

TO CONVERT BITUMEN POWDER TO BITUMEN EMULSION

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“ I declare that this thesis is the result of my own research except as cited references.  
The thesis has not been accepted for any degree and is not concurrently submitted in  
candidature of any other degree”.

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## **ABSTRACT**

Bitumen is an important organic liquid in the nature. The main characteristics which are highly viscous, black, sticky make bitumen are the suitable to use in paving road .This is a major application for bitumen otherwise also use as an intermediate product for roof surfacing, waterproof boats and others. By emulsifying process, bitumen emulsion was creating to reduce the viscosity and get lower temperature for spraying or mixing purposes. But in bitumen emulsion industries, packaging maintenance is a major problem. High cost in packaging because of only drum and large tank is suitable to use for commercialized. For this research, the problem will be solving by converting bitumen emulsion to bitumen powder. Silicon dioxide was used as an additive to produce bitumen powder from bitumen emulsion. The trade name of silicon dioxide is SIPERNAT an is use as a powdering agent for manufacturing fine-particle powders with good dispersion characteristics In previous invention silicon dioxide is used as a additive to convert liquid bitumen to powder To remove the water from bitumen emulsion drying method was applied by using spray dryer.

## ABSTRAK

Bitumen merupakan antara bahan organik yang terpenting di alam semulajadi ini. Ciri-ciri bitumen yang mempunyai kelikatan yang tinggi, berwarna hitam dan melekit apabila bergabung dengan permukaan lain menyebabkan ia sesuai digunakan dalam pembinaan jalan. Antara kegunaan lainnya ialah sebagai bahan perantara dalam penghasilan atap rumah dan gam untuk menampal kapal dan sebagainya. Dalam proses emulsifikasi bitumen, ia dapat menurunkan kelikatan bitumen dan boleh menjalankan proses menyemburan dan pancampuran bitumen dalam suhu yang rendah. Tetapi dalam sektor perindustrian bitumen emulsi, proses pembungkusannya menjadi masalah. Ini adalah disebabkan dalam pembungkusan bitumen emulsi, hanya tong drum dan tangki besar sahaja yang sesuai dijadikan sebagai medium pembungkusan. Oleh itu, dalam kajian ini, bitumen emulsi akan ditukar menjadi serbuk bitumen. Silicon dioksida digunakan sebagai bahan tambahan untuk menjadikan bitumen emulsi ke serbuk bitumen. SIPERNAT adalah nama komersial bagi silicon dioksida yang digunakan oleh industri dan ia sesuai digunakan sebagai agen penukaran bahan kepada serbuk. Proses penyingkiran kandungan air didalam bitumen emulsi dilakukan dengan menggunakan *spray dryer*. Proses ini akan menukarkan bitumen emulsi kepada bitumen serbuk.

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**LIST OF ABBREVIATION**

<b>SiO<sub>2</sub></b>	Silicon dioxide
<b>t.g.a</b>	Thermogravimetric Analysis
CP	centipoises

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

Bitumen in the form of asphalt is obtained by fractional distillation of crude oil. Bitumen being the heaviest and being the fraction with the highest boiling point, it appears as the bottom most fraction. It is in the form of tar is obtained by the destructive distillation of organic matter, usually coal. Bitumen has been used widely in industrial especially in paving road. It is the prime feed stock for petroleum production from tar sands. In the past, bitumen was used to waterproof boats, and even as a coating for buildings; it is possible, for example, that the city of Carthage was easily burnt down due to extensive use of bitumen in construction (*www.usq.edu.com, 2005*). Besides that, bitumen is also used an intermediated product in roofing.

Bitumen is an organic liquid which are highly viscous, black, sticky and wholly soluble in carbon disulfide. Therefore, by using emulsified process we can reduce a viscous, boiling temperature and adhesiveness that we called bitumen emulsion.

There are some problems in bitumen emulsion industries. One of the major problems is a maintenances in packaging bitumen emulsion. By converting bitumen powder from bitumen emulsion, the problem will be solved.

## 1.2 Problem Statement.

At the working site, according to conventional methods (bitumen emulsion), tank lorry is required, and large scale machine equipment such as the melting kilns, storage tanks, melted material weighing machines for the melted materials, hot firing furnaces, and others equipment are necessary. As a result, plant equipment cannot be simplified, large fire is required, production control becomes more complicated and the number of personnel required will be increase. But in bitumen powder all the equipment are unnecessary.

Besides that, in bitumen emulsion industries the major problem that many industries faced in packaging maintenance. Normally in packaging bitumen emulsion, they use drum and large tank. This material is very suitable because of its can give a proper condition for the bitumen emulsion to bringing in other place. Therefore, this is not economical and cannot give a lot of profit to the industry. Although, we recycle back the entire drum that its use in paving road, it is can't reduce a cost production of bitumen emulsion. So, by converting bitumen emulsion to bitumen powder its can reduce packaging cost. For example, in packaging its can use a plastic bag to pack bitumen powder. The cost of packaging by using plastic bag is cheaper than using are drums or large tank.

Furthermore, bitumen emulsion can't give optimize space in packaging. This situation happed because of emulsified process using a lot of water to mix with the bitumen. For the example, in one drum can contain 100 kg of bitumen emulsion. From emulsified process 60 percent by weight is bitumen an the rest is water ( 40 percent by weight). At working site in paving road, all the water that contain in bitumen emulsion was vaporize after contact with the hot weather conditions. So, for the actual weight that contain in one drum is only 60 percent by weight. As the result, a lot of drum must be used in packaging bitumen emulsion. But, when packaging bitumen powder in the same

drum, is totally can contain 100 percent of bitumen. Its can minimize the space in packaging bitumen.

On other hand, it is very complicated to export bitumen emulsion because of is very high cost. Related from the previous problem that's about bitumen emulsion maximize the space in packaging, therefore to export bitumen emulsion, it must use a lot of ship or flight. It very expansive in cost production. That mean, it will be reduce a profit for the industry.

### **1.3 Objective of Research**

Objective of this research is to study properties of bitumen emulsion and capability to convert bitumen powder from bitumen emulsion.

### **1.4 Scope of Research**

To achieve the objective, scopes have been identified in this research. The scopes of this research are listed as below:-

- 1.0) To study properties of bitumen emulsion by using thermogravimetric differential analyzer( t.g.a)
- 2.0) To study the properties of bitumen emulsion by using viscometer.
- 3.0) To study how to convert bitumen powder from bitumen emulsion by using spray dryer

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Bitumen

Bitumen is a category of organic liquids which are highly viscous, black, sticky and wholly soluble in carbon disulfide. Bitumen molecules can contain thousand of carbon atoms. This make bitumen one of the most complex molecules found in nature. It is natural state; it is not recoverable through a well like conventional petroleum.

On average, Bitumen is composed of

Carbon – 83.2%

Hydrogen – 10.4%

Oxygen- 0.94%

Nitrogen – 0.36%

Sulphur – 4.8%

*(wikipedia, 2006)*

Bitumen in the form of asphalt is obtained by fractional distillation of crude oil. Bitumen being the heaviest and being the fraction with the highest boiling point, it appears as the bottom most fraction. It is in the form of tar is obtained by the destructive distillation of organic matter, usually coal.

## 2.2 History of Bitumen

In British English, 'bitumen' is often used interchangeably with both 'asphalt' and 'tar'. In American English, 'bitumen' is most commonly used in engineering jargon to explicitly include both asphalt- and tar-based materials. In Australian English, 'bitumen' is used as the generic term for road surfaces (*www.usq.edu.com, 2005*). . In 1719, a Cree named Wa-Pa-Su (meaning the swan) presented a sample of bitumen as waterproofing on birch bark canoes and they also made smudge pots to ward off mosquitoes in summer time. (*www.usq.edu.com, 2005*).

Natural bitumen is probably the oldest petroleum product to be used by man. The ancient Egyptians used it for embalming mummies and in jewelers (*ascelibrary,2005*). Through the ages it has been used in Middle Eastern countries for water-proofing and constructional jobs. The bitumen was obtained from natural seepages out of the ground in various parts of the Middle East. (*wikipedia,2005*) Although naturally occurring bitumen are still available they account for only a very small percentage of the bitumen used today.

Most geologists believe that naturally occurring deposits of bitumen are formed from the remains of ancient, microscopic algae and other once-living things. These organisms died and their remains were deposited in the mud on the bottom of the ocean or lake where they lived. Under the heat and pressure of burial deep in the earth, the remains were transformed into materials such as bitumen, kerosene, or petroleum. A minority of geologists, proponents of the theory of abiogenic petroleum origin, believe that bitumen and other hydrocarbons heavier than methane originally derive from deep inside the mantle of the earth rather than biological detritus. (*www.usq.edu.com, 2005*).

### 2.3 Emulsion

Generally, emulsion is defined as dispersion of liquid droplets in another immiscible liquid. Dispersion refers to finely divided droplets, and immiscible means that one liquid will not dissolve the other. (*freepatent,2005*). Most people are familiar with emulsion. We see them every day. Milk, mayonnaise, water-based paint, and hand lotion are few examples. Every person on earth has made an emulsion at home time in their lives, hopefully on a daily basis. Every time someone washes their hands, they make an emulsion of dirt and oil particles stabilized by surfactant (soap) in water.

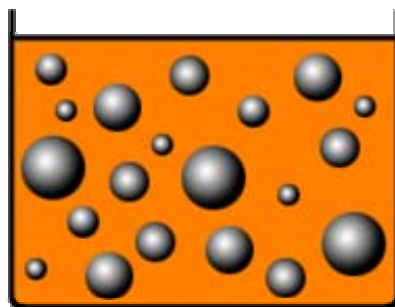


Figure 2.1: There is an upper limit to the bitumen content of an emulsion, which mainly depends on the relative volume of the two phases. When reaching this limit there is insufficient room for more droplets without deforming them. (*asphalt-akzonobel,2006*)

The droplets will be packed so tightly that they will partly adhere to each other and eventually the water entrapped between the droplets will become water droplets. The result will be a water-in-oil emulsion, or an inverted emulsion. Such an emulsion has the nature of bitumen, with high viscosity. The limit to bitumen content is in the range 70% to 80% and depends mainly on the size distribution of the particles. (*freepatent,2006*)

## 2.4 Bitumen Emulsion

### 2.4.1 History of Bitumen Emulsion

In 1922, two British scientists patented their invention of bitumen emulsion, which was further improved the following year when a new patent was filed in France. The use of bitumen emulsion rapidly spread throughout the world. (K. Louw, K. Spence and P. Kuun, *Jurnal*). The manufacture of bitumen emulsion is in an anionic character and make it from the soap formed by neutralizing long chain fatty acids such as tall oil, oleic acids, stearic acids and naphthenic acids. But in anionic bitumen emulsion presented adhesive problem with certain aggregates such as quartzites and granites. To overcome this problem cationic bitumen emulsion are created in late 1950's.

Bitumen is a highly viscous material, which is useful in road making and for other purposes. Because of its high viscosity it is difficult to use in the cold or in an untreated condition. One method of reducing the viscosity is by heating the material and using it hot. Another is by adding a light petroleum distillate as diluents. This will thin the bitumen and allow its application at lower temperature. However, the distillate will subsequently be lost by evaporation and may give rise to environmental problems. (wikipedia, 2005) A third, with which is by forming an emulsion of bitumen in water. Emulsions have lower viscosities than the starting bitumen and can be used at lower temperatures for spraying or mixing purposes.

However, the dispersion of bitumen in a medium of much lower viscosity such as water is an unfavorable process on hydrodynamic grounds. This problem is further complicated by the desirability in certain applications to utilize emulsions containing relatively high bitumen phase volumes without sacrificing emulsion fluidity. As the bitumen and water go through an emulsion of colloid mill, the bitumen is divided into a very small droplet. The water contain an emulsifier, also known as a surface active agent

or surfactant. It is able to move to a surface or the point where two immiscible liquids come together.

#### **2.4.2 Types of Bitumen Emulsion**

There are many types of bitumen emulsion. The different types of bitumen Emulsion is depending on their surfactant agent of emulsifier. Generally there have different types of surfactant agent that we use in process to produce bitumen emulsion. Suitable surfactants include anionic surfactants, mixtures of anionic and non-ionic surfactants, cationic surfactants (which are preferred) and mixtures of cationic and non-ionic surfactants.

To obtain a stable bitumen emulsion, an emulsifier has to be added. The bitumen droplets are kept apart due to the effect of the ionized emulsifier molecules, which orientates themselves to the surface of the droplets forming an electrostatic force field. The stability of the emulsion depends to a great extent on the strength of this force field. If the emulsifier is of cationic type, the droplets will be positively charged (cationic emulsion) while with an anionic emulsifier the charge will be negative (anionic emulsion).

Most countries have specifications for both anionic and cationic emulsions. These two categories of emulsions are divided into three classes, depending on the stability of the emulsion when in contact with aggregate or pavement surfaces, i.e. rapid, medium and slow setting emulsions. A rapid setting emulsion has little or no ability to mix with an aggregate, a medium setting emulsion is expected to mix with a coarse but not fine aggregate and a slow setting emulsion is designed to mix with fine aggregate.



### 2.4.3 Cationic Bitumen Emulsion

In the mid 1940s cationic emulsions were introduced, which meant a major technical improvement. (*freepatent,2006*). The cationic emulsifiers are generally based on long hydrocarbon nitrogen compounds, such as alkyl amines. The alkyl amines are powerful surface-active compounds with great influence on the surface tension. They can be modified in a number of ways to meet almost any requirement. Most emulsifiers need to be reacted with an acid before they can function and the pH of the emulsion will by this be lower than 7. The acid, in most cases hydrochloric acid, reacts with the nitrogen and forms an ammonium salt. In a batch plant this is often done by slowly adding acid and emulsifier to warm water under continuous agitation. When all emulsifier has been added and dissolved, the pH is adjusted by adding more acid until the correct pH is reached. In a continuous plant, the emulsifier is injected into the water line. Acid is added the same way and the reaction takes place before the water enters the mill. Liquid and easily dispersible emulsifiers are preferable in this type.

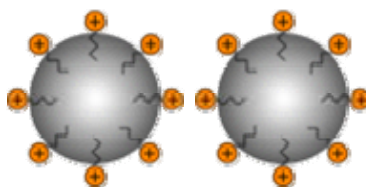


Figure 2.2: Cationic Emulsion (*asphalt-akzonobel, 2006*)

### 2.4.4 Anionic Bitumen Emulsion

Anionic emulsions were first developed in the early 1900s. They found their applications but the growth in use was relatively slow. (*wikipedia, 2005*)

Anionic emulsifiers are normally based on fatty acids. A fatty acid molecule consists of a long hydrocarbon chain and terminates with a carboxyl group. The emulsifier solution

is prepared by reacting the anionic emulsifier with sodium hydroxide. This reaction is called saponification. The pH of an anionic emulsion is higher than 7 and the emulsion normally contains an excess of sodium hydroxide, which eventually reacts with any natural acids contained in the bitumen.

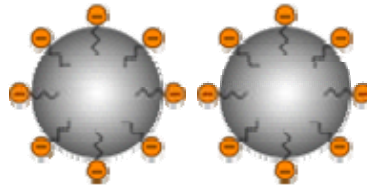


Figure 2.3 : Anionic Emulsion. (*asphalt-akzonobel, 2006*)

#### 2.4.5 How Are Bitumen Emulsion Stabilized

The emulsifier coats the surface of each bitumen droplets. The emulsifier has chemical structure that is partially oil soluble, and partially water soluble. The oil soluble portion of the molecules (also known as a “tail”) embeds itself in bitumen droplet’s surface, and the water soluble portion (the “head”) remains above the droplet’s surface exposed to the water surrounding the droplet. (*asphaltinnovation, 2005*) The emulsifier tail anchors the head in place, and the head acts to protect the surface of the bitumen droplet from contact with other bitumen droplets. The head protect the surface through one of two mechanisms. In one case, the head is so large that two approaching droplet surface simply can’t get close to each other. In other case, the head of emulsifier has an electrical charge, either positive or negative, that repels the same charge on the other droplets which keeps the surface from touching. When the emulsifier uses size for stabilization, also known as steric hindrance, it is known as a nonionic emulsifier(

nonionic means “no electric charge”). Charge emulsifier are classified as either cationic (meaning “positive electrical charge”) or anionic (meaning “negative electric charge”).

## **2.5 Acid**

Acid is used to form a salt from the emulsifier and for lowering the pH to a predetermined level. When using a cationic system hydrochloric acid is used. In an anionic system sodium hydroxide is used.

## **2.6 Stabilizer**

When using a cationic system calcium chloride can be added to the emulsion to increase the stability and prevent swelling of the bitumen droplets caused by osmosis, in example, when there is a high content of salt in the bitumen and the water is pulled into the bitumen droplets.

In an anionic system trisodium polyphosphate can be used for the same purpose.

## **2.7 Silicon Dioxide**

To convert bitumen powder from bitumen emulsion, silicon dioxide is used as additive in converting process. The idea is created from the previous invention to convert liquid bitumen to bitumen powder. Silicon dioxide is also called *sipernat* for the trade name. (*Chemical Tradename Dictionary, Wiley- Vch, page 338*). According to the invention there is prepared a powder (or pulverulent) bitumen concentrate containing 10 to 85 weight %, preferably 40 to 80 weight %, of synthetic silica. As synthetic silicas there can be used precipitated silica as well as pyrogenically produced silica. The

precipitated silica can have a BET-surface area of 120 to 500 m<sup>2</sup>/g. In a given case it can be ground by a steam jet, spray dried or spray dried and ground. The pyrogenically produced silica can have a BET-surface area of 100 to 400 m<sup>2</sup>/g. The bitumen concentrate of the invention can be produced by simply mixing the liquid bitumen with the synthetic silica or spraying the liquid bitumen on the synthetic silica. The bitumen concentrate of the invention has the advantage that in spite of the high proportion of bitumen it is powdery, temperature stable and flowable. Furthermore according to another aspect of the invention, the use of the powdery bitumen concentrate in asphalt mixture for building roads, results in a surprisingly high stiffening effect. (Muller, Karl-Hans and Barthel Walter, *pulverulent bitumen concentrate its use, freepatent, 1979*)

### **2.7.2 General Description of Silicon Dioxide**

Crystalline silicon dioxide (in several forms: quartz, cristobalite, tridymite) is an important constituent of a great many minerals and gemstones, both in pure form and mixed with related oxides. Beach sand is mostly silica. SiO<sub>2</sub> is formed by strong, directional covalent bonds, and has a well-defined local structure: four oxygen atoms are arrayed at the corners of a tetrahedron around a central silicon atom:

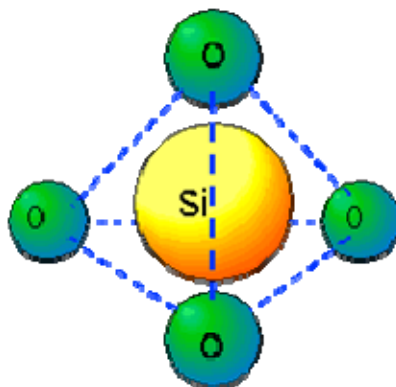


Figure 2.4: Structure of Silicon Dioxide (*wikipedia, 2006*)

The oxygen atoms are electronegative, and some of the silicon valence electron density is transferred to the oxygen neighbors, but it is incorrect to regard the material as a salt of a  $\text{Si}^{+4}$  ion with  $\text{O}^{-2}$  ions, as is sometimes seen in the literature. The directionality of the bonds is essential to the observed structures.

## 2.8 Drying Process

The term “drying” is used to refer to removal of other organic liquid, such as benzene or organic solvent from solid. Drying generally means removal of relatively small amount of water from material. Drying method and process can be classified in several different ways. Drying process can be classified as batch, where the material are inserted into the drying equipment and drying proceed for a given of time of continuous, where the material is continuously added to the dryer and dry material is continuously removed.

### 2.8.1 General Characteristic of Dryers

Removal of water from solid is most often accomplished by contacting them with air of low humidity and elevated temperature. Less common, although locally important, drying processes apply heat radiatively or dielectrically in these operations as in freeze drying, the role of any gas supply is that of entrainer of the humidity. (M.W Stanley, *Chemical Process, Selection and Design*, Butterworth Heinemann, 1990, page-268)

The nature, size and shape of the solid, the scale of the operation, the method of transporting the stock and contacting it with gas, the heating mode, are some of the many factors that have led to the development of considerable variety of equipment. The most elaborate classification of dryers is that of Kroll (1978) which assigns one of the letters for the kind of operation. As modified by Keey (1972), it comprises 39 main classes and the total of 70 with subclasses. (M.W Stanley, *Chemical Process, Selection and Design*, Butterworth Heinemann, 1990, page-272)

They take into account the method of operation, the physical form of the stock, special features, scale of production and drying time. The wide spread of these numbers reflect the diversity of individual design of the same general kind of equipment, differences in moisture content, and differences in drying properties of various materials. Fluidized bed dryers, for example, are operated as batch or continuous, for pharmaceuticals or bitumen, at rates of hundred or many thousands of pound per hour.

The important characteristic of the dryer is the residence time distribution of solid in it. Dryers in which particles do not move relative to each other provide uniform time distribution. In spray, pneumatic conveying, fluidized bed, and other equipment in which the particles tumble about, a substantial variation in residence time develops. Spray and pneumatic conveyor have wide time distribution while rotary and fluidized bed units have narrower but far from uniform ones. Differences in particle size

also lead to nonuniform drying. In pneumatic conveying dryers particularly, it is common practice to recycle a portion of the product continuously to ensure adequate overall drying. In other cases recycling may be performed to improve the handling characteristic when the feed material is very wet.

## **2.8.2 Types of Dryer Equipment**

There are many types of dryer in our chemical environment. All the equipment are depend on the situation and the characteristic of the material that desired to dried.

### **2.8.2.1 Rotary Cylindrical Dryers.**

Rotary cylindrical dryers are suited for free- flowing granular materials that require drying times of the order of one hour or less. Materials that tend to agglomerate because of wetness may be preconditioned by mixing recycle dry product.

Such equipment consists of a cylindrical shell into which the wet material is charged at one end and dry material leaves at the other end. Drying is accomplished by contact with hot gases in parallel countercurrent flow or with heat transfer through heated tubes or double shell. Designs are available in which the tubes rotate with the shell or are fixed in space.

Diameters typically are 4-10 ft and length are 4-15 diameters. The product of rpm and diameters is typically between 25 and 35. superficial gas velocities are 5- 10 ft/sec but lower values may be needed for fine product, and rates up to 35 ft/sec may be allowable for coarse materials. To promote longitudinal travel of the solid, the shell is mounted on a slope of 1 in 40 or 20. (M.W Stanley, *Chemical Process, Selection and*

*Design, Butterworth Heinemann, 1990, page-275*) In countercurrent dryer the exit temperature of the solid approaches that of the inlet gas. In the parallel current dryer, the exit gas is 10-20 degree above that of solid. For design purpose the temperature of exit solid in parallel flow may be taken as 100 degree.

Flights attached to the shell lift up the material and shower it as a curtain through which the gas flows. The shape of flights is a compromise between effectiveness and ease of cleaning. The number is between 2 and 4 times the diameter of the shell in feet, and their depth is between 1/12 and 1/8 of the diameter. Holdup in the dryer depends on details of design and operation, but 7-8 % is a usual figure. Cross sectional holdup is larger at the wet end than at the dry end. An 85% free cross section commonly is adopted for design purpose, the rest is taken up by flights and settled and cascading solid.

### **2.8.2.2 Drum Dryer**

Solution, slurries and pastes may be spread as a thin film and dried on the steam heated rotating drums. Twin drums are commonly rotate in opposite directions toward the nip of the feed, but when lumps are present that could damage the drums, rotation is in the same direction. Top feed with an axial traveling distributor is most common. An agitator is also provided to keep solids in suspension.

For mechanical reasons the largest drums made are 5 ft dia by 12 ft 188 sqft of curved surface. A 2x2 ft drum is also listed in manufacturers catalog. The materials come off as flakes 1-3 mm or less thick. They are broken up to a standard size of about 1/4 in. square. That process makes fines that are recycled to the dryer feed. Drying times fall in the ranges of 3-12 sec. Many laboratory investigations have been made of drying rates and the heat transfer coefficient, but it appears that the only satisfactory basis of sizing



plant equipment is pilot plant data obtained with a drum of a foot or more in diameter. Usually plant performance is superior to that of pilot plant units because of steadier long time operation.

A safe estimate of power requirement for double drums dryers is approx 0.67 HP / (rpm) (100sqft of surface). Maintenance can be as high as 10 %/yr of the installed cost. Knives last from 1 to 6 month depending on abrasiveness of the slurry. Competition for drum dryers are solid belt conveyor that can be handle greater thickness of paste materials, and primarily spray dryers that have large taken over the field.

### **2.8.2.3 Fluidize Bed Dryers**

Free flowing granular materials that required relatively short drying times are particularly suited to fluidize bed drying. When longer drying times are necessary, multistage, recirculation or batch operation of fluidize beds still may have advantages over other modes.

A fluidize bed is made up of mass of particles buoyed up out of permanent contact with each other by flowing fluid. Turbulent activity in such a bed promotes high rates of heat and mass transfer and uniformly of temperature and composition throughout. The basic system include a solids feeding device, the fluidizing chamber with perforated distributing plate for the gas, an overflow duct for removal of the dry product, a cyclone and other equipment for collecting fines, and the heater and blower for the gaseous drying medium.

Much ingenuity has been applied to the design of fluidized bed drying. Shallow bed are easier to maintain in stable fluidization and of course exert a smaller load on the air blower. Pressure drop in the air distributor is approximately 1 psi and that through

the bed equals the weight of the bed per units cross section. The cross section is determined by the gas velocity needed for fluidization. It is usual allow 3-6 ft of clear height between the top of the bed and the exhaust duct. Fines that are entrained are collected in a cyclone and blended with the main stream since they are very dry due the small size. Normally entrainment is 5-10 % but can be higher if the size distribution is very wide. It is no regarded as feasible to permit high entrainment and recycle back to the drying chamber, although this is common practice in the operation of the catalytic cracking equipment. Here the fluidized mass is of auxiliary spheres, commonly of plastic such as polypropylene, into which the solution is sprayed. The feed material deposits uniformly on the spheres, dries there, and then is knocked off automatically as it leaves the drier and leaves the auxiliary sphere behind. When a mass of dried particle can be provided to start a fluidized bed drying process, solution or pastes can be dried after deposition on the seed material as on the auxiliary spheres. Such as process is employed, for instance, for growing fertilizer granules of desired large sizes, and has largely replaced rotary dryers for this purpose.

#### **2.8.2.4 Spray Dryer**

Suitable feeds to a spray dryer are solution or pump able pastes and slurries. Such a material as atomized in a nozzles or spray wheel, connected with heated air or flue gas conveyed out of the equipment with pneumatic or mechanical type of conveyor. Collection of fines with the cyclone separator or filter is the major aspect of spray dryer operation. The flow from nozzles is largely downward so that the dryer is slimmer and taller. In some operation, the heated air is introduced tangentially, and then the process is called mixed flow. Most of the entries are parallel flow, but the heavy-duty detergent is in counter flow, and the titanium dioxide is either parallel or mixed flow. Counter flow is thermally more efficient, result in less expansion of the product particles, but many be harmful to thermally sensitive products because they are exposed to the high air

temperature as they leave the dryer. The flat-bottomed dryer contact the exiting solid with cooling air and is thus adapted to thermally sensitive materials.

Two main characteristic of spray drying are the short drying time and the porosity and small, rounded particles of product. Short drying time is a particular advantages with heat sensitive materials. Porosity and small size are desirable when the material subsequently is to be dissolve or dispersed .

The means residence time of the gas in a spray dryer is the ratio of vessel volume to the volumetric flow rate. Because of slip and turbulence ,the average residence times of the particles are substantially greater than the mean time of the air, definitely so I the case of countercurrent of mixed flow. Surface moisture is removed rapidly, in less than 5 sec as a rule, but falling rate drying takes much longer. Nevertheless , the usual drying operation is completed in 5-30 sec. The residence time distribution of particles is dependent on the mixing behavior and on the size distribution. The coarsest particles fall most rapidly and take longest for complete drying. If the material is heat sensitive , very tall towers in parallel flow must be employed. Otherwise, countercurrent or mixed flow with high air temperatures may suffice. In some cases it may be feasible to follow up incomplete spray drying with the pneumatic conveyor.

The conical section is for gathering and efficient discharge of the dried product. The lateral throw of spray wheels required a vessel of large diameter to avoid accumulation of wet material on the walls. Length to the diameter ratios of 0.5-1.0 are used in such cases. The downward throw of nozzles permits small diameters but greater depth for a given residence time. L/D ratio of 4-5 of more are used.

Proper atomization of feed is the key of successful spray drying. The three devices of commercial value are pressure nozzles, pneumatic and rotating wheels of various design. Usual pressure employed in nozzles range from 300 to 400 psi. and the orifice diameters are 0.012- 0.15 in. an acceptably narrow range of droplet sizes can be

made for are feed of particular physical properties by adjustment of pressure and diameter. Multiple nozzle are used for atomization in large diameter tower. Because of the expense motive are of steam, pneumatic nozzles are used mostly in small installation such as a pilot plant, but there are most suitable for dispersion of stingy materials such as polymers and fibers. The droplets size increase as the motive pressure is lessened, he range of 60 -100 psi being usual. Many different shapes of orifice and vanes are used for feed of various velocities, erosive ness, and clogging tendencies.

The main variables in operations of atomizers a feed pressure, orifice diameter, flow rate and motive pressure for nozzles and geometry and rotation speed of wheels. Enough is known about these factors to enables prediction of size distribution and throw of droplet in specific equipment. A clear choice between nozzles and spray wheels is rarely possible and maybe arbitrary.

## **2.9 Equipment for analysis**

### **2.9.1 Thermogravimetric Analysis (TGA)**

Thermogravimetric Analysis or TGA is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature. Such analysis relies upon a high degree of precision in three measurements: weight, temperature, and temperature change. TGA is commonly employed in research and testing to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues.

A sample is placed into a tare TGA sample pan which is attached to a sensitive microbalance assembly. The sample holder portion of the TGA balance assembly is subsequently placed into a high temperature furnace. The balance assembly measures the initial sample weight at room temperature and then continuously monitors changes in sample weight (losses or gains) as heat is applied to the sample. TGA tests may be run in a heating mode at some controlled heating rate, or isothermally. Typical weight loss profiles are analyzed for the amount or percent of weight loss at any given temperature, the amount or percent of non combusted residue at some final temperature, and the temperatures of various sample degradation processes.

## 2.10 Viscosity

It can be define the viscosity is measure the resistance to the fluid flow as a result of intermolecular cohesion. In other words, viscosity can be seen as internal friction to fluid motion which cab then lead to energy loss.

Viscosity can be imagine as a fluid as agroup of molecules. When the fluid is force to moved, the first fluid molecule which receive the force will transfer the momentum to the molecule of the same layer in front. At the same time, it also interact with the molecule in the top and bottom layer, which generate a kind of friction which is referred to as internal friction. This internal friction give rise to shear stress between different layer of fluid molecules. In the viscosity we can make relationship between the shear stress and shear rate. The relationship between viscosity, shear stress and shear rate is linear.

$$\tau = \mu du/dy$$

in this equation it shown that increasing the viscosity, make a shear stress also increase.  $Du/ dy$  is represent as a shear rate of the fluid. It also as a resistance of the between the fluid and the wall. The viscosity property can be seen as internal friction or resistance to

the flow. The higher the viscosity, the greater the resistance the flow must overcome in order to move forward. In gradient constant the fluid call Newtonian fluid. Since it is can measure of internal friction of the fluid, it can change with the pressure and the temperature and its value for certain condition can be obtain through an establish empirical correlation.

## CHAPTER 3

### METHODOLOGY

#### 3.1 Introduction

For this research the type of method that is used is experimental method. Drying process is the main process to convert bitumen powder from bitumen emulsion. There are four part of this experiment. Firstly, to determine the properties of bitumen emulsion by using thermogravimetric analyzer (t.g.a), viscometer and oven to obtain the solid weight. Secondly, is measurement of particle size for the fine powder of silicon dioxide. Third, preparation of homogenous solution between bitumen emulsion and silicon dioxide. Fourth, to convert bitumen powder from bitumen emulsion by drying method.

The main part of this research is to prepared homogenous solution combination of silicon dioxide and the bitumen emulsion. This experiment will be find the potential silicon dioxide as a additive agent to convert bitumen emulsion to bitumen powder.

## 3.2 Material

### 3.2.1 Bitumen Emulsion

The sample of bitumen emulsion that used as main material is sponsored by ACP-DMT SDN. BHD that located in Port Klang. The types of bitumen emulsion that used in this research is cationic bitumen emulsion.



**Figure 3.1** Bitumen emulsion