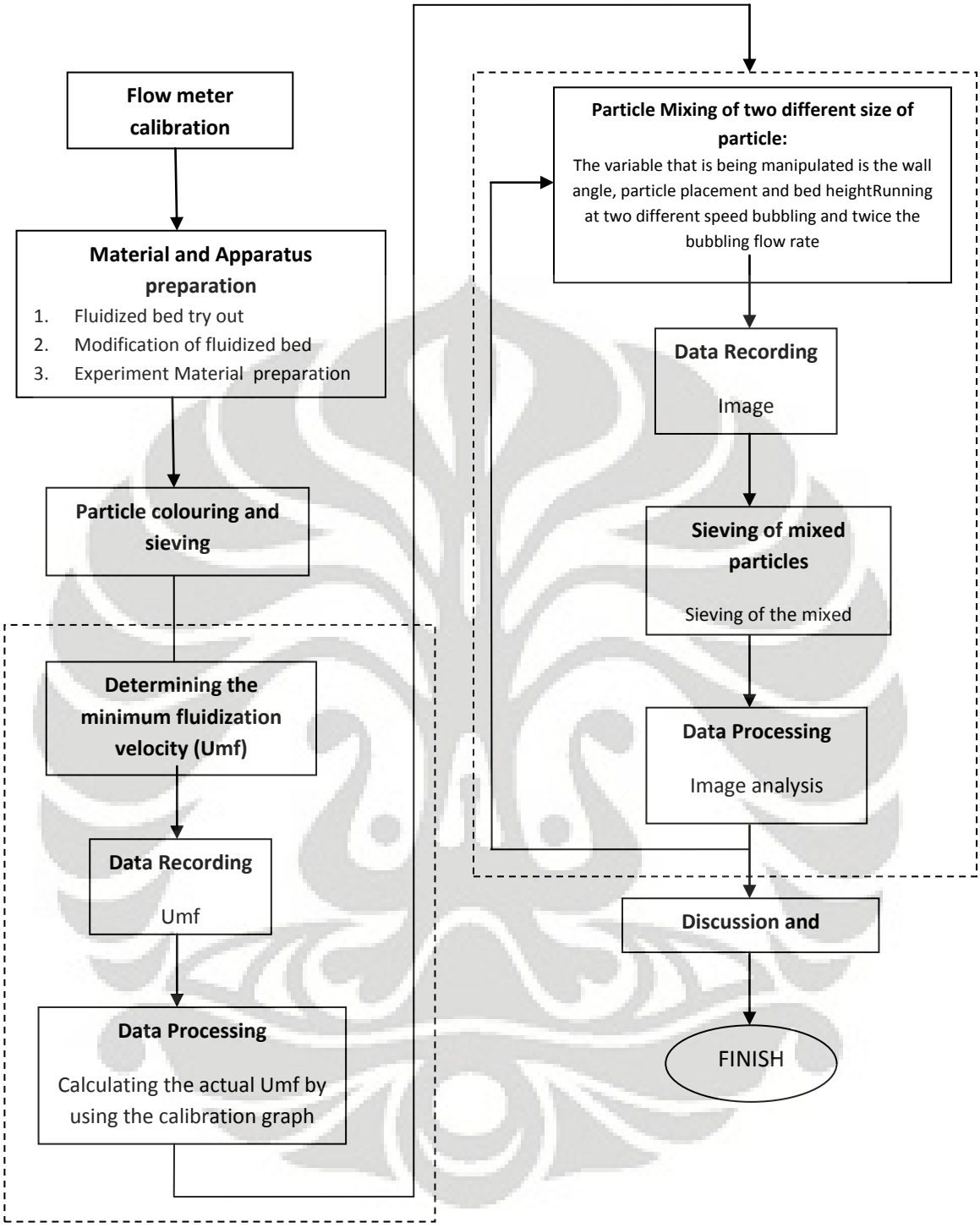


Figure 3.1 Research Diagram Flow

3.2 Material and Apparatus

3.2.1 Experimental Apparatus

The main apparatus for this experiment would be the fluidized bed which is assembly with other parts to run the experiment. The lists of the main equipments are:

1. Adjustable wall Fluidized bed
2. Small and large scale Air Flow meter
3. Plastic pipe
4. Air flow compressor

Moreover there are also a few additional apparatus that has different purposes that is being used to aid the experiment which consists of:

1. Spray paint which was used in this experiment to colour the glass beads
2. Pestle and mortar that are being used to grind the particles
3. Scale is used for weight measurement purposes
4. Siever is used to separate the mixed particles
5. Glassware equipments such as beaker and bowl

The equipments that are listed below are used for data gathering for this research project, which are:

1. Digital Camera (Olympus µ1040)
2. Camcorder

3.2.2 Experimental Material

The basic materials that are being used in this research are:

1. Sands that was used for the fluidized bed try out
2. Ballotini glass beads with a particle size range of 106-212 microns and 250-425 microns for the mixing simulation

3.3 Experimental Variable

The variable that could be determined from this experiment are:

- Wall angle
- Bed height
- Particle placement

3.4 Experimental Method

The experimental method was conducted in a few stages which are the fluidized bed modification steps, the sieving of the particle to breaks down the ballotini glass beads into a few particles range, colouring of particles, determination of minimum fluidization velocity of each particles range, and the mixing of different particles ranges on different wall angle, bed height and particle placement and lastly the recording of the image results.

3.4.1 Fluidized Bed Modification

This apparatus was modified to match the purpose of investigating fluidized bed in different wall inclination. The modifications made are

1. Distributor

Along with adjustment of particles being used in the fluidized bed the grid needs to be modified as the particle become smaller and the plate would not give uniform bubbling along the distributor. This would cause slugging and spouting. The only available plate that can be used is copper sintered plate and it has smaller orifice. Trials were done on both plate with water as the particle and sintered plate shows a better uniform bubbling along the plate.

2. Fluidized bed wall sealing

An improvement on the wall sealing was also done during the research. The reason behind sealing modification is caused by leaking that occurs along the wall and around the distributor when fine particle was poured into the bed. Leak tightness become more critical as the size and density of the powder decrease, since a small leak has a much greater proportional effect for powder of low U_{mf} (Geldart, 1986). Sealing rubber was stripped from the wall and around the distributor and changed it with sealing rubber which exhibit elastic property.

3. Replacement of flow meter

Trials done on the large gas flow meter where ballotini glass bead with size range 40-90 micron was placed in the bed. The valve was gradually opened and the particle bed started to bubble however the flow meter did not show changes. Then the large flow meter was replaced with 2 different size flow meters to increase the accuracy of the flow meter since finer particle has much smaller U_{mf} and at low flow rate large flow meter does not demonstrate significant increment or static measurement.

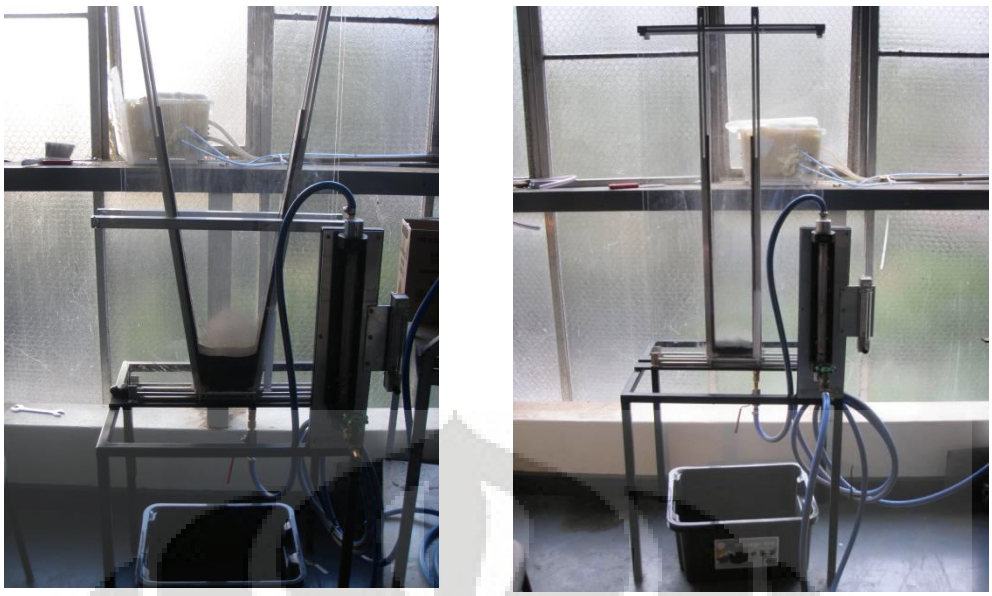


Figure 3.2. Actual images of the Fluidized Bed apparatus (left: incline wall, right: straight wall)

3.4.2 Flow Meter Calibration

The procedure for the flow meter calibration is as follow:

1. Connecting the plastic pipe from the air connection to the flow meter and the pipe connected to the fluidized bed is connected to an already calibrated gas flow meter.
2. Setting the desired flow values on the flow meter
3. Read the values on the calibrated gas flow meter
4. The data are then taken for 5 different values in the flow meter and a calibration graph was then created which could be seen in the appendix

3.4.3. Sieving Analysis

There were only two particle ranges in stock for ballotini glass beads, which are 106-212 micron and 250 -425 micron. Because the particle size range of the 250-425 micron is too wide it was then decided to reduce the particle size range into two ranges. the 255-425 microns was separated between the range of 250 -355 and 355 to 425 microns. The procedure is as follows.

1. Assembly the siever tray by putting the largest tray on top and the smallest tray on the bottom.
2. Pour some amount of particles into the top tray
3. Close the lid and turn on the machine with a timer
4. After the timer has stop the machine will turns off automatically. Open the lid and dissembled the sieving tray and pour the remaining particle on each tray to its own range.

5. The procedure was then repeated for a couple of times to obtain the amount of particles desired

3.4.4. Particles Colouring

Some of the particles are coloured so the mixing that occurs inside the fluidized bed could be seen visually in this case the ballotini glass beads in the range of 106-212 microns is being coloured. The steps that need to be taken are:

1. Particle is poured into aluminium tray
2. The particles are then coloured by spray paint while stirring so the colour would spread evenly
3. After leaving the paint to dry for a few hours it is being grinded by using pestle and mortar because the paint tends to agglomerate the particle
4. The last step is sieving the particles to standardize the particle range

3.4.5. Determining the Minimum Fluidization Velocity (Umf)

There are three ranges of particle size. In order to run out the actual experiment the minimum fluidization of each velocity needs to be measured. The following steps show how the minimum fluidization velocity is measured:

1. Particle is placed inside the bed.
2. Gradually opening the valve slowly until the first bubble is spotted.
3. At this stage the reading on the flow meter is written down because this is the Umf of the particles.
4. This is then done for the other two particles on incline and straight wall and with two different bed heights of 5 and 12 centimetres.

3.4.6. Mixing Observation Experiment

The coloured particle which has particle size range between 106-212 microns is being mixed with the particle size 355-425 microns and 250-355 microns one at a time. The procedure is as follow.

1. The wall of the fluidized bed is set to be straight or angle wall
2. Than the desired bed height was determine to measure the quantity of the particles needed to make that bed height. The ratio of the particle is 50:50 of the weight mass

3. The placement of particle was then determined whether the bigger particle will be placed on the top or the bottom. After it is being determined the particles that are going to be placed at the bottom is poured slowly into the fluidized bed to make a layer. After that the particle that are going to be put on the top is then being pour slowly on top of the existing layer of particles
4. The valve was then opened slowly until it reach the bubbling point. After it reach the bubbling point it is being run out for five minutes before shutting the valve abruptly. At this point the image on the side of the bed was taken
5. The valve is then opened again and it is put to twice the bubbling flow rate and being let to run for another five minutes before shutting the valve abruptly. The image on the side of the bed was also taken
6. After the experiment has finished the mixed particles from inside of the bed is drained and then taken to the siever to separate between the particles so it can be used for another experiment
7. The experiment was then re-conducted again by changing the variable of wall angle, particle placement and bed height of 2, 4 and 10 cm

3.4.7. Data Gathering

The procedure of the data gathering during the experiment:

1. Closing the valve abruptly after each experiment
2. Taking a photograph from the side of the bed using digital camera

3.4.8. Data Processing

Because the data that was obtained from the experiment was in the form of image a further processing using computer software called Adobe Photoshop 9.0™ was used to determine the mixing and non mixing area of each images.

The procedure is as follows

1. Opening any of the image file by using adobe photoshop
2. Selecting the wanted area for example the mixing area with the lasso tool

3. Go to the image menu and then choose the option histogram. The total pixel recorded in that area is then calculated
4. The other part of the image was then measured as well
5. Then the fraction of each mixing and non mixing area was calculated by using the formula below

$$\text{Mixing area Percentages} = \frac{mixing\ area}{(mixing + non\ mixing\ area)} * 100\% \qquad (1)$$

$$\text{Non- mixing area percentages} = \frac{non-mixing\ area}{(mixing + non\ mixing\ area)} * 100\% \qquad (2)$$

CHAPTER 4
RESULTS AND DISCUSSION

In this chapter the results from the experiment is being discussed thoroughly. Before further discussion on the result there are a few assumptions that are being made prior to the calculations of result in the image analysis. As it is being mentioned in the experimental method the result of this experiment is based on the image analysis taken from the sides of the fluidized bed. It is being considered that the image seen on the sides is a representative of the mixing inside the middle of the bed of particles. So then an image analysis could be performed.

4.1 Minimum Fluidization Velocity (U_{mf}) of Each Particle

The investigation of minimum fluidization velocity for each particle range of 106 -212, 250-355 and 355-425 microns were carried out. The data that was taken was at two different bed heights of 5 cm and 12 cm. The resulting results for each particle range are tabulated below. As it can be seen from the results the minimum fluidization velocity for an incline wall is higher compared to a straight wall fluidized bed. This is probably caused by the geometry of the incline wall fluidized bed that whereas the diameter of the chamber increases as it goes upwards therefore creating a decrease in velocity. That is why the incline wall fluidized would required higher flow rate to fluidize compared to a straight wall fluidized bed.

Table 4.1. Minimum fluidization velocity of each particle range

		106-212 microns		*250-355 microns*		*355-425 microns*	
Bed Height	Wall angle	Bubbling ($1 \times 10^{-4} m^3/s$)	Slugging ($1 \times 10^{-4} m^3/s$)	Bubbling ($1 \times 10^{-4} m^3/s$)	Slugging ($1 \times 10^{-4} m^3/s$)	Bubbling ($1 \times 10^{-4} m^3/s$)	Slugging ($1 \times 10^{-4} m^3/s$)
5 cm	Straight	0.68	4.15	3.90	17,9	4.73	20,4
	Incline	0.73	4.15	4.17	17,9	5.02	21,0
12 cm	Straight	1.07	5.33	7.00	12,5	8.13	17,9
	Incline	1.07	4.44	6.65	12,5	8.52	18,5

4.2 Image Results Analysis

When the experiment was carried out it appears that the particles won't get fully mix even at a higher flow rate. Stagnant material appears on the bottom of the bed and on the sides of the bed as with an example of the result shown in figure 4.1 Therefore from all the data gathered the mixing and non mixing area was first determined and then calculated based on the fraction of the area.

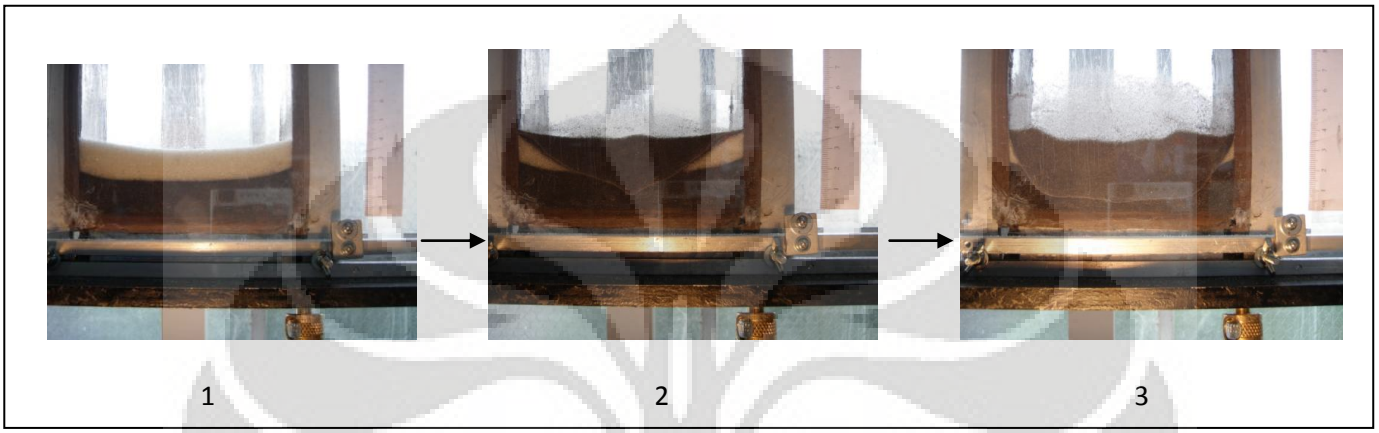


Figure 4.1. *Mixing of 106 -212 and 355 – 435 microns at a bed height of 4 cm with the coarser particle placed on the top*

Notes:

1. The image of the particles before fluidizes; 2. The result after the particles are fluidized at bubbling flow rate; 3.The result after the particles are put at a flow rate of twice the bubbling flow rate

After processing all the image results gathered from the experiment the mixing and non mixing area could be calculated. The corresponding result for each mixing of 106 212 microns with 250-355 microns and 106 – 212 microns with 355-425 microns was then created into a list of matrix results. *Table 4.2* is the complete results for binary mixture of 106-212 microns and 250-355 microns. From the corresponding table there a few gaps. These gaps were caused by complexity of sieving and limited time provided. Because the particle range of 106-212 and 250-355 is quite close it was hard to separate both of the particles through sieving as it has been disclose in experimental section.

Table 4.2. *Matrix result of 106-212 microns and 250-355 microns*

Bed Height (Cm)		2		4		10	
Bottom	Straight		mass fraction		mass fraction		mass fraction
		mixing	27.22%	mixing	46.5%	mixing	62.01%
		non mixing	72.78%	non mixing	53.5%	non mixing	37.99%
		mixing	60.00%	mixing	73.60%	mixing	75.05%
		non mixing	40.00%	non mixing	26.40%	non mixing	24.95%
	Angle	mixing		mixing	51.10%	mixing	63.93%
		non mixing		non mixing	48.90%	non mixing	36.07%
		mixing		mixing	72.80%	mixing	89.24%
		non mixing		non mixing	27.20%	non mixing	10.76%
Top	Straight	mixing	34.87%	mixing	41.18%	mixing	
		non mixing	65.13%	non mixing	58.82%	non mixing	
		mixing	65.03%	mixing	73.86%	mixing	
		non mixing	34.97%	non mixing	26.14%	non mixing	
	Angle	mixing	26,10%	mixing	41.68%	mixing	
		non mixing	73,90%	non mixing	58.32%	non mixing	
		mixing	60.00%	mixing	72.82%	mixing	
		non mixing	40.00%	non mixing	27.18%	non mixing	

bubbling flowrate
twice bubbling flowrate

Table 4.2 is indicating the mass fraction of mixing and non mixing area. The bottom and top refers to the placement of the coarser particle. While the area that is shaded is showing the mixing and non-mixing area in twice bubbling flow rates or slugging flow rates and the area that is unshaded is the mixing and non mixing area on bubbling flow rate.

Table 4.3. *Matrix result of 106-212 microns and 355-425 microns*

Bed Height (Cm)		2		4		10	
Bottom	Straight		mass fraction		mass fraction		mass fraction
		mixing	28.3%	mixing	44.2%	Mixing	75.0%
		non mixing	71.7%	non mixing	55.8%	non mixing	25.0%
		mixing	54.4%	mixing	51.1%	Mixing	79.6%
		non mixing	45.6%	non mixing	48.9%	non mixing	20.4%
	Angle	mixing	22.1%	mixing	58.2%	Mixing	78.2%
		non mixing	64.9%	non mixing	41.8%	non mixing	21.8%
		mixing	51.1%	mixing	70.3%	Mixing	84.2%
		non mixing	48.9%	non mixing	29.7%	non mixing	15.8%
Top	Straight	mixing	16.8%	mixing	29.1%	Mixing	66.7%
		non mixing	83.2%	non mixing	70.9%	non mixing	33.3%
		mixing	42.3%	mixing	68.9%	Mixing	85.6%
		non mixing	57.7%	non mixing	31.1%	non mixing	14.4%
	Angle	mixing	14.3%	mixing	33.2%	Mixing	67.8%
		non mixing	85.7%	non mixing	66.7%	non mixing	32.2%
		mixing	49.5%	mixing	63.3%	Mixing	85.1%
		non mixing	50.5%	non mixing	36.7%	non mixing	14.9%

bubbling flowrate
twice bubbling flowrate

Table 4.3 is the result of mixing binary particles consisting of 106-212 microns and 355-425 microns. There are no gaps in this table because the experiment for the binary mixtures of 106-212 microns and 355-425 microns could be completed. The same outcome from data *table 4.3* is the same with *table 4.2*. An increase in flow rate is also followed by an increase in area of mixing. For example the data when 355-425 microns is placed on the top with straight angle side shows an increase of mixing area from 13.8% to 42.3% at the 2 cm bed height. The same could also be seen in the other data.

4.3 Effect of Particle Placement

One of the main purposes of this research is to see whether the arrangement of particle would affect the mixing inside the fluidized bed. To fully analyse the understanding of the

effect of particle placement the image result will be used as well as the result from the image processing to get in depths understanding on the phenomena.

(a) *(b)*

Figure 4.2. The comparison of particle arrangement between straight wall fluidized bed at 4 cm with binary particles of 106 – 212 microns and 355-425 microns at bubbling flow rate (a: coarse particle placed on the bottom, b: coarse particle placed on the top)

Figure 4.2 Shows the comparison of mixing inside a straight wall fluidized bed with different particle placement. The reason why it is being analyze at bubbling flow rate is because the difference of mixing area at twice the bubbling flow rates doesn't have significant amount of differences. The colored particle is the particle with a particle size range of 106 -212 microns whilst the white colored or the uncolored particle is the 355-425 microns. From the image results it could be seen that when the coarser or larger particle is placed on the bottom it shows a better mixing compared to when the coarser particles are placed on the top.

(a) (b)

Figure 4.3. The comparison of particle arrangement between incline wall fluidized bed at 4 cm with binary particles of 106 – 212 microns and 355-425 microns at bubbling flow rate (a: coarse particle placed on the bottom, b: coarse particle placed on the top)

Figure 4.3 is the comparison of mixing inside an incline wall fluidized bed with different particle placement. The same results are obtained in the incline wall fluidized bed. The mixing area when the coarse particle is placed on the bottom is larger than when the particles are placed at the top. From the image results processing the resulting mixing area of the incline wall with the larger particles placed on the bottom was found to be 58,2 % while the mixing area when the larger particles was placed on the top was found to be 27,8%. The explanation for this is maybe caused by the voidages of the larger material. When the larger particle is placed on the bottom the smaller particles will fall easily to fill the voidages between the larger particle with not only help from the fluidization but also from gravity. But when the small particle is placed on the bottom it becomes dense and the voidages are smaller compare to the larger particles so it's harder for the larger particles to enter the voidages between the smaller particles.

4.4 Bed Height

The bed height was investigated whether or not it has an impact on the mixing inside the fluidized bed. There are three different bed heights that were performed during the experiment. The differences of the mixing area was then analyse between the three different bed heights.

a) 2 cm b) 4 cm c) 10 cm

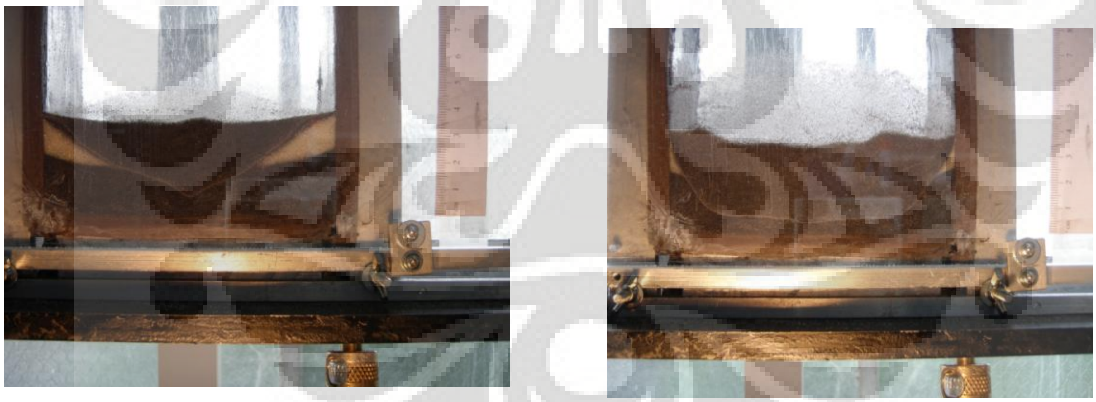
Figure 4.4. Mixing pattern at different bed height

From the figure above the increase of bed height is also being followed with the increase of mixing area. The mixing area at 2 cm for example was smaller compared to the mixing area in 4 cm and so on. Quantitatively speaking the mixing area for 2 cm is 13,8 % while the mixing area for 4 cm is 38% and the mixing area for the 10 cm is 66,7 %. The same results also happen with different setting of the fluidized bed for example wall angle and particle placement. The reason behind this was explained by Tanfara et al the higher the bed height

the more centralized the flow of the gas in addition creating spout like fluidization which ultimately gives better mixing. The stagnant wall also contributes largely to the big differences of mixing area. As it been earlier discussed some stagnant material appears during the experiment on the bottom of the bed. At a higher bed height the stagnant material stopped accumulating at a particular height. Therefore the non mixing area becomes smaller as the bed height increases.

4.5 Effect of Flow Rate on Mixing

The mixing experiment was performed at two different flow rates. From the results obtained in table 4.2 when the particle is on slugging flow rate or twice the bubbling flow rate the mass fraction area of mixing is bigger compared when it's on bubbling flow rate. For example the data on the straight and the coarser particle placed on the top shows that when it's on a bed height of 2 cm the bubbling flow rate only has a mixing area of 22.22%. While when the flow is increased into twice the bubbling flow rate the area of mixing increases from 22.22% into 60%. An image example of the result is also shown in figure 4.5. This phenomenon could be explained with common sense the higher the flow rate the higher will be the force exerted from the air flow into the particles in addition the particles will be stir more vigorously.



a) bubbling flow rate b) twice bubbling flow rate

Figure 4.5. Mixing pattern at different flow rate 4 cm bed height with straight wall fluidized bed with coarser particle placed on top

4.6 Particle Size

There are two binary systems in this research. The first binary system is the mixing of 106-212 microns and 250-355 microns and the second binary mixing is the 106-212 microns and 355-425 microns. At 2 cm bed height the 250 μm particles shows better mixing than

355µm particle range. As the bed height increases the 355µm particle range has better mixing than 250µm particle range. Proposed hypothesis for this result is at higher bed the mixture between 106-212 µm and 255-355 µm (mixture A) has lower fluidization velocity than between 106-212 µm and 355-425 µm (mixture B). Therefore the finer particle which is 106-212 µm, is more reluctant to segregate at mixture B this can cause false judgment that the mixture has better mixing.

4.7 Influence of Wall Geometry

Examining the influence of bed geometry over degree of mixing was the main purpose of this research project. After gathering a set of results a comparison of the straight and angle wall fluidized bed was made. First the mixture of 355 – 425 and 106 -212 µm will be examined. As it shown in figure 4.6 when the comparison is made on the bed height of 2 cm the straight wall fluidized bed shows a better mixing compared to the angle wall fluidized bed at both particle placement case. Moving to the 4 cm bed height results when the coarser particle are placed on the top angle wall has superior mixing compared to the straight wall the result when the coarser particle are placed on the top is the same as well but not too far-off. The same could be said for the 10 cm bed height.

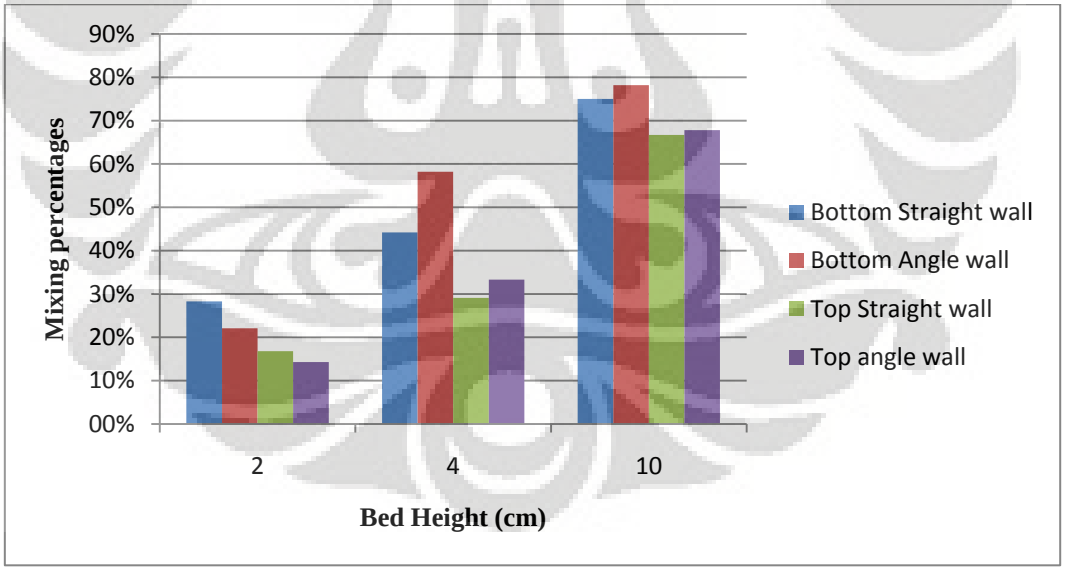


Figure 4.6. Mixing percentages of 355 - 425 µm and 106 -212 µm at bubbling flow rate

When the particles are mixed at twice the bubbling flow rates the results doesn't show any consistency. The results from the bed height of 2 cm illustrate that when the particle is placed on the top the angle wall shows a better mixing but when compared to the 4 and 10 cm bed height the mixing of straight wall fluidized bed compared to a angle wall fluidized bed is

slightly better when the larger particles are placed on the top but when the larger particle are placed on the bottom the angle wall shows a larger mixing area.

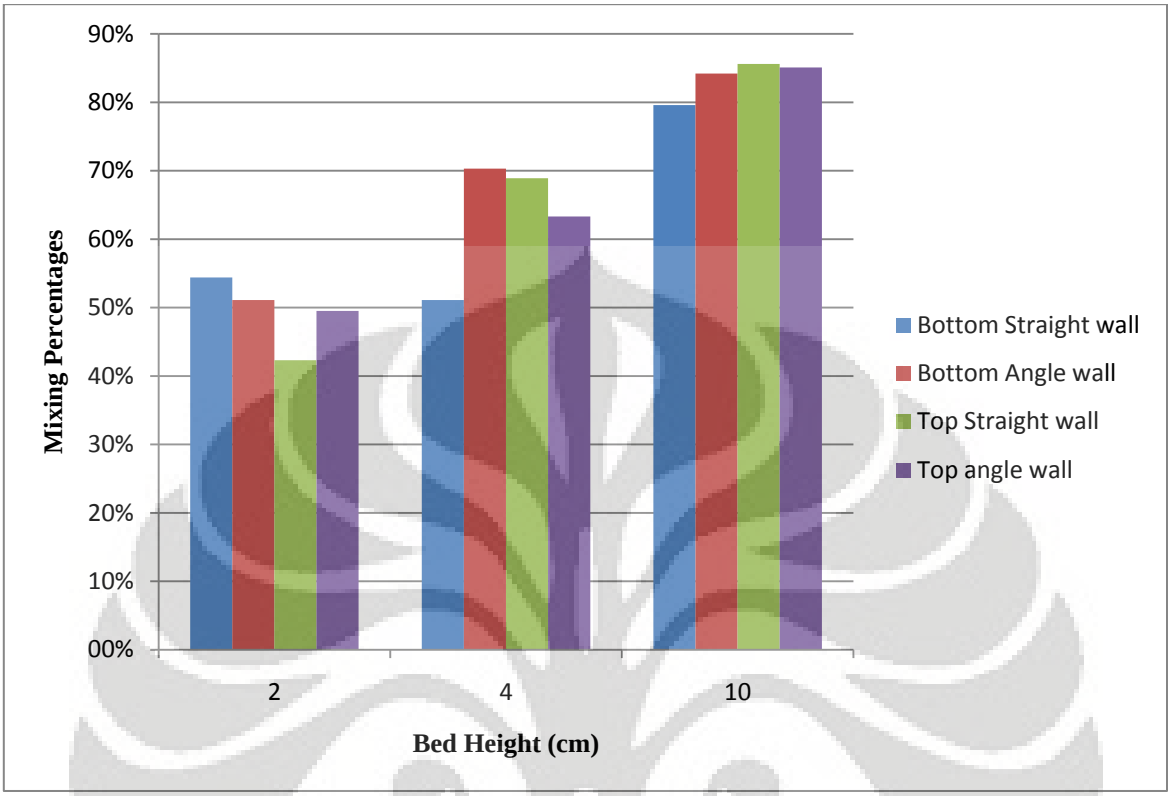


Figure 4.7. Mixing percentages of 355 - 425 µm and 106 -212 µm at twice bubbling flow rate

Further discussing the results on figure 4.8 because of limited results the discussion will be mainly on one point of results that is when the coarse particle is placed on the bottom. The result on the 10 cm bed height satisfies the current theory that is stated (Wormsbecker, 2008). The mixing degree of angle wall bed is greater than straight wall. Looking further on to the result on the 4 cm bed height the same could be said but when the coarser particle are placed on the top the difference of mixing area between angle and straight wall fluidized bed are not too significant although it shows that the angle wall is slightly higher.

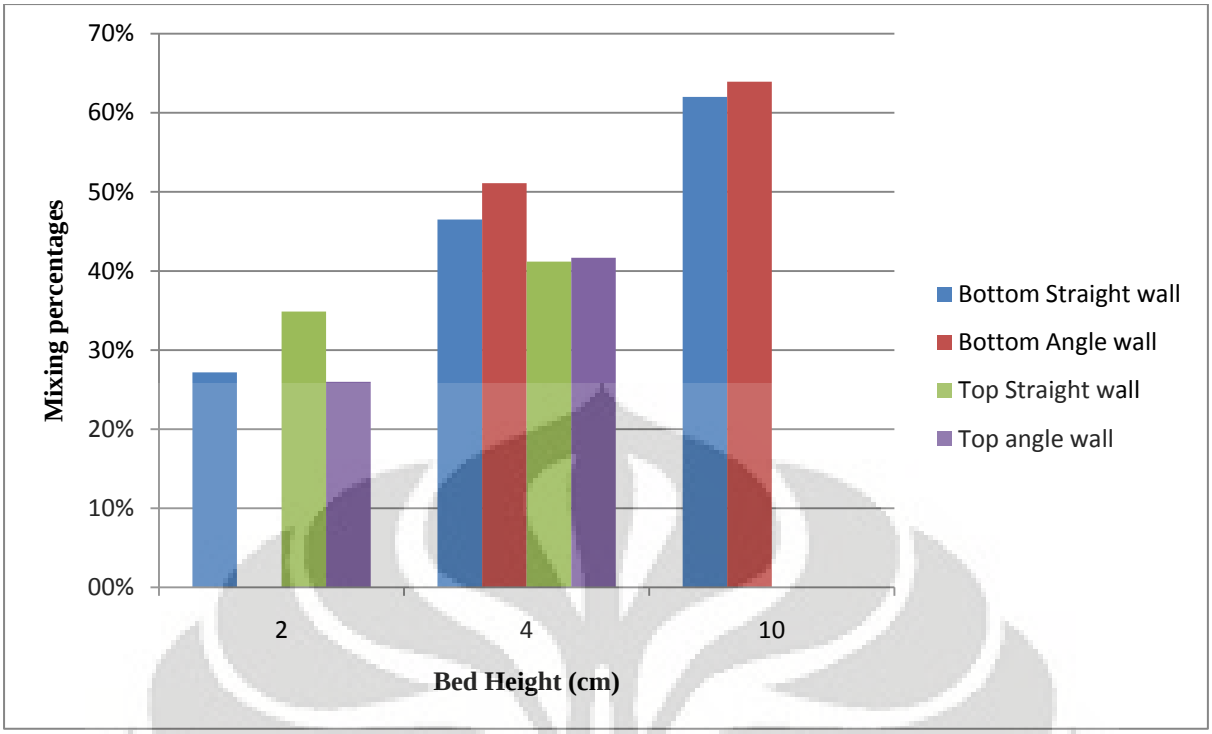


Figure 4.8. Mixing percentages of 250-355 µm and 106 -212 µm at bubbling flow rate

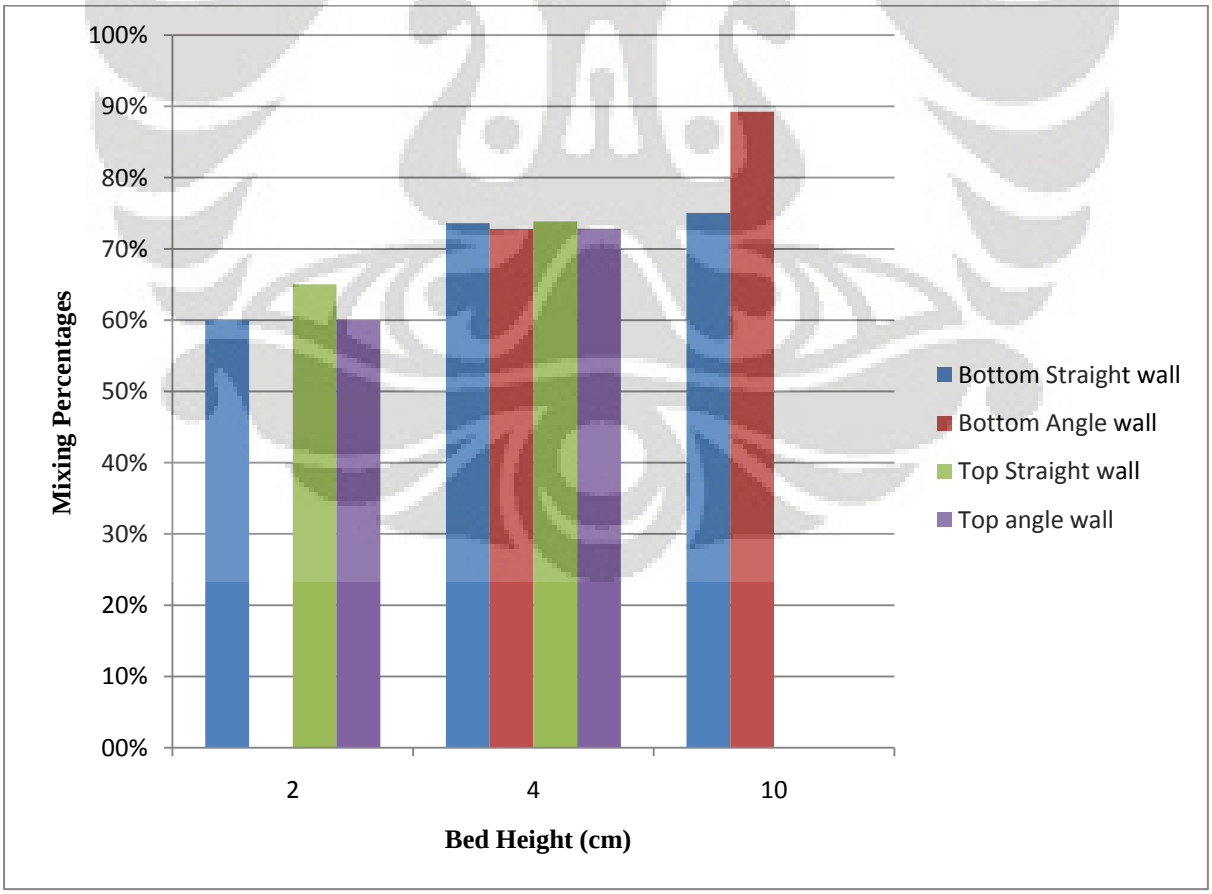


Figure 4.9. Mixing percentages of 250- 355 µm and 106 -212 µm at twice bubbling flow rate

As it has been discussed in the literature review the degree of mixing of conical fluidized bed was superior compared to the cylindrical fluidized bed when Michael Wormsbecker conducted the experiment. The reason why the conical or incline wall fluidized bed creates better mixing is because the diverging cone base eliminates the dead spaces on the bottom of the bed to enhance solid motion. Although the method that Michael Wormsbecker used was different to the one being used in this research the results should be more or less the same with Michael Wormsbecker's work.

The corresponding results gathered from this experiment does not gives an exact straight to the point answer. At one point the result is according to theory but at one point it differs. The variation in results maybe due to inaccuracy of image results and the design error of instrument which was not in good operating condition from the start of the project. To get more accurate result maybe a sampling technique to take a sample from the middle of the bed should be invented to study the understanding on what's going on inside the fluidized bed rather than observing from the sides. But it could be said that on most occasion the angle wall shows a better mixing when compared to the straight wall although there are some cases where the straight wall is creating a greater mixing. It could be concluded that the angle wall fluidized bed is a better option for mixing although it will needs some future work to get a more in-depth understanding due to the possible inaccuracy of results gathered from this experiment.

4.8 Error Analysis

Image analysis was done by determining the boundary line between mixing area and non-mixing area. However the angle when the picture was taken by digital camera may not be consistent enough. Some of the taken images are taken from an angle that is different with the other image result. It will then effect the composition of the mixing and non mixing area from the image processing results.

Because of the geometry of this fluidized bed where the width of the Perspex glass is considerably greater than the thickness, is more subject to electrostatic charging effects than normal fluidized beds. In most of the cases a layer of particle was built up on inner side of Perspex glasses, totally obscuring the phenomena under study (Geldart, 1986). In this case the layer of particle could segregated and cause false segregation pattern on the wall. As a result wrong determination of boundary layer between mixing and non- mixing area was made.

- **Stagnant Wall**

When conducting the experiment a stagnant wall can be seen through the sides of the glass. The initial thought of the stagnant wall was to be believed caused by the different size particle segregation. Therefore to prove this theory another experiment was conducted. In theory if one particle with same size is mixed it should be well mixed. Then a particle with the same size but different colour is placed inside the bed and then the bed is fluidized. After leaving it for some time another image was taken from the sides of the bed. Surprisingly the two particles of the same size did not mix. There was significant amount of particles unfluidized adjacent along the wall sighted this can be seen in figure 4.10 so the only explanation of the stagnant wall happening is due to design malfunction.

Figure 4.10. Mixing of particle in the same size

Another investigation is carried out on the imperfect fluidized bed design. An image was taken from the top of the fluidized bed with an empty bed which can be seen in figure 4.11 Through the image taken it could be seen that the sealing that was installed is creating gaps between the distributor and the bed. When a particle is in this area the air flow won't comes in contact with particles. The particles won't get fluidized because automatically the particles are in the dead spot where the air flow from the distributor will not reach this area.

Figure 4.11. Top view of inside the fluidized bed with empty bed

To further explain the bubbling of the distributor a thin layer of fine powder is placed inside the bed with the valve is opened so the bubbling of the distributor could be seen. After the bubbles could be seen another image was taken from the top of the bed which is showed in *figure 4.12* after analysing the image it was founded that the bubbling only occurred in the middle of the bed, a boundary line was drawn on the image to make the bubbling area clearer. From the image the thought of dead spot happening in the bed can be proven as it seen there are dead spot on every corner of the distributor. This is an evidence on the flawed of distributor design.

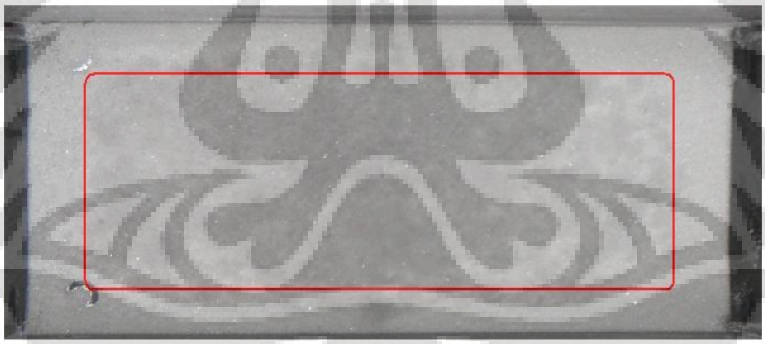


Figure 4.12. Top view of fluidized bed with thin layer of fine particle

CHAPTER 5
CONCLUSION

From the experiment that was conducted and the analysis from all the result obtained it can then be concluded that:

1. The bed height has a contributing factor to the mixing degree. As the bed height increases the mixing area also increases. The results shows at the binary systems of 106-212 and 355 – 425 microns the mixing area increases as the bed height increase for the 2 cm 13,8%, the 4 cm 38% and the 10 cm 66,7%

2. The position of the particle placement could affect the mixing degree. If the coarser particle are placed at the bottom it shows a better mixing rather that when the coarser particle are placed at the top. the mixing area for the 4 cm with straight angle for the coarse particle placed on the bottom is 44,2% while when the coarse particle are placed on the top is 29,1%

3. The resulting mixing area for the 4 cm with the same particle placement is 44,2% for the straight wall and 58,2% for the angle wall whilst for the 10 cm the mixing area is 75% for straight wall and 78,2% for angle wall. The result from the effect of wall geometry doesn't show any straight forward answer but on most occasion the angle/ incline wall fluidized bed prove to be a better mixing choice rather than a straight wall fluidized bed although it still needs more work to prove this theory.

4. The mixing area will increase along with the increment of flow rate. The area of the 2 cm bed height increases form 22,2 % to 60%

REFERENCES

Geldart, D. (1986). *Gas Fluidization Technology.* John Wiley & Sons Ltd.

Michael Wormsbecker, A. A. (2005). Segregation by Size Difference in a Conical Fluidized Bed of Pharmaceutical Granulate. *Powder Technology* , 72-80.

Rhodes, M. (2008). *Introduction to Particle Technology.* Wiley InterScience.

Rowe, P., & Nienow, A. (1976). Particle Mixing and segregation in Gas fluidised Bed. *Powder Technology 15* , 141-147.

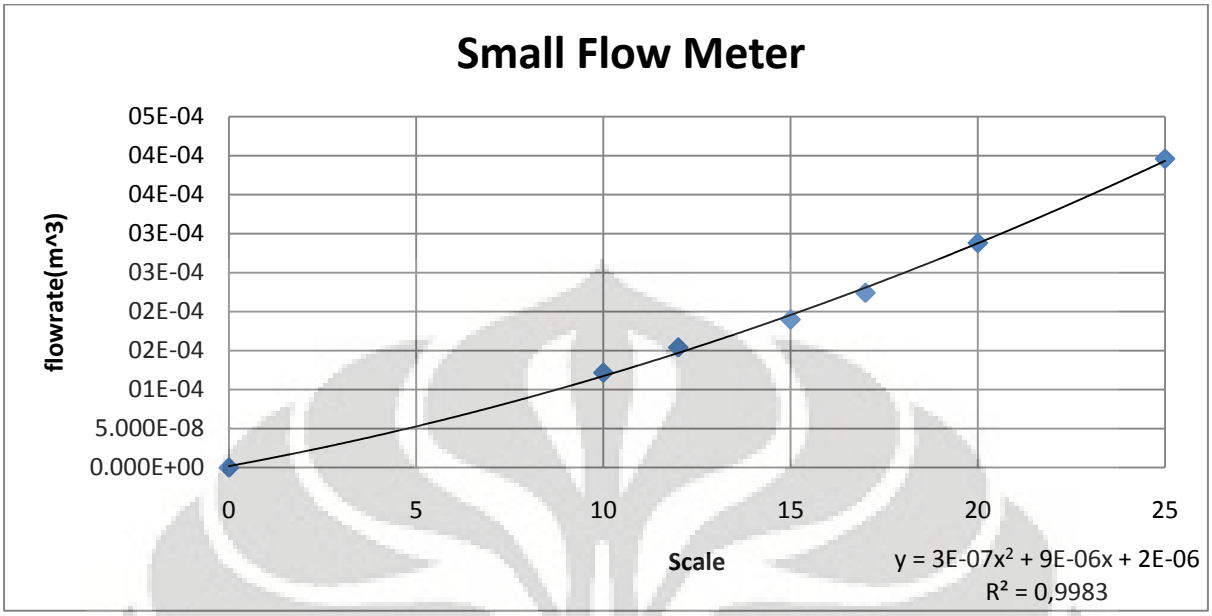
Sensing, G. I. (2005, august). *Precision Humidity Measurement for Pharmaceutical Fluid Bed Tablet Coating.* Dipetik March 15, 2009, dari GE Industrial: http://www.gesensing.com/products/resources/application_notes/930_109A.pdf

Shannon, P. (1959). *Phd Dissertation.* illinois institute of technology.

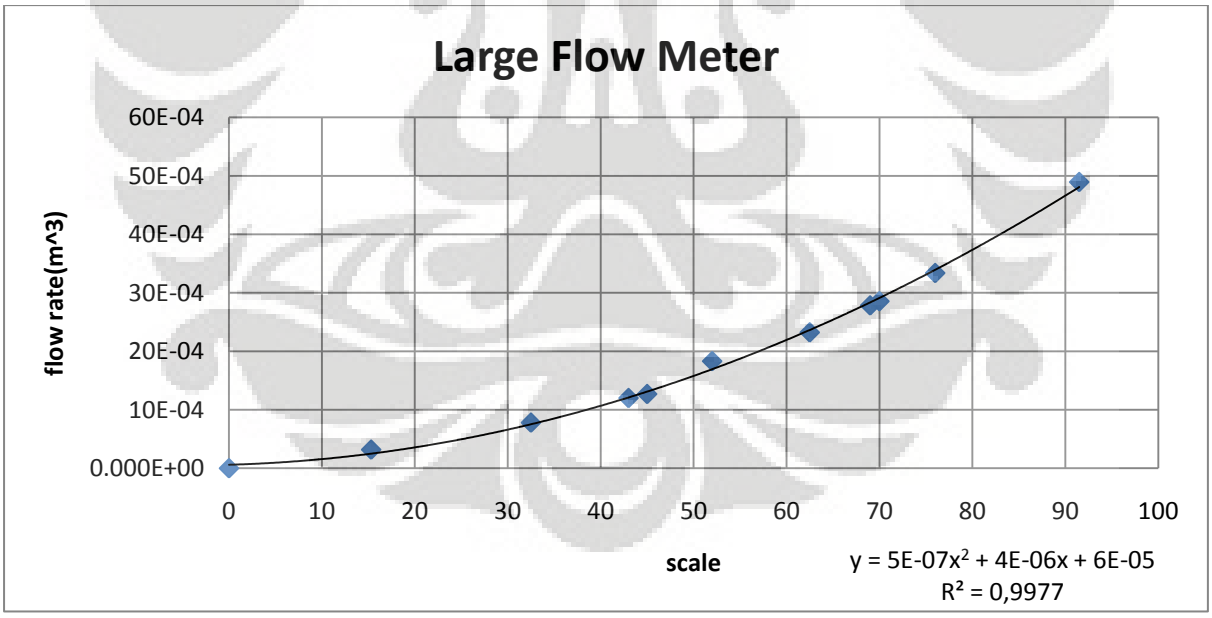
Tanfara, Helen, Pugsley, Todd and Winters, Conrad(2002)'EFFECT OF PARTICLE SIZE DISTRIBUTION ON LOCAL VOIDAGE IN A BENCH-SCALE CONICAL FLUIDIZED BED DRYER',Drying Technology,20:6,1273 — 1289

Wormsbecker, M. (2008). *STUDY OF HYDRODYNAMIC BEHAVIOUR IN A CONICAL* . Canada: Univeristy of Saskatchewan.

APPENDIX A



Calibration graph for small gas flow meter

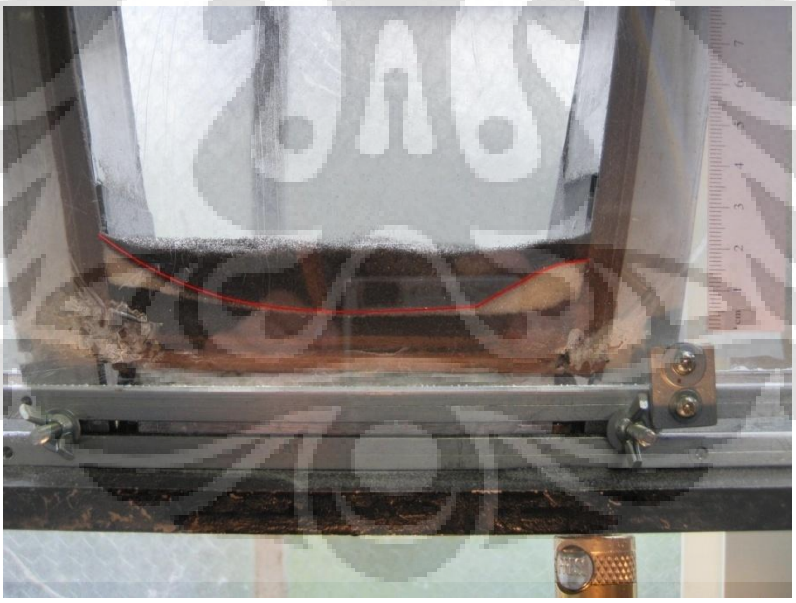


Calibration graph for large gas flow meter

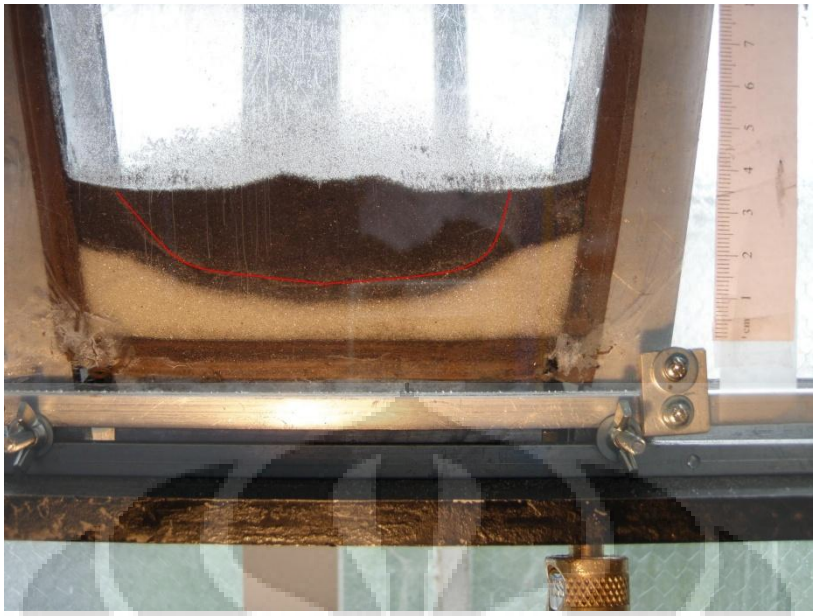
APPENDIX B

EXAMPLE IMAGE RESULTS

106-212 and 250-355 microns at 2 cm bed height bubbling flowrate



106-212 and 250-355 microns at 2 cm bed height slugging flowrate



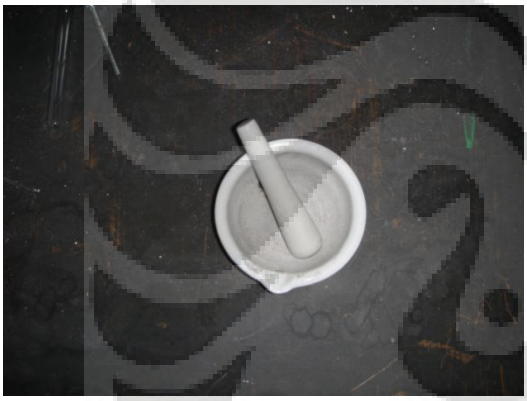
106-212 and 355-425 microns at 4 cm bed height bubbling flow rate

106-212 and 355-425 microns at 2 cm bed height slugging flow rate

APPENDIX C

Material and apparatus used in the experiment

Siever



Pestle and mortar

Ballotini glass beads