

**Development and Characterization of Porous Epoxy/Bentonite Clay particles through
Water-Oil Homogenization Method**

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**A project report submitted in partial fulfilment of the
requirements for the award of Bachelor of Engineering
(Hons.) Petrochemical Engineering**

**Faculty of Engineering and Green Technology
Universiti Tunku Abdul Rahman**

September 2015

DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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Development and Characterization of Porous Epoxy/Bentonite Clay particles through Oil-Water Homogenization Method

ABSTRACT

This research work is carried out to develop and characterized porous particles by using epoxy resin and bentonite clay through water-oil homogenization method. Various bentonite loading was used to study the development of PEB particles and to investigate the physical and chemical properties of produced PEB particles. Characterization of PEB particles based on the particle size and distribution, density, microstructure and morphology properties was then carried out. The produced PEB particle is prepared by mixing of epoxy/bentonite with polyamine by using mechanical stirrer for 2-3 minutes and homogenized for 3-4 minutes with fixed mixture: water ratio of 1:2, while the homogenized mixture cured at 80°C for 24 hours using oven. Five different percentage of bentonite loading (10wt%, 20wt%, 30wt%, 40wt%, and 50wt %) was used to produced PEB particles. The effect of mixture: water ratio was observed and the fixed mixture: water ratio of 1:2 was obtained and used for PEB particles production. Characterization of PEB particles has been done, the particles size of produced PEB particles is varied according to the different bentonite loading and average density of produced PEB particles is decreases with increasing in bentonite loading. Besides, the surface structure and porosity of produced PEB particles varies according to different percentage of bentonite loading.

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LIST OF SYMBOLS / ABBREVIATIONS

PEB	Porous epoxy/bentonite clay
DGEBA	Diglycidylether of bisphenol A
ECC	3, 4-Epoxy cyclohexylmethyl-3, 4-epoxy cyclohexane carboxylate
KOH	Potassium Hydroxide
KBR	Potassium Bromide
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope

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Appendix A	Particles Size Analysis full report
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CHAPTER 1

INTRODUCTION

1.1 Background of study

During current globalization, application of porous particles have become very famous and received extensive attention for many applications such as in drug delivery, photocatalysis, fuel cells, tissue engineering and absorbents, due to its enhanced unique physical and physicochemical properties. The characteristic of porous particles depends vitally on the colloidal/emulsion preparation step. Some properties of porous particles can be affected greatly by the particles size, shape and also the accumulation of nanoparticles composing the colloidal suspension. Besides that, the size and accumulation of nanoparticles will define the inter-particle pore size. Meanwhile in the event of nanoparticles shape, encapsulated porous particles can be produced by introducing particles of flat and sheet geometry such as graphene as the starting colloidal particles (Chang & Jang, 2014).

Polymer based porous particle particularly of spherical shape has obtained high demand in many industries such as catalyst and gas absorption for decades. The polymer based porous particles can be classified into three types which are macroporous, mesoporous, and microporous. This classification depends on its pore size correspondingly, which is more than 50nm, between 50 - 2nm, and less than 2nm. Meanwhile, there are important characteristics that differentiate the polymer porous particles from gel-type polymer particles which are the high crosslinking between the chain and the porosity nature. These changes eventually highly contribute to various features of polymer based porous particle such as the capability to absorbed diluents with differences in polarity as well as increases in fragility and large surface area.

Meanwhile, the chemical nature, functionality, size and its dispersity are the other features that are shared with the non-porous particles.

Generally, different applications of polymer based porous particles require different combination of the features as mention earlier. In addition, the wide applications of porous particles in industries such as catalyst and sensors are dependent on the size of the particles. The sizes of porous particles are inversely proportional to the surface area, where smaller size of porous particles provide large specific surface area and vice versa. Eventually, the porous particles somehow are impractical to some industries due to the high processing and maintenance cost. The application of porous particle has always become a milestone for various researchers to come out with a new idea and development in order to achieve most compatible technology to produce porous particles. (Chang & Jang, 2014).

This research work is mainly focused on the development of epoxy based clay porous particles which are classified into three stages. The first stage is focused on the production of porous particles from epoxy resin as a matrix of bentonite clay particles. Water-oil homogenization method was used to produce the porous epoxy/bentonite clay (PEB) particle using homogenizers. Bentonite clay which is also known as impure clay, consisting generally of montmorillonite was used as binding site for epoxy resin during homogenization. Polyamine was used as curing agent or hardener for epoxy resin in this research (Kolar & Svitilova, 2004).

In the earliest stage, the ratio of epoxy/bentonite mixture to water was varied and the effect on the production of porous epoxy/bentonite clay particles was studied. In the second stage, the effect of bentonite loading on the production of PEB particles at constant mixture/water ratio was observed. In the third stage, characterization of PEB particles done by using various testing methods which are scanning electron microscope (SEM), particles size analyser and density. The purpose of characterization was to study and analyses the surface morphology such as size and shape as well as surface porosity presence on the PEB particles, the average particle size and distribution and the density of PEB particles.

1.2 SIGNIFICANCE OF STUDY

In this project, it was planned to develop a new porous particles through water-oil homogenization of epoxy and bentonite clay in water. Mostly the production of porous particles is done by various method or technique, where the production involves high technology and processing cost. Moreover, the production of porous particles reflects badly on the environment by which the capability for porous particles to function as a transport system also raised the concern on the transport of heavy metals and other environmental contaminants. Environmental factor have always become the vital role upon discovery or invention of new technology or invention that benefits both human and environment (Tsujioka, 2009).

The production of porous PEB particles was carried out by using water-oil homogenization method, where water was used as a dispersing medium. Epoxy resin which is low cost and non-toxic was used as matrix phase which can be easily disposed in order to minimize the impact on environment. Among the various different types of thermosetting resins available commercially, epoxy resin was found to be most compatible material to produce porous particles are due to its high strength and modulus as well as the low shrinkage (dimensional stability) which makes it easy to be processed into smaller particles (Tsujioka, 2009).

Bentonite clay was used due to it impermeability to water, and has a great water binding ability, which allows better dispersion of bentonite in water during homogenization. Besides, bentonite clay consist no additives, non-toxic and no chemical contains which makes it safe, environmental friendly and to be utilized in various applications. Polyamine hardener was used as curing agent for epoxy resin, where the mixing ratio are not critical and provide good toughness which makes it more compatible for epoxy resin. The combination of these particles with different types of properties results in more reliable and good performance PEB particles with enhanced

properties (Fekete, et al., 2014).

Upon the mixing and homogenization process, the developed porous particles were characterized to determine the particle size, particles size distribution, density and surface morphology. Characterization of PEB is very important in proving the development of stable porous particles using epoxy and bentonite clay. Besides that, the effect of mixture/water ratio and bentonite loading on the development or characteristics of developed porous particles were determined.

1.3 PROBLEM STATEMENT

(i) High technology and cost of processing of porous particles.

Generally, porous particle provides excellent properties such kinetic properties make it very useful in many applications such as gas adsorption, die removal and as filler in polymer nanocomposite. Unfortunately, the processing method or separation processes of porous particles are very crucial while the processes are typically expensive. There were many different types of porous particles that can be produce with some limitations which leads to expensive and complex production. In this case, porous tungsten trioxide (WO_3) was produced by using surfactants as templates and most of the times, the surfactant cannot be removed completely which may present as impurities or unwanted material that respectively affects the material performance. In order to overcome this problem, bubble gas was used instead of surfactants to form pores. Even though, this method are effective, the processing procedure is complicated which needs electrochemical device which is costly and limiting scaling-up of production. Hence in this research, a simple and low cost method was introduced which are mixing and homogenization of epoxy and bentonite clay in the presence of polyamine hardener in water medium to produce porous particles. Mixing using

mechanical stirrer and homogenization of the mixture in water medium are very simple technology and cost effective as compared to many commercially available processing of porous particles (J, et al., 2012).

(ii) Limited functionality or application of porous particles.

The complex and costly processes of porous particles production results in its limited application in a few industries such as the use of mesoporous particles in gas adsorption and microporous particle as fillers in polymer composite. In this case, the development to produce porous particles takes into consideration, because most of the porous particles undergo some modification to alter the pore sizes by using different types of templates such as carbon sphere. These templates sometimes affect the porous particles performance, for an example, mesoporous particles required carbon sphere as template but the presence of the carbon could affect or damage the catalytic performance, so the removal of carbon is required. This lead to the introduction of new method such as hydrothermal processes instead of template, where the processes of porous particles to become more complicated and expensive (raw, 2015).

(iii) Some porous particles are chemically reactive, toxic or not environmentally friendly.

The processing of porous particles can be highly risky or hazardous either to the human and environment. Generally, most of the porous particles such as activated carbon and porous carbon which are used commercially are chemically reactive and toxic. Recently, the application of nano sized particles plays a vital role in many industries because of their unique physico-chemical properties such surface morphology. Unfortunately, the nano sized especially in crystalline forms porous particles may lead to possible hazard to human's health, including

the ability to penetrate into the lung and its systemic circulation. Moreover, the toxicological properties of nano particles may lead to perturbation of intracellular mechanism of cells in human body. Meanwhile, the large scale processing of porous particles such as activated carbon which in the form of powder in industry may lead to dust pollution to surrounding environment. Although the nano particles technology may provide some benefits to the industry, their toxicological properties should be carefully addressed (Corporation, 2014).

1.4 OBJECTIVES OF STUDY

The aims of this project are:

- i. To produce porous epoxy/bentonite clay particles (PEB) through water-oil homogenization method.
- ii. To observe the effect of epoxy/bentonite/hardener mixture to water ratio on the development of PEB particles at constant bentonite loading.
- iii. To develop PEB particles at various bentonite loading through water-oil homogenization method at optimum mixture: water ratio.
- iv. To characterize the produced porous epoxy/bentonite clay particles for their particle size and distribution, density, microstructure and morphological properties.

1.5 PROJECT FLOW

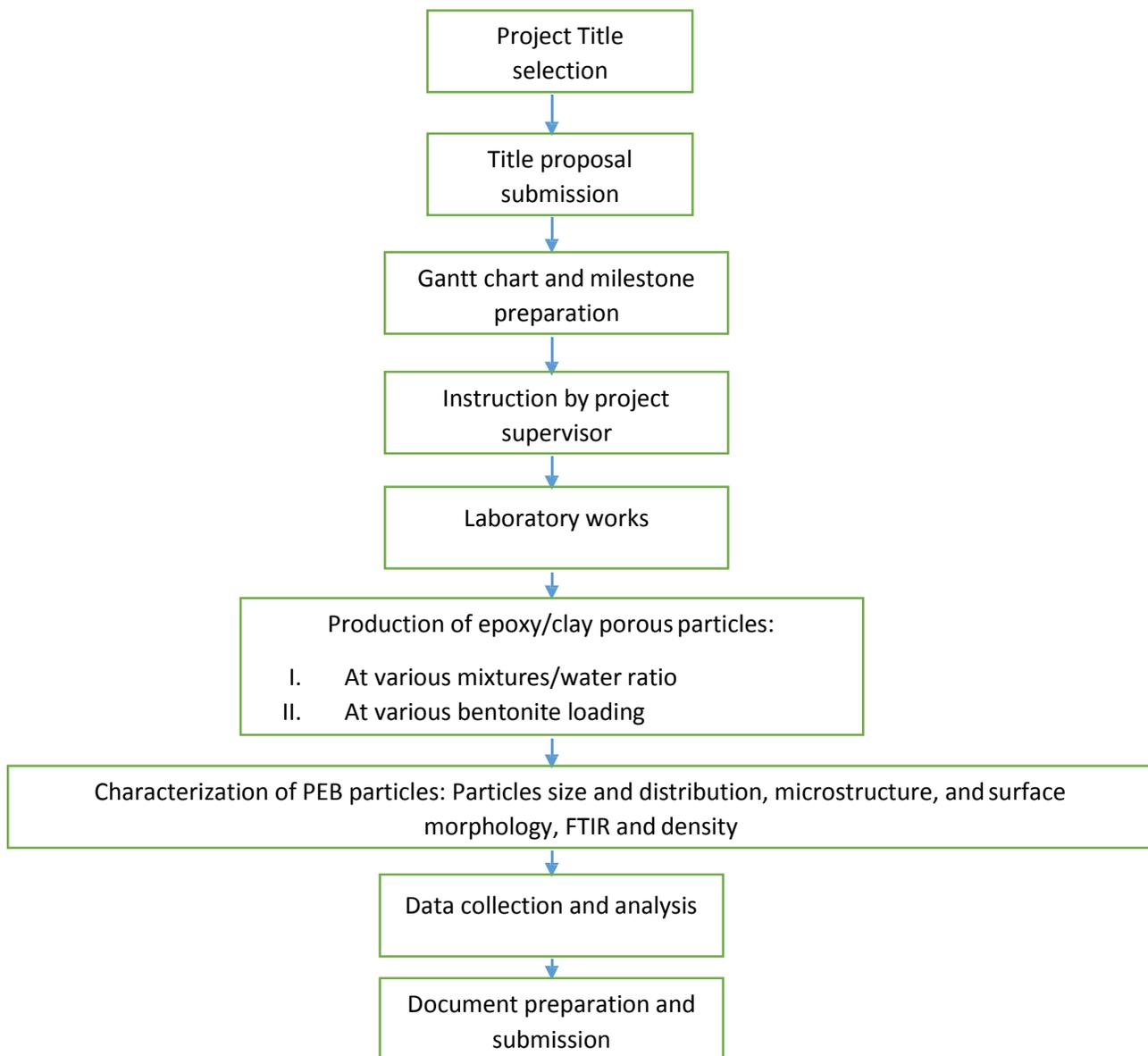


Figure 1.1 Project flow chart

CHAPTER 2

LITERATURE REVIEW

2.1 Epoxy resin

2.1.1 Introduction

Epoxy resin is the class of thermoset polymeric materials. Epoxy resin also represents highest performance resin among other resins which currently available at this time. The general nomenclature of epoxy involves the chemical bond which consists of single oxygen atom which is bonded with two atom of carbon. Epoxy resin available in wide grades varies from low viscosity liquid to high melting solids. Epoxy resin is used exclusively in many industries due to its unique combination of properties compare to other resins. Figure 2.1 shows the general nomenclature of epoxy group (AB, 2015).

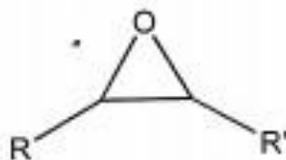


Figure 2.1: General nomenclature of epoxy group

Epoxy is formed from an extended molecular chain structure such as vinylester with reactive sites at both end. Within synthetic resin, the reactive sites are shaped by the epoxy group rather than organic compound group. The absence of organic compound

group implies as the synthetic resin has notably sensible resistance towards water. The molecule of epoxy additionally consists of two ring group at the center that are prepared to significantly absorbed the stresses of thermal and mechanical which are higher than the linear group while offer the synthetic resin with excellent toughness and resistant properties. (Cripps & Gurit, 2015).

The Figure 2.2 shows the idealized nomenclature of an epoxy. Organic compound groups absence at intervals of the molecular chain.

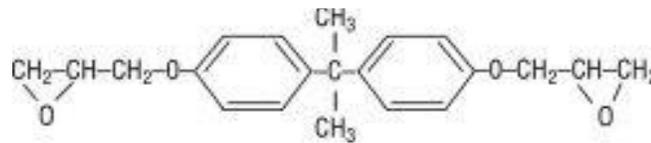


Figure 2.2: Idealized nomenclature of epoxy

The curing process of epoxies is different compared to polyester resin where the curing of epoxies done by hardener instead of catalyst. The most commonly used curing agent is polyamine, the chemistry of the following reaction implies that the epoxy binding to each amine site in order to form molecular structure with three dimensional. Commonly primary or secondary amine group will react with epoxy molecules (Cripps & Gurit, 2015).

The mixing ratio of epoxy and amine molecules should be properly determined in order to produce homogenous mixture. If the mixing takes place not according to proper mixing ratio, hence some of the hardener or organic compounds were unreacted and settled around the matrix which tend to affect the physicochemical properties of cured product.

2.1.2 Properties and Application of epoxy resin

Thermosetting polymers, such as epoxy resin is famous and widely used as high performance materials in many industries which involving polymers. Epoxy resin commonly used as polymer composite and adhesive in industries. Besides, the poor impact resistance of epoxy, limits it's usage in many applications. The poor impact resistance of epoxy results from high crosslinking density of it. Many researcher carried out studies and research on the modification of highly cross-linked epoxy in order to increase the impact resistance of epoxy (Lan, et al., 2011).

Currently more than a total of fifty unlike substances fulfills the characterization for an epoxy resin, with the hundreds of different kind of hardener, its straight forward to know that where the epoxy resin properties is changed to fulfill the foremost different necessities. Even so, some basic properties are continuously present (AB, 2015).

Application of advance composite usually used the epoxy resin as the polymer matrix along with reinforced fibers. The unique properties of epoxy resin such as specific strength, chemical resistance and thermal stability makes its most compatible adhesion to embedded fiber. (Zhou, et al., 2008). One of the prominent properties of epoxy resin is the ability to stick and adhere to mostly all substrate in resin form, because the presence of hydroxyl group and ether bond which makes the contact between epoxy and its substrate are not disturbed by the tensions. Hence, the surface tension of epoxy playing a vital role by providing surface energy for many materials (AB, 2015).

In general, epoxy resin most applicable in surface treating concrete because epoxy highly resistant towards alkaline. Epoxy have very slight shrinkage throughout hardening of the concrete surface which results in better orientation throughout the hardener. No alternative hard plastic will has better mechanical properties compared to epoxy resin, it's mostly due to the lowest shrinkage that built-in tensions are avoided (dow, 2009).

In general, epoxy highly resist toward vapor transmission. By using special technique, epoxy created diffusion where it's more applicable to use as adhesion for wet concrete. Epoxy resin is a good electrical insulators and combined with it's highly resist towards chemical and moisture which makes epoxy applicable in electronic manufacturing as well as the embedment of transformers.

The properties of epoxy resin can be modify easily according to the application needed. The curing agent such as amine as well as other alternative substance can significantly alter the properties of epoxy. The epoxy can be find in the form of liquid with low viscosity to solid with high melting point. Hence the physical state of epoxy makes it's more compatible in many major industries where epoxy can combine properties with other materials which leads to high application in plastic industries. (europe, 2015).

Moreover, the admirable adhesive properties of epoxy leads it's become more popular in electronic industries (Cheng, et al., 2009). Even though, the epoxy is an electrical insulators, the properties of epoxy can be changes according to its application to make it compatible for specific application, for an example epoxy filled with silver can be used for electrical conduction. (europe, 2015)

The epoxy can be classified into two groups which are glycidyl epoxies and non.glycidyl epoxies. This two types epoxies varies in application according to its properties. The glycidyl epoxies consist of aromatic ring which makes it to be highly resist towards ultraviolet radiation which makes its compatible for outdoor application. Diglycidylether of bisphenol A (DGEBA) and 3, 4-Epoxy cyclohexyl-3'4'-epoxycyclohexane carboxylate (ECC) are the most common monomers that consists in both glycidyl and non-glycidyl epoxies. The nomenclature of both monomer are given in Figure 2.3 (a) and (b), respectively. Generally, the DGEBA oligomers obtained by the synthesis of DGEBA based resins through the addition of bisphenol A and epichlorohydrine. The Figure 2.3 (c) shows the nomenclature of DGEBA oligomer.

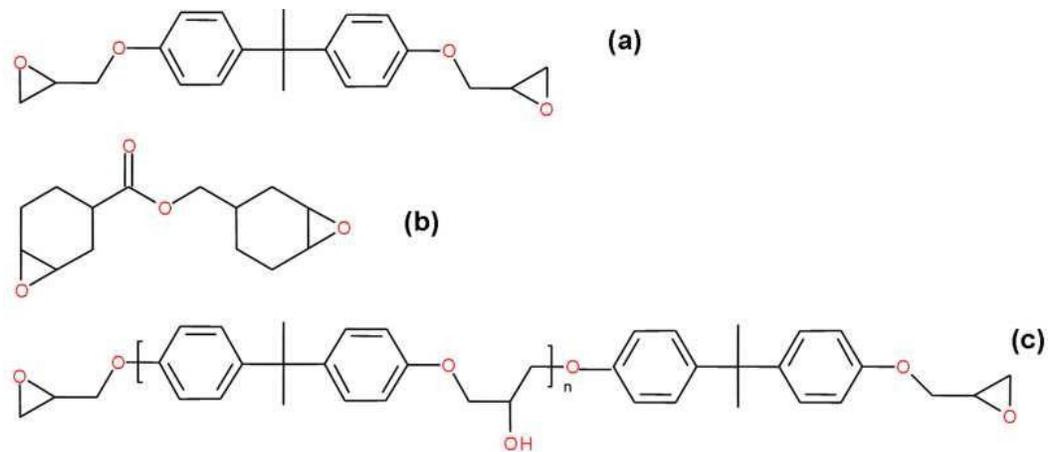


Figure 2.3 a) Diglycidylether of bisphenol A (DGEBA); b) 3, 4-Epoxy cyclohexylmethyl-3, 4-epoxycyclohexane carboxylate (ECC); c) DGEBA oligomer (González, et al., 2012).

The high purity and low oligomer content of diglycidyl ether of bisphenol A promises uniform performance, exceptionally low viscosity and may offer improved elevated temperature properties over normal epoxy resins. This type of resin grade is principally utilized in filament winding, electrical laminates and encapsulation applications (dow, n.d.)

Frequently, epoxy resins are blended, filled, or changed with reactive and non-reactive components. It's then necessary to regulate the concentration of the solidification agent to cure solely the portion of the mix that's reactive, for example, the resins and any reactive diluent present. Chemically changed liquid epoxy without diluents, this type of resin offers good flow and gloss and might be developed for construction industry. The following type of epoxy resin is employed in building applications, higher than or below ground, on road and bridge construction, further as underwater applications (dow, 2009).

Moreover, there are some common application of epoxy resin, which are epoxies are usually used in paint industries which can be dried faster and provide excellent protective layers which is also tough. In highly technical application, epoxies are used for embedding samples for their use under electron microscope. It is not limited for

technical application; while the epoxies as wonderful composites, it's usually used in the manufacturing of various molds and casts laminates and plastic toolings. In addition epoxies also play a vital role in aerospace industry as a structural matrix material. Meanwhile epoxies known with their versatile nature, where artist have also used epoxies as painting medium by mixing with pigments to obtain colors (Corporation, 2015).

2.1.3 Epoxy hardeners and curing system

Epoxy can be classified as thermosetting material. Reaction of epoxy always involves curing agent or hardener to increases the crosslinking of epoxy in order to make the epoxy harder. The curing agent can be classifying into two major class. The first class usually employs amine compounds which are primary and secondary amines. The second class usually employs anhydrides and carboxylic acids. The formulation of epoxy/hardener proportion and the selection of epoxy resin and types of curing agent must select carefully in order to achieve desired properties of final products (Pham, 2011).

Epoxy hardener with amine compound is one the prominent and broadly used curing agent in industry. As mentioned earlier, first class of amine curing agent comes with two types of nomenclature which are primary and secondary amines which both are extremely reactive with epoxies. According to the substituents of these amines, it can be classified into three groups which are aliphatic, cycloaliphatic and aromatic amines. This substituents can highly effect the properties of epoxy such as thermal and chemical resistance properties. Understanding of substituents effect on the production of thermoset materials playing a vital role on controlling the quality of final products (Aziz, 2010).

Meanwhile, understanding the chemistry of curing agent with epoxies is very important because it can merely influence the reactivity of epoxy as well as its viscosity. Basically, the amine consists of nitrogen where the epoxy group will react to hydrogen

atom which bonded with nitrogen atom hence the formation of hydroxyl group increases whenever the epoxy group opened. Cross linking occurs when the hydrogen in the amine group inter linking with large molecule in epoxy group where the amine molecules should at have at least two or more active amine hydrogen which makes the cross linking chain becomes more longer which relatively increases the viscosity (Aziz, 2010).

The molecular weight of epoxy will be relatively high when the gap between the chains is bigger, this will results in lower crosslink density which makes the epoxy more flexible where can resist more impact and high resistance towards chemical. Besides that, the understanding of amine value which indicates the amount of active amine hydrogen of curing agent is relatively important for determination of amount of nitrogen in the final products where it can also use for quality assurance test for the amine based curing agents. Figure 2.4 shows the mechanism of epoxy with amine based curing agent (Pham, 2011).

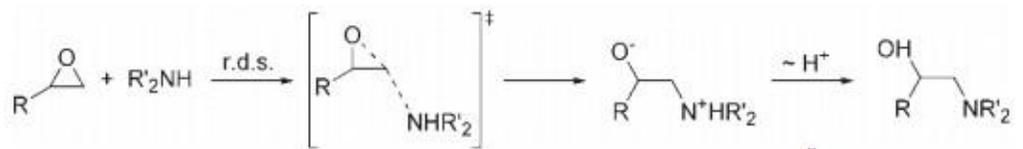


Figure 2.4: General mechanism of epoxy with amine based curing agent

2.2 Porous particles

2.2.1 Introduction

Porous particles, particularly those that are spherical in shape, are used in various applications for many years. Porous particles can be classified as macroporous, mesoporous, and microporous based on the dimensions of the pores, severally; more than 50 nm, in between 50-2 nm and less than 2nm (Fekete, et al., 2014).

The two main characteristics that distinguish porous particles are porous nature and better crosslinking degree, as compared to from gel-type polymer compound particles. Differences in these features, its lead to different characteristics such as acceptance of high solvent and large surface area with different polarity. Some performance such as chemical nature and size are alternative options that the porous particles share with non-porous particles (Dolan, 2015).

The polymer porous particles usually produced from heterogeneous polymerizations by using two immiscible liquids, where the heterogeneous mixture is formed. There is some important techniques available to produced polymer porous particles such as precipitation, dispersion and suspension (Gokmen & Prez, 2012).

Principally, the porous particles produced by low energy input and low cost of homogenization or emulsion process. Organic solvent and water usually involves in this process. Emulsion can be categorized into two which are oil-water emulsion and water-oil emulsion. For oil-water emulsion, the oil will act as external phase while the water acts as dispersed phase, for water-oil emulsion is vice versa. The homogenization mixtures with presence of block copolymer were merely blended. Studies shows that, the technique and preparation condition significantly affect the production of porous particles (Takami & Murakami, 2014).

2.2.2 Properties and application of porous particles

Porous particles are famous and show great interest in the field of nanotechnology and engineering. Porous particles are also broadly employed in many applications such as tissue engineering, drug delivery and bio molecular analysis due to its particles size and distribution, and as well as the surface microstructure. In the application that involves gas analysis, porous particles are employed as fixed phase in gas chromatography. Porous particles with manageable surface morphology are also extremely preferred as a beginning material for particle exchangers, and as chemical catalysts. Furthermore, the porous particles used as drug delivery carriers in drug delivery application and also employed as staging in tissue engineering. (Wu & Clark, 2007)

The capabilities of physical absorption, electrochemical absorption of the porous particles leads to applications such as scavenging. Scavenging is an alternative field where porous polymer is efficiently used to remove impurities from organic media. Scavenger resins preferably retain chemical group by selectively react and thus take away unwanted compounds from a combination. The swelling properties of porous particles makes its compatible to use to remove unwanted species from organic media or from water. The absence of porosity in porous particles makes its favorable in gas absorption application (Guillarme & Fekete, 2013).

In addition, the porous particles are employed as solid phase extraction application, where small particles are packed accordingly in cartridge which absorbed the hydrophobic solutes from an analyte. In the second stage of the process, by using organic liquid the solid waste is washed from the sorbent. Meanwhile, developed solutes are prepared for the analysis, where the interaction time will be very short thus requires high absorption activity from particles which act as sorbent. Taking this into consideration, a porous particles with hyper cross-linked is needed due to its very high surface area. (Gokmen & Prez, 2012).

The static properties of porous particle such as stiffness and strength are rely not solely on the number of porosity, but also on how the particles are distributed and other characteristics such as the pore structure and size. Porous material such as ceramics, foams like metal or polymeric and molded metals are being applied in light-weight applications. For this material the porosity is also considered into the material. The porosity is described by volumetrical quantity, size and form of pores, cellular structure, and is often homogeneous and equally distributed. Designing components from such materials seems to be comparatively straight-forward once the particle properties are established, and if native variations within the stress-strain field owing to the pores will be ignored (Hardin & Beckermann, 2006)

Porous particles have become progressively widespread in many high impact sectors owing to their light-weight structure, mechanical endurance, and biomimetic properties. Recent progresses in refined material design, development, and its characterization methods have sustained methodologies to modify their properties based on its application (Corporation, 2014).

The technical challenge to comprise and forecast the response of those materials under external stimuli has dominated the interest of engineers. As porous structures exhibit material properties of huge sensible importance, and so the capability to consistently and effectively characterize these structures could be a perspective of good importance to multiple scientific fields. Porous particles are usually found in natural structures such as wood, bone and sponge and also similar with robust and lightweight structures. Human made synthetic porous particles are mainly from polymers and ceramics which have been widely capitalized due to the distinctive combinations of physical and mechanical properties offered by porous materials. Interest are mainly focused on the exploitation of its ability to be incorporated into robust, and stiff light-weight structures particularly due to their ability to soak up energy, vibration and sound (Gupta, 2015).

2.2.3 Current development in porous particles

Presently, the development of porous particles is playing a vital role in current issues such as energy and micro reactor. Application which related to energy, the hydrogen gas is considered as greener gas compared to other fossil fuels for coming future. A massive quantity of analysis is presently being dedicated to metal-organic frameworks as where hydrogen gas used as storage materials (Fekete, et al., 2014).

Micro-porous polymer based particles also playing a vital role in energy based application. Other applications which related to energy such as sequestration and carbon capture which also needed the porous polymer particles which will be very helpful in the same field. Recently, the porous hydrogel particles are developed in bioengineering field where, the porous employed as scaffolds in tissue engineering. Porous particles have played a significant role in micro-sensors application such as multicolor fluorescent and permeation-selective micro-beads which allow instantaneous sensing of pH, temperature and oxygen, besides, the polymer microsphere are greatly employed as a fluorescent sensor in aqueous media (Hogan, 2015).

Besides, micro-reactors are also getting a high remarks due to its ability to reduce impact on the environment and cheap processing. The polymer porous particles is used to capture harmful particles or impurities by absorbing organic species and convert them into non-toxic substance through enzymes or immobilized catalyst. If the porous particle is intended with stimuli-responsive characteristics it can also increase application of porous particles in many other applications. A trigger can be an addition of chemicals, rise in temperature, changes in pH value, or the external magnetic and electric fields (Gokmen & Prez, 2012).

2.2.3.1 Water/oil homogenization/emulsion

Generally, homogenization or emulsion known as mixing of two types of unmixable liquids. In recent days, production of porous particles carried out through water/oil homogenization method which is cheapest and best method to produce it. Usually water/oil homogenization method involves two phases which are dispersion medium and dispersed phase, for water/oil emulsion, the water will act as dispersed phase well the oil act as the dispersion medium or external phase. During emulsion, the small droplets are formed by shear of dispersion and usually will coated by emulsifier. The droplets throw into disordered in three different types of flow, which are simple shear flow, rotational flow and elongational flow where sometimes the profile used to be very complex depends on the geometry of homogenizer (Cassiday, 2014).

During homogenization, the droplets size can be affected when the emulsifier concentration reach an optimum level, where the energy input from the homogenizer is highly affect the droplets size. Besides that, the amount of emulsifier that needed for emulsion are depends on the energy input that used to produce emulsion. Some of the emulsifier can be categorized into two group which are hydrophobic and hydrophilic, where the concentration of water and oil of the emulsifier classified it as hydrophobic or hydrophilic. If the emulsifier contain of 35% of oil soluble and 65% of water soluble then the homogenization to be prepared is the oil/water emulsion where the oil formed as small droplets while the water acts as a dispersion medium (molecularrecipes.com, 2014).

2.3 Epoxy/clay blending porous particles

2.3.1 Introduction

Epoxy based nanocomposite system has been remarked as a most famous among the polymers layered silicate or clay nanocomposites, because of their numerous application in many field due to the ease of processing. The properties and structures of epoxy/clay particle depends on the curing agent used as well as the clay modifiers and also the processing method.

The most common curing agent used in the epoxy based system is amine and anhydride. Different type of curing agent gives a different morphology and properties to the epoxy based system. The selection of curing agent must be considered very carefully because curing agent is a part of network structures. Moreover it's also affects the viscosity and reactivity of formulation. Besides that, the curing agent also determines the formation of type of chemical bond formed and cross-link junction formed. Furthermore, the stoichiometric ratio of epoxy/curing agent have major effect on the performance and network structures produced (Aziz, 2010).

Besides that, the processing technique of epoxy/clay system also influences the clay morphology. Mechanically stirred, ultrasonic homogenizer and high shear mixing are most common method to produces the dispersion by dispersed clay into epoxy matrix. According to the processing method, establish that when rising in clay concentration, the modulus of nanocomposite will also increase radically. The addition of clay particles into pure epoxy will relatively decreases the tensile strength of nanocomposite. Even tough with small quantity of layered silicate, the epoxy/clay nanocomposite showed a great improvement in thermal mechanical properties such as heat resistance, high modulus, increased strength, and also reduces coefficient of the thermal expansion. The enhancement is achieved due to the interfacial interaction between the epoxy matrix and silicate and also the aspect ratio of clay particles. (Azeeza, et al., 2013).

2.3.2 Epoxy based porous particles

Among other thermosetting materials, epoxy resin provide some special characteristics in term of chemical properties, by which the absence of byproducts or volatile materials during curing reaction and also the low shrinkage upon curing are very beneficial. Besides, epoxy resins can be cured in wide range of temperature with controlled degree of cross linking (Azeeza, et al., 2013).

Epoxies usually used in polymer industry in different ways, for an example the combination of epoxies with glass fiber in order to produce high strength composite which provides sufficient mechanical, chemical and electrical properties. Moreover epoxies also used in electronics industry for casting of numerous electronics component and in powder coatings in metal substrate. A foremost outlet of epoxy is also including adhesive and protective coatings in appliances. In the recent development epoxies based nanocomposite a successful replacement of micrometer size filler composite (Companies, 2015).

In the synthesis of epoxy/clay nanocomposite, epoxies resin consider as the most flexible resin system with optimal glass transition temperature by using diglycidyl ether of biphenol A (DGEBA) resin. Meanwhile, high performance application such as aircraft, vessel, and tank and pipes will be required resin system with higher mechanical properties and high glass transition temperature which based on trifunctionaltriglycidyl p-aminophenol (TGAP) (Kotsilkova, 2007).Moreover, the addition of organoclay can significantly improve the stiffness and strength of the epoxy resin system to enhance its functionality and also provide smart alternative to higher concentration of more usually used filler and as well as to fiber reinforced plastics.

Initially, the epoxy based porous particles are prepared by the dispersion of efficient amount of nanoparticles into the unreactive part of the resin system and mixed to reactive part of the resin and eventually cured. The processing method is decided based on the chemical and physical properties of the nanoparticles and epoxy resin itself. Commonly, metal oxide based nanoparticles are used as received. Besides that, some

nano particles with high density and highly entangled such as Tungsten trioxide (WO_3) needs special modification in order to obtain enhanced properties (Rangari, 2011).

2.3.3 Clay based porous particle

Clays usually are aluminosilicates and hydrous silicate which typically containing oxygen, magnesium, hydroxyl or silicon with numerous associated cations where this hydroxyl group are arranged into two dimensional sheets. The 1nm thick silicate layers as the structural frame work indicates the clay particles as silicate layer, where the alumina and silica sheets covered by the silicates layer which combines together to different extent (materials, 2015).

In general, clays can be categorized as hydrophilic in nature which has high tendency to swell or interact with water. Some modification must be made on the clay surface to make compatible with organic polymer. Phosphonium ions and ammonium ions which also known as organic cations were used as organic modifier for clay. The modification made involves the exchange of inter layer inorganic cations with organic salts. This adjustment will results in the expansion of interlayer space as well as increase the spacing to certain range. Thus, the changes provide the polymer and its precursor an efficient diffusion into the interlayer space. Montmorillonite type's clay is the famous among other types of clay where it's used to prepare the polymer clay nanocomposite. The montmorillonite owing a special consideration between the smectite group where its capability to provide widespread inter layer swelling and expansion this is because of their peculiar structure (Kiliaris & Papaspyrides, 2010).

Moreover, preparation of clay based nanocomposite usually done by using three most common method which are solution casting, melt intercalation and In-situ polymerization. According to In-situ polymerization where the epoxy and liquid monomers are interpolated in the clay layers as well as polymerize within the clay layers which results the interlayer distance to expand, where heat is used to initiates this polymerization by using appropriate initiator. Besides, this method provides a polymerization route and appropriate reagents to be selected in order to produce a better

similarities or affinity between the clay and polymer. Meanwhile the mixing of clay particles with matrix in molten state are involves in Melt intercalation polymerization, the diffusion between polymer and clay occurs when the clay layer has suitable affinity between the polymer matrixes. Finally, solution casting method, where the polymer clay nanocomposite is prepared by using solution such as chloroform and as well as water. During this polymerization process, where the polymer acts as medium which dissolved as the clay particles dispersed. As solvent molecules interchange the polymer and clay dispersed solution will intercalated within the clay layers (Nguyen & Baird, 2006).

Meanwhile, the polymer which intercalated will settled on the clay layers even though the solvent is removed. Through this method, it's stated that the entropy will rise by the, desorption of solvent molecules where the this desorption also acts as driving force between solution and intercalated polymer (Azeeza, et al., 2013).The nomenclature of nanocomposite can be determined from the dispersion degree of the clay particles in polymer matrix. Common types of morphology can be obtain from the polymer matrix and clay interaction which are intercalated and exfoliated. Figure 2.5 shows more detail about those types of morphology.



Figure 2.5 types of morphologies structure

As shown in the diagram in Figure 2.5, the intercalated structure is obtained when the diffusion occurs between the polymer chain and silicate layers, which leads to the formation of inorganic layers and different types of polymer matrix layers. Meanwhile, the formation of exfoliated occurs during separation of silicate layers and randomly dispersed all over the polymer matrix. By comparison, the exfoliated type is more desirable because they provide more improved properties (Nguyen & Baird, 2006).

CHAPTER 3

METHODOLOGY

3.1 Materials

3.1.1 Epoxy resin

Epoxy resin used in this research work is the clear epoxy resin of liquid epichlorohydrin and bisphenol A, which is supplied by the Zarm Scientific & Supplies Sdn Bhd (Company No. 841571-D). Several properties of the clear epoxy resin are shown in the Table 3.1 below.

Table 3.1 Properties of clear epoxy resin

Properties	Value
Epoxide Weight (g/eq)	182-193
Epichlorohydrin (ppm)	5.0 Max
Density at 25 °C (g/ml)	1.167
Viscosity at 25 °C (mPa*s)	11005-14005
Water Content (ppm)	700 Max.
Color, Pt-Co	75 Max
Hydrolyzable C1 (ppm)	0-500

3.1.2 Amine Hardener

Clear epoxy hardener was used as curing agent with an amine value of 272 (mg KOH gm⁻¹) and also supplied by the Zarm Scientific & Supplies Sdn Bhd (Company No. 841571-D). The properties of this material are shown in Table 3.2.

Table 3.2 Properties of Epoxy Hardener Clear

Properties	Value
Colour(Gardner)	1
Appearance	Clear liquid
Specific gravity@77degF	1.03
Flash point (closed cup) (deg F)	205
Amine value (mgKOH/g)	272
Viscosity , cps	450

3.1.3 Bentonite Clay

Bentonite clay was purchased from Ipoh Ceramics (M) Sdn. Bhd. Analysis on the physical properties of bentonite clay are shown in Table 3.3 as obtained from characterization using Malvern Mastersizer, particle size analysis. The density of raw bentonite was determined to be 2.513 gcm⁻³. Bentonite clay was pre-dried in vacuum oven of 80°C for 24 hours prior to the density measurement using gas pycnometer (Accupyc 1330).

Table 3.3 Physical properties of Bentonite Clay

Properties	Value
Bulk Density (g/cm ³)	2.5
Average Particle Size (µm)	2.54
Specific Surface Area (m ² /g)	2.677

3.2 Preparation of porous epoxy/bentonite (PEB) particles

3.2.1 Stages of preparation

Phase I: Mixing of epoxy resin, bentonite and epoxy hardener using mechanical stirrer

Phase II: Homogenization of mixture through water based method

Phase III: Curing of homogenized emulsion in oven

Phase IV: Crushing and sieving of cured PEB particles

The overall process of PEB preparation are simplified in flow chart as shown in Figure 3.1.

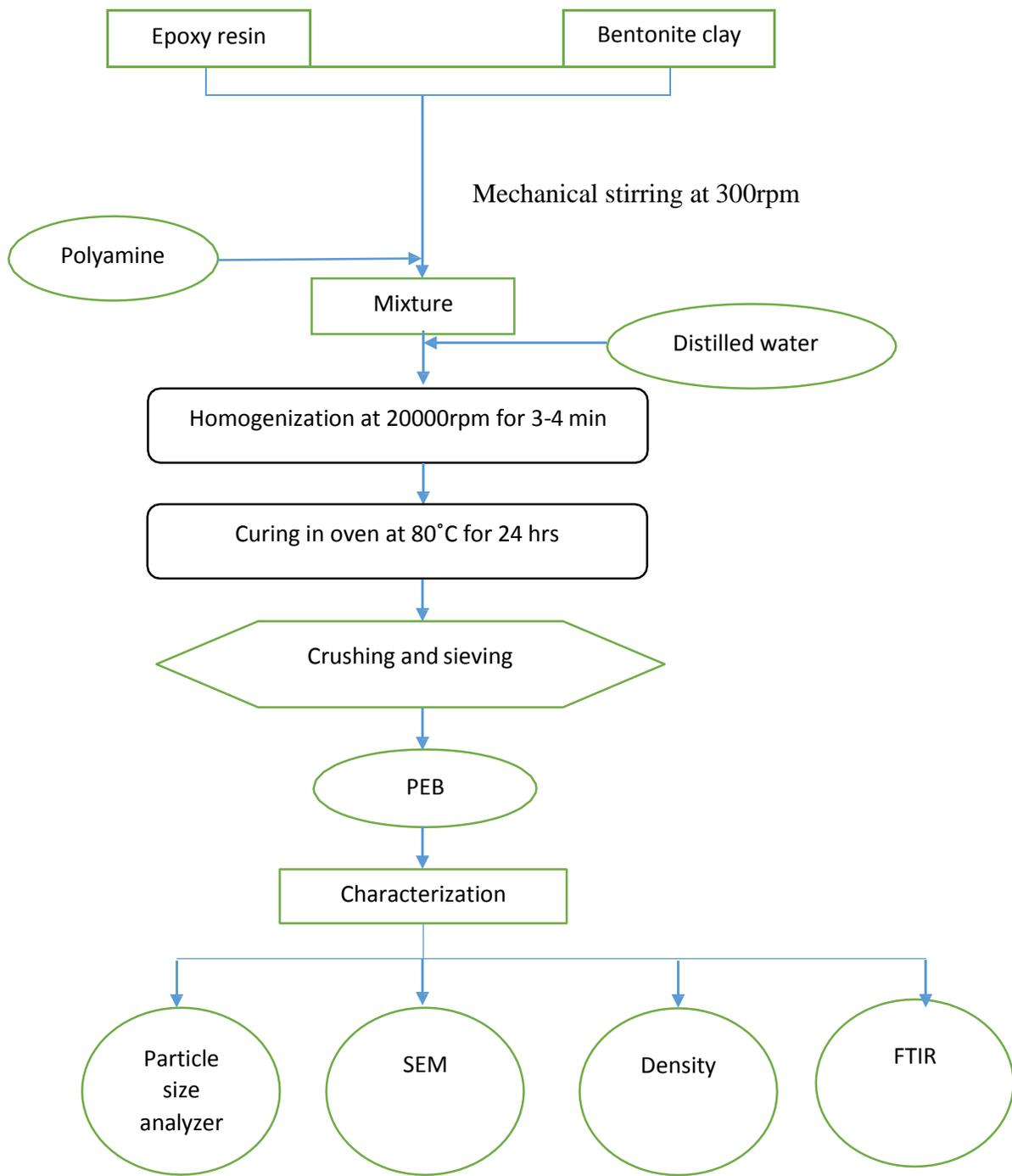


Figure 3.1: Process flow on PEB production

3.2.2 Effect of Epoxy: Hardener: Bentonite mixture to water ratio

PEB particles was developed according to same stages as in Section 3.2.1. The effect of mixture to water ratio was studied and the physical observation on the development of epoxy droplets/particles were observed. The composition formulation for the mixing/homogenization are as shown in Table 3.4.

Table 3.4: Ratio of PEB mixture to water

Mixture (g)	Water (g)	Ratio / (g)
60	140	1
60	130	1
60	120	1:2
60	110	1

The bentonite loading was kept constant at 10wt% and the physical development of PEB particles was observed at different mixture: water ratio. Characterization of the produced particles was carried out.

3.2.3 Effects of bentonite loading on the PEB particles production

Steps on the preparation of PEB particles are similar as in section 3.2.1. The effect of bentonite loading on development of PEB particles were studied. The composition formulation for the preparation of PEB particles is as in Table 3.5. The ratio of mixture: water is kept constant at 1:2 and the effect of various bentonite loading on the formation of PEB particles were observed. Characterization on the produced particles were carried out.

Table 3.5: Formulation for PEB production with variation in Bentonite loading.

Epoxy,(g)	Hardener,(g)	Bentonite (wt%), (g)	Water, (g)
40	20	10% (6)	132
		20% (12)	144
		30%(18)	156
		40%(24)	168
		50%(30)	180

3.3 Characterization of PEB particles

3.3.1 Physical Observation on the particles formation

Physical observation was carried out to observe whether epoxy/bentonite mixture formed the particles or have phase separated during the homogenization and curing process. This observation is important to determine the optimum mixture: water ratio to produce the particles. The optimum mixture: water ratio were than used at second stage of PEB particles production at various bentonite loading.

3.3.2 Density

The density testing is carried out by using Gas pycnometer (Accupyc 1330). This device uses some gas displacement to measure the density of solids or other such as porous, non-porous and monolithic particles. This test was carried out on all the five PEB powder samples to determine their density and compared to study, how the different percentage of bentonite clay of the PEB particles affect the density.

Density of raw bentonite and produced PEB particles were measured through this method.

3.3.3 Particle Size Analysis

The particles size analysis was carried out using Mastersizer 2000, Hydro2000 MU (A). The purpose is to study the variation on the particle size and particle size distribution as the effect of different bentonite loading on the produced PEB particles.

3.3.4 Scanning Electron Microscope (SEM) analysis

FESEM-JEOL 6701-F scanning electron microscope was used to provide images of samples in detail by using electron beam. The PEB particles were sputter coated with titanium particles prior to observation. The acceleration voltage used for this scan was 20Kv. The SEM was used to observe the surface morphology and shape of PEB particles samples with two different magnifications which were (50X, and 100X). The presence of porosity on the PEB particles can be proven through this study.

3.3.5 FTIR

PEB particles, raw bentonite and epoxy mixture were subjected to FTIR analysis. The IR spectrum of the raw materials was recorded by using Spectrum RX1 Perkin Elmer analyser. The spectra was recorded from 4000 to 400 cm^{-1} wavelength with 4 scan. The PEB and bentonite powder were prepared using potassium bromide (KBR) standard.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This research is mainly intended to develop a multifunctional porous particle through water-oil homogenization of epoxy and bentonite clay mixture. The idea is to disperse and coat bentonite clay in epoxy resin in the presence of hardener and then to swell the epoxy coated bentonite clay in water at high speed to form swollen epoxy/bentonite clay droplet which can be cured to retain the droplet shape. Removal of water by further heating from the cured swollen epoxy/bentonite clay particles will then produce a porous structure on the surface of cured epoxy/bentonite clay particles. The development of porous particles through this method involves 3 stages mainly the mixing of epoxy resin, bentonite clay and polyamine hardener using mechanical stirrer, followed by homogenization of the mixture in the presence of water at high speed to produce mixture droplet and finally curing of the mixture droplet at elevated temperature (Corporation, 2014).

Bentonite clay with high swelling capacity in water was used as the template for the deposition of epoxy resin and hardener. During the mechanical stirring of the mixture materials, bentonite clay will be coated by epoxy resin and polyamine hardener to certain extends, depending in the ratio of epoxy resin/hardener to bentonite clay. The mixture are then transferred into water and homogenized at very high speed to form droplet. Water are used as the dispersion medium for the formation of epoxy/bentonite clay droplets due to the difference in the nature of epoxy resin which are hydrophobic and water which are hydrophilic. Due to difference in chemical nature, mixture of epoxy/bentonite clay will not dissolve in water instead bentonite clay tend to absorb

water and swell in the presence of epoxy resin and polyamine hardener as the coating in the form of droplet. The homogenized mixture was then cured in oven at 80 °C to allow the epoxy/bentonite clay droplet to cure and retain its structure. Epoxy droplet will be cured in the presence of polyamine hardener while holding the swollen bentonite clay and further drying of the particles at 100°C will result in the removal of water and formation of porous structure on the surface of epoxy/bentonite clay particles. The concept of producing PEB particles are as shown in Figure 4.1.

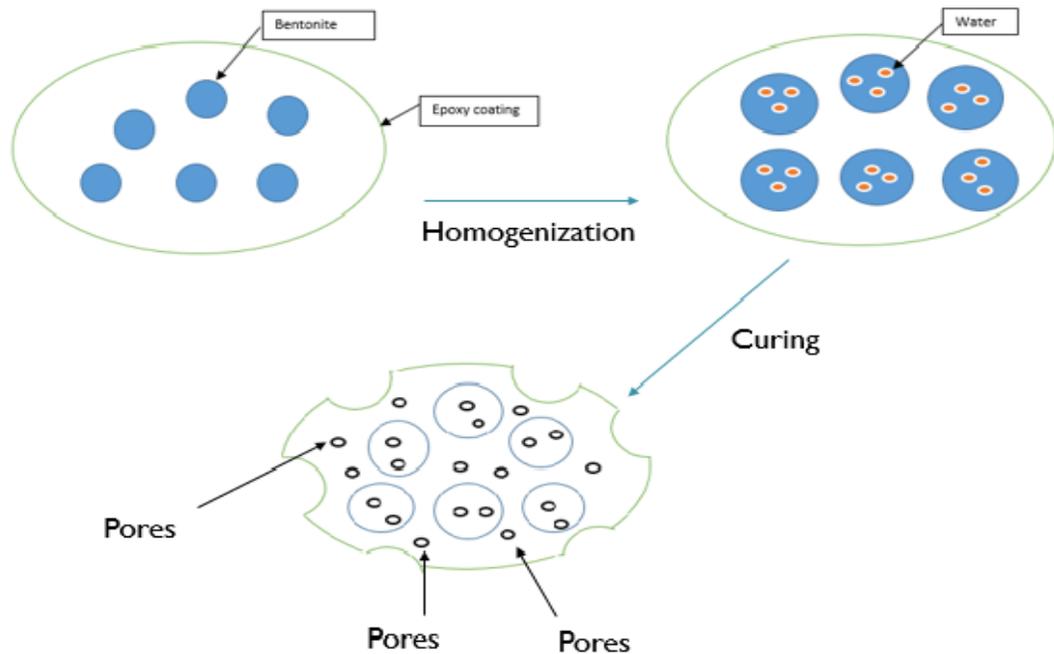


Figure 4.1: Concept of productions of PEB particles

However, the main concern of this research work is to determine the suitable mixture (epoxy/bentonite clay/hardener) to water ratio which allows the formation of mixture droplet in water at high homogenization speed. This is due to the fact that very high or low mixture: water ratio may result in the phase separation in between epoxy/bentonite clay mixture and water instead of forming mixture droplet. Hence the right amount of water is required to allow the formation of mixture droplets in water during homogenization stage. Besides that, bentonite clay loading is also a variable that

need to be considered in the development of porous epoxy/bentonite clay particles. Low loading of bentonite clay may result in the formation of hard epoxy particles due to low water penetration into bentonite clay which are coated with thick resin layer. Whereas very high bentonite clay loading may result in poor wetting of epoxy and the production of imperfect porous epoxy/bentonite clay particles or particles with very low porosity (Gokmen & Prez, 2012).

Similar study on the development of porous hollow epoxy/calcium carbonate particles were studied by Lan, (2009), who successfully produced porous hollow epoxy particles through water-based method as an advanced filler for polymer composite preparation. However, the method required the leaching of calcium carbonate from the produced epoxy/calcium carbonate particles using a strong acid which resulted on the porous hollow structure of the epoxy particles. Lan, (2009) have successfully developed porous hollow epoxy particles at mixture: water ratio of 30:70 and homogenization speed of above 15,000 rotation per minute (rpm). However, this ratio of mixture to water and homogenization speed cannot be used for epoxy/bentonite clay system due to different swelling capacity of bentonite clay as compared to calcium carbonate.

Thus the effect of mixture: water ratio on the development of epoxy/bentonite clay droplet in water was studied on the initial stage of this research work at constant bentonite loading. Followed by the study on the effect of bentonite loading on the characteristics of produced epoxy/bentonite clay particles such as average particle size and particle size distribution, density, morphological properties (particle shape and presence of porosity) through scanning electron microscope and fourier transform infrared analysis. The optimum bentonite clay loading which can be used to develop epoxy/bentonite clay particles with optimum porosity, larger specific surface area, narrow particle size distribution and a smaller average particle size as well as low density were then determined through the characterization of the produced particles (JJ, et al., 2012).

4.2 Effect of mixture: water ratio

Preliminary study was conducted to determine compatible epoxy mixture: water ratio for the production of porous epoxy/clay particles. The observation was made based on cured epoxy mixtures in order to identify on how the water ratio affect the phase separation or the dispersion of epoxy/bentonite/water to produce porous particles as a final outcome. Study was done by using four different epoxy mixture: water ratio weigh in gram which are 60g: 140g, 60g: 130g, 60g: 120g, 60g: 110g, respectively (change in ratio). Figures 4.2 to 4.5 show the effect of respective mixture: water ratio.



Figure 4.2: Epoxy mixture: water ratio, 60g: 110g



Figure 4.3: Epoxy mixture: water ratio, 60g: 120g



Figure 4.4: Epoxy mixture: water ratio, 60g: 130g



Figure 4.5: Epoxy mixture: water ratio, 60g: 140g

As the results obtained, epoxy/bentonite clay particles cannot be produced at mixture: water ratio of 60:110, 60:130 and 60:140, as illustrated in Figures 4.2, 4.4 and 4.5, respectively. At low mixture to water ratio of 60:110 the amount of water is not sufficient to disperse the mixture into small droplets hence the mixture tend to form layer are cured into a hard epoxy/bentonite clay brick. Meanwhile, at higher mixture to water ratio above 60:130, the amount of water are too large that the homogenized droplet tend to be unstable and forms a gel-like structure which was cured into a flexible thin film instead of powder particles. Commonly, the epoxy will be dispersed into smaller droplet to produce emulsion-like solution in water upon homogenization at high speed. However, as the water ratio was increased, the epoxy could not dispersed well in water and tend to phase separated into low density layer on top of water. (oman, 2009)

Figure 4.3 shows the development of powder like spherical particles of epoxy/bentonite clay at mixture: water ratio of 60:120. Thus, from this observation, it can be said that bentonite clay is well coated by epoxy and dispersed uniformly which make the droplets become more stable, and can be cured to form the powdered particles. From the physical observation of produced particles, it can be concluded that the optimum mixture: water ratio that can be used to homogeneously disperse epoxy/bentonite mixture into stable droplet in water is 60g: 120g as shown in Figure 4.3. Hence, this ratio of mixture to water was chosen to further study the effect of bentonite loading on development of porous epoxy/clay particles.

4.3 Effect of Bentonite loading on the porous epoxy/clay particles

4.3.1 Physical Observation

Observation was made on effect of bentonite loading on the development of porous epoxy/clay particles (PEB). Five different sample of porous epoxy/clay particles were produced containing five different bentonite clay loading which are 10wt% (PEB10), 20wt% (PEB20), 30wt% (PEB30), 40wt% (PEB40) and 50wt% (PEB50). The effect of bentonite loading on porous epoxy/clay particles through physical observation on

appearance of the particles was done. Figures 4.6 to 4.11 shows the physical appearance of raw bentonite clay and the produced epoxy/bentonite clay particles at various bentonite loading, respectively.



Figure 4.6: Raw bentonite



Figure 4.7: PEB10



Figure 4.8: PEB20



Figure 4.9: PEB30



Figure 4.10: PEB40



Figure 4.11: PEB50

From Figures 4.6 to 4.11, it can be observed that the trend of physical characteristics of raw bentonite and five different samples changes according to the percentage of bentonite loading. The porous epoxy/clay particles with 10wt % (PEB10) of bentonite loading produced with large particles as compared to PEB20, PEB30, PEB40 and PEB50. Besides, PEB20 also produced large particles but relatively smaller as compared to PEB10. As the percentage of epoxy relatively higher than the bentonite for PEB10 and PEB20, upon homogenization the epoxy disperse slowly and epoxy droplets sufficient enough to well coated most the bentonite particles in order to form porous epoxy/clay particles.

Meanwhile, PEB30 produced in the form of powder with very fine and soft particles. The fine particles of PEB30 can be easily crushed because of high percentage of bentonite as compared to epoxy, where the presence of high percentage of clay particles reduced the tendency of epoxy particles to stick tightly together upon curing. Hence, PEB40 and PEB50 particles produced in the form of powder with much fine particles compared to PEB10, PEB20 and PEB30. It can be observed that the physical characteristics of PEB40 and PEB50 is very similar to raw bentonite that indicates as the bentonite loading increase, the tendency of epoxy particles to wet bentonite clay was decreased and disperse easily upon homogenization and couldn't coated well the bentonite particles upon curing (Alekseeva, et al., 2015).

4.3.2 Scanning Electron Microscope (SEM) Analysis

Characterizations on the surface morphology and shape of the porous epoxy/clay particles (PEB) is done through Scanning Electron Microscope (SEM). Surface morphology and shape of the porous particles are one of the important parameter which can enhance the mechanical properties and alter the total surface area of the particles. Meanwhile the surface morphology characteristics such as porosity and surface void of the particles relatively influence and improved the interfacial bonding where matrix enter on the porosity or surface voids in order to produce interlocking. In this research work, the porosity, surface void and the surface roughness of the porous particles is observed and as well as the effect of bentonite loading on the porosity and shape of the porous epoxy/clay particles. Figures 4.12 and 4.13 show the SEM micrograph of raw bentonite, and PEB particles at the magnification of 50 X and 100 X, respectively (Zielinski & Kettle, 2013).

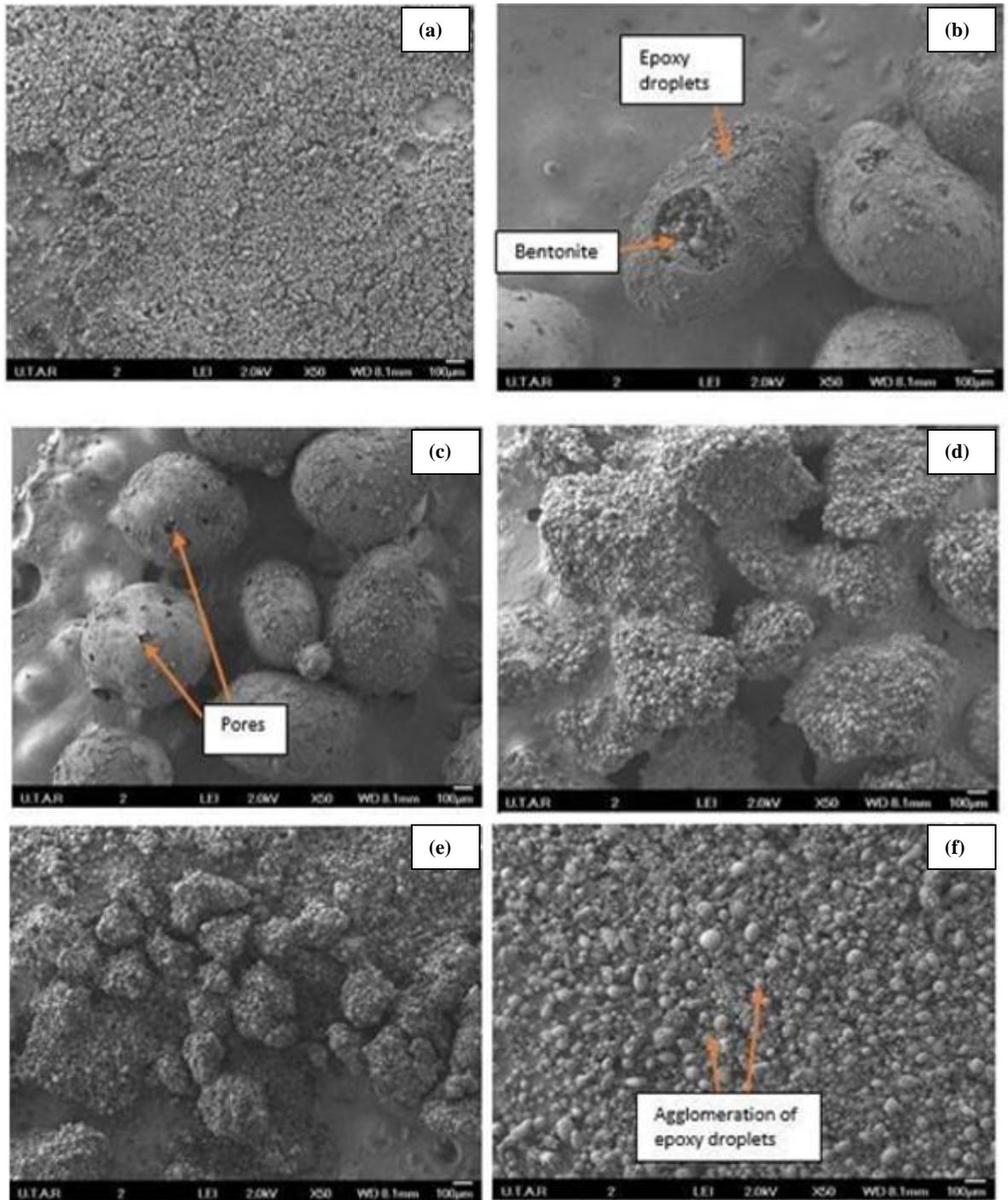


Figure 4.12: SEM micrographs of : (a) Raw bentonite, (b) PEB10, (c) PEB20, (d) PEB30, (e) PEB40 and (f) PED50 particles at 50 X magnification

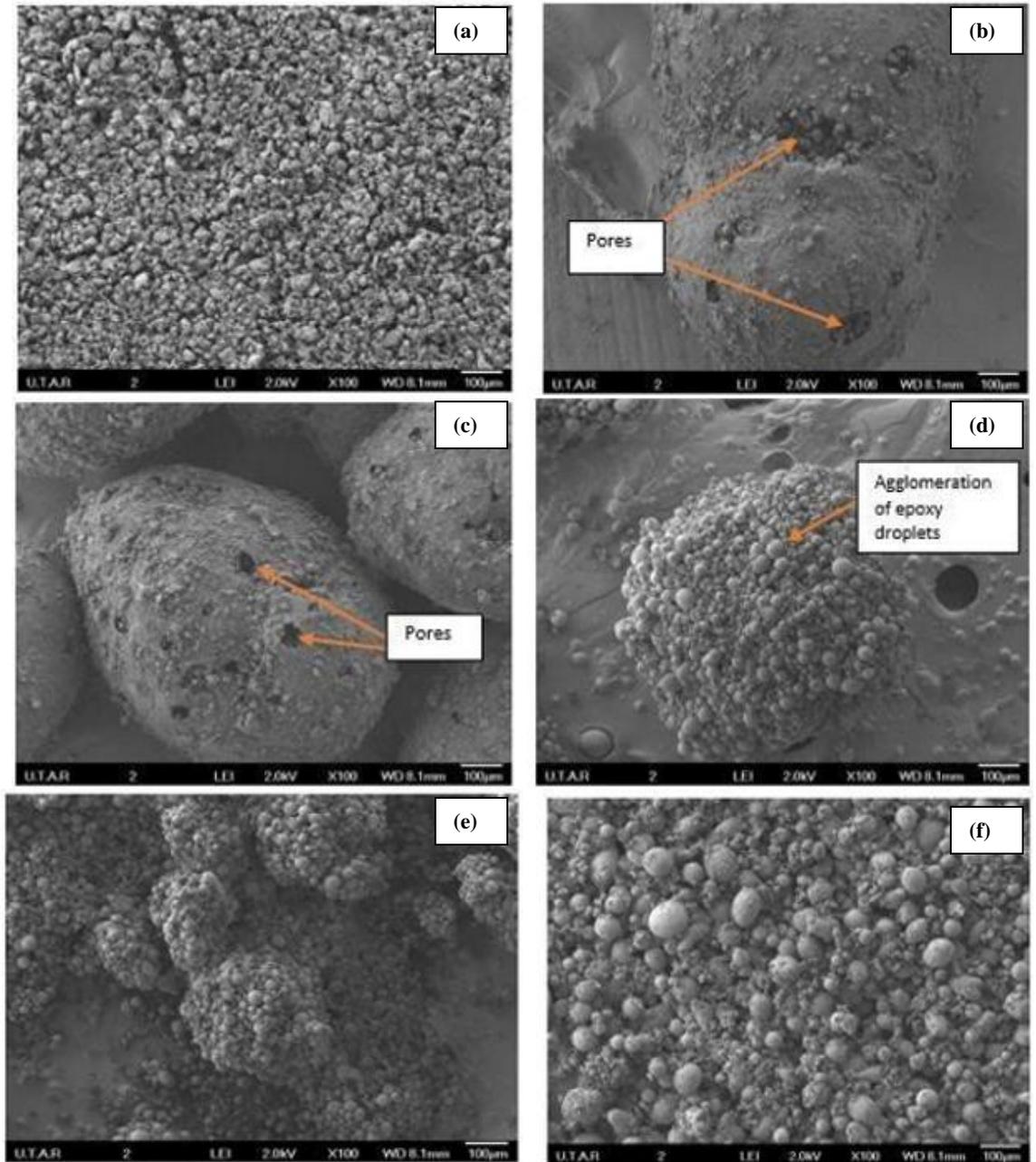


Figure 4.13: SEM micrographs of : (a) Raw bentonite, (b) PEB10, (c) PEB20, (d) PEB30, (e) PEB40 and (f) PED50 particles at 100 X magnification

Figures 4.12 (a) and 4.13 (a) shows the particles of raw bentonite which are in irregular shape with flat surface. By the SEM observation, the bentonite particles are in agglomerate form where the surface area is smaller due to its irregular shape (agglomerate form). The agglomeration of the bentonite particles with flatten surface are mostly not compatible in some application such as reinforcement of polymer matrix, where the agglomeration of bentonite particles tend to act as stress concentration point and site for the initiation of fracture cracks. Besides that, the irregular shape of particles also prevents good dispersion of reinforcement filler which lead to formation of agglomeration. Even though, the bentonite not compatible used as filler, hence its can be used as cheapener (Zielinski & Kettle, 2013).

Figure 4.12 (b) and 4.13 (b) shows the SEM micrograph of PEB10 with 10wt% of bentonite loading. It can be seen that, the particles is irregular circular shape with few pores and rough surface. The porosity of the porous epoxy/clay particles is introduced during the homogenization of epoxy and bentonite. The particles size of PEB10 is much larger than raw bentonite particles, where this due to coating of bentonite particles by the epoxy droplets. From the figure 4.12 (c) and 4.13 (c) its shows that the particles PEB20 with 20wt% of bentonite loading, where the shape is more in spherical shape and it's also produced more pores with rough surface compared to PEB10. The pores in PEB particles usually formed when the water absorbed by the coated bentonite particles purge out of the epoxy droplet during the heating process. Meanwhile, the particles size of PEB20 is much smaller than PEB10. It can be observed that, when increasing in bentonite loading the particle size become more to circular shape with rough surface. PEB20 particles produced with more porosity and well coated by the epoxy droplets, can be favorable in gas absorption application, where the high number of pores have ability to trap the gases (Zielinski & Kettle, 2013).

From the Figure 4.12 (d), 4.13 (d) and 4.12 (e), 4.13 (e) shows the SEM micrograph of PEB30 and PEB40 with 30wt% and 40wt% of bentonite loading respectively. From the observation, the PEB30 and PEB40 particles become irregularly in shape and also the particles produces with high surface roughness, where the large agglomeration is formed by the epoxy droplets. Meanwhile, the pores and surface voids can be seen but not as visible as PEB10 and PEB20. Besides that, there is gaps can be

seen on the surface of the particles which caused by the agglomeration of epoxy droplets. The high amount of bentonite loading reduces the wetting of bentonite particles by epoxy where the amount of epoxy not sufficient enough to well coat the bentonite particles.

Lastly, the Figure 4.12 (f) and 4.13 (f) shows the SEM micrograph of PEB50 with 50wt% of bentonite loading. It can be seen that, the produced particles are in irregular shape and with smaller surface area. The shape of the PEB50 produced particles is almost similar to the shape of raw bentonite particles. It can be observed that, the formation of agglomeration of epoxy droplets on the surface of raw bentonite where the wetting of bentonite particles couldn't be done by the epoxy droplets due to insufficient amount of epoxy to coat the bentonite particles. During homogenization process, the high amount of bentonite loading leads to poor dispersion of PEB particles where the high amount of bentonite swells in water during homogenization process which results in poor surface morphology with undesired shape of porous epoxy/clay particles (Zielinski & Kettle, 2013).

4.3.3 Density of PEB particles

Density is one of the important parameter in determining the properties of PEB particles. The density measurement is conducted on raw bentonite, PEB10, PEB20, PEB30, PEB40 and PEB50 by using gas pyrometer. In this research work, characterization through density measurement is conducted to studies on how the different bentonite loading alter the density of PEB particles and comparison made between the density of raw bentonite and PEB particles. Table 4.1 shows the average density of raw bentonite and other PEB particles produced.

Table 4.1: Average density of raw bentonite and PEB particles

Sample	Average Density (gcm^{-3})
Raw bentonite	2.453
PEB10	1.528
PEB20	1.463
PEB30	1.435
PEB40	1.403
PEB50	1.441

Basically when the bentonite is mixed with epoxy the density is decreased with the initial bentonite loading from 10 wt% to 40 wt%. This might be due to the coating of bentonite by epoxy-hardener mixture which has a lower density as compared to the raw bentonite or due to the formation of porous structure of PEB.

As can be seen from the SEM images, it is very clear that the PEB particles have more circular shape and larger in size. Which means the volume of individual PEB particles is larger than the raw bentonite particles. Thus increasing the volume of individual particles will result in decreased density if the particles based on the formula by which density are inversely proportional to volume (A.Webb, 2011).

Besides, when the porosity present on the structure, the overall mass of the particles will be reduced. Thus at similar volume of sample measurement during density test, the mass of the sample are lower. Since density are directly proportional to mass, decreasing mass of the sample may result in decreased density due to the presence of porous structure of PEB as compared to solid raw bentonite.

However, when the bentonite loading is very high (at 50 wt%), the coating/wetting of epoxy-hardener on raw bentonite become less and this can also be proven from the SEM observation. At high bentonite loading, some of the bentonite particles remain intact on the surface of PEB particles due to low coating/wetting by epoxy-hardener mixture. Besides, the amount of porosity also was decreased at higher bentonite loading as can be seen from the SEM observation which may result in

increased mass per volume of the sample during density measurement, subsequently increased the density of the PEB particles. Conversely, with increasing bentonite loading the density of PEB particles were decreased up to 40 wt% bentonite and increased slightly at 50 wt% bentonite loading. Meanwhile the density of PEB particles are relatively lower as compared to raw bentonite even at high bentonite loading (A.Webb, 2011).

4.3.4 Particle Size Analysis and Distributions

Analysis of particles size and distribution of porous particles are one of the important characterization in order to study how the particles size and its distribution affect the quality and performance of porous particles. Analysis of particles size and its distribution also commonly done to classify some materials or products. Particles size analysis and its distribution playing a vital role to determine the quality and performance of the porous particles by influence the mechanical properties, flow and compaction properties of particulate materials. In industry applications, controlling of particles size and its distribution is very important, where in paint industries, the particles size of a specific particles can significantly affect the appearance properties include the gloss of the paint particles. In this research work, analysis of particles size and its distribution is done for raw bentonite, PEB10, PEB20, PEB30, PEB40 and PEB50. Table 4.2 shows the physical properties of the raw bentonite and PEB particles (Micromeritics, 2014).

Meanwhile, Figure 4.14 to 4.19 shows the particles size distribution curve of the raw bentonite and each samples. The full results of particles size analysis and its distribution done is attached at Appendix A.

Table 4.2: Physical properties of raw bentonite and PEB particles

Sample	Specific Surface Area (m²/g)	Mean Diameter (μm)	Particles Distribution Range (μm)
Raw bentonite	0.422	21.924	1.352 – 97.957
PEB10	0.115	63.209	12.332 – 389.973
PEB20	0.132	52.192	12.332 – 447.749
PEB30	0.221	33.604	1.782 – 447.749
PEB40	0.241	30.765	1.782 – 339.653
PEB50	0.170	46.235	2.350 – 195.450

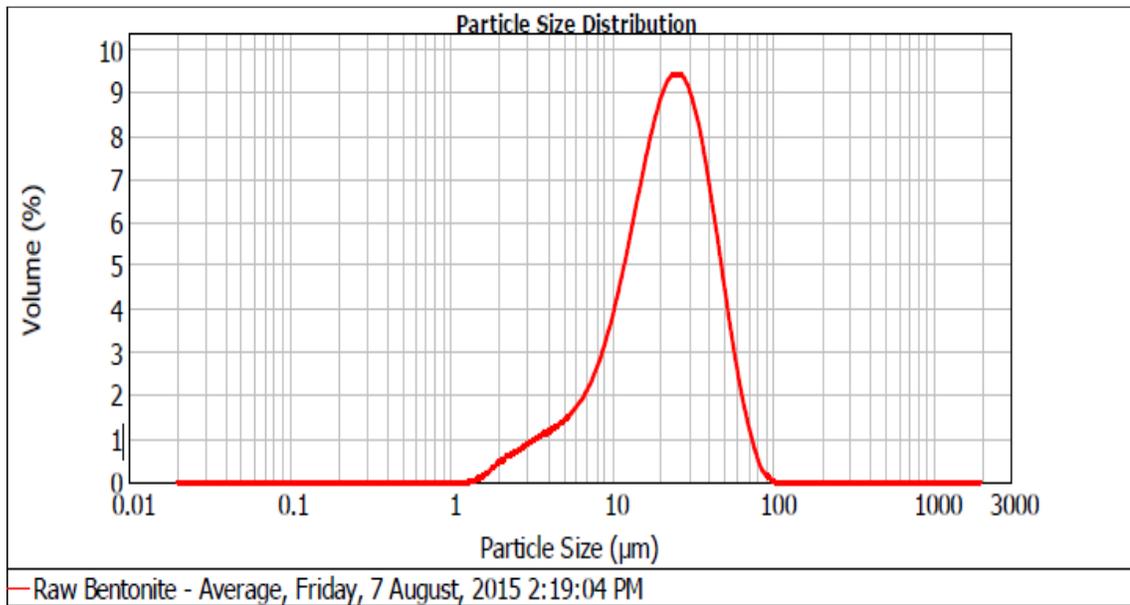


Figure 4.14: Particles Size Distribution of Raw Bentonite

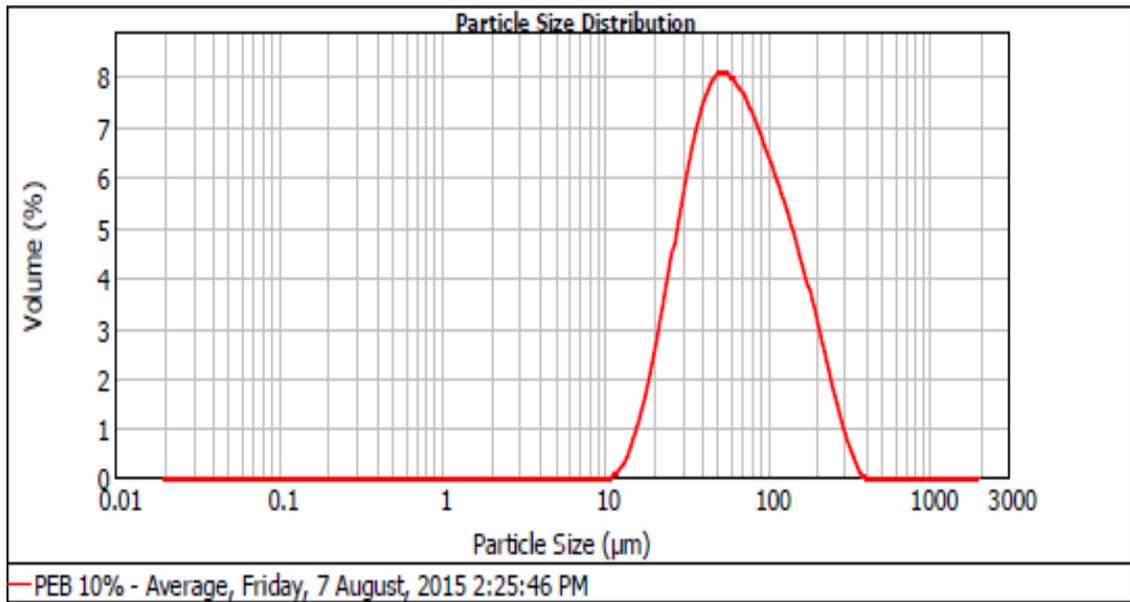


Figure 4.15: Particle Size Distribution of PEB10

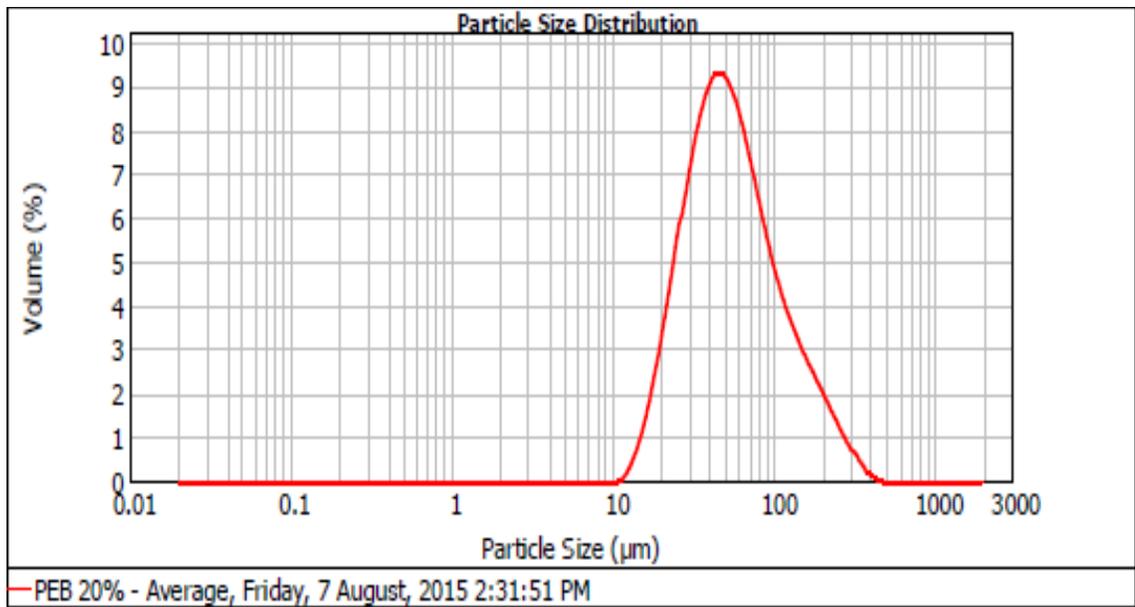


Figure 4.16: Particle Size Distribution of PEB20

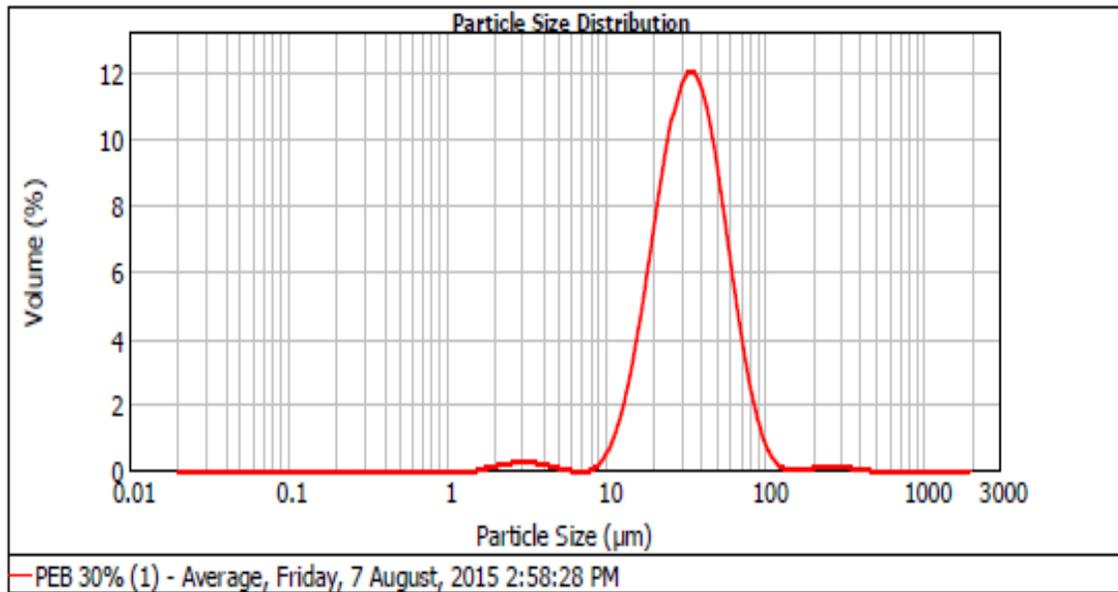


Figure 4.17: Particle Size Distribution of PEB30

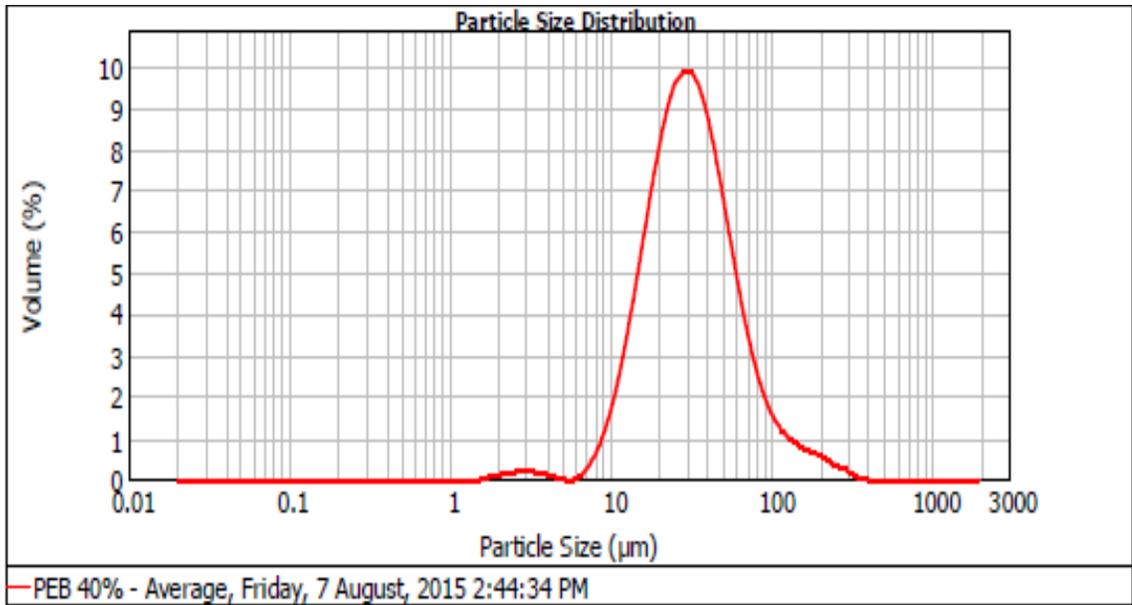


Figure 4.18: Particle Size Distribution of PEB40

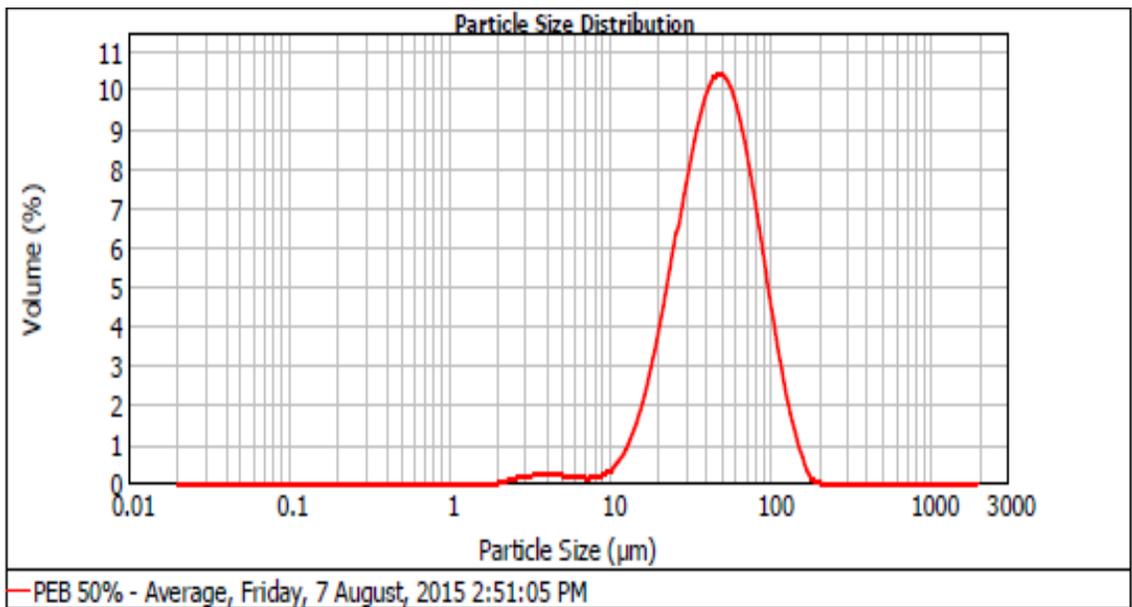


Figure 4.19: Particle Size Distribution of PEB50

From the Figure 4.14 its shows the common distribution of raw bentonite, where the particles size range between $1.352\mu\text{m} - 97.957\mu\text{m}$. The distribution curve of raw bentonite broader towards smaller particles size range $1.352 - 10\mu\text{m}$ which indicated the presence of smaller particles. From table 4.2 it can be observed that the trend of mean diameter of the particles decreases from 10wt% of bentonite loading to 40wt% and increased again at 50wt%. At 50wt% the mean diameter become larger again, this due to the high volume of bentonite loading results to the formation of agglomerations of bentonite particles on epoxy droplets, where epoxy amounts not sufficient to well coated the bentonite particles. This effect can be seen obviously on the SEM micrograph of the PEB particles.

Figure 4.15 and 4.16 exhibits the particles size distribution for PEB10 and PEB20. It can be observed that, for PEB10 and PEB20 the distribution is almost similar and it's broader towards large particle size, which indicated the particles size of PEB10 and PEB20 has larger particles with range between $12.332\mu\text{m} - 389.97\mu\text{m}$ and $12.332\mu\text{m} - 447.749\mu\text{m}$ respectively. For PEB10 and PEB20 with small volume of bentonite loading increased the wetting of epoxy and lead to production of large particles, where the bentonite particles trapped inside the epoxy droplets and well coated by the epoxy.

Meanwhile for PEB30 and PEB40 the particles size range between $1.782\mu\text{m} - 447.749\mu\text{m}$ and $1.782\mu\text{m} - 339.653\mu\text{m}$ respectively. Its shows that, the PEB30 and PEB40 has smaller particles compared to PEB10 and PEB20. Besides that, the particles size distribution curve that obtained for PEB10 and PEB20 have only one distribution, meanwhile, for PEB30, PEB40 and PEB50 have almost two distribution. From Figures 4.17, 4.18 and 4.19 can be seen that, PEB30, PEB40 and PEB50 have two distribution where the smaller distribution for each sample shows that the present of some fine particles. Size of the fine particles that presents in PEB30, PEB40 and PEB50 are in the range between $1.782\mu\text{m} - 5.383\mu\text{m}$, $1.782\mu\text{m} - 4.688\mu\text{m}$ and $2.350\mu\text{m} - 6.180\mu\text{m}$ respectively. The present of the fine particles may because of the bentonite particles not attached or wet by the epoxy droplets where the bentonite particles agglomerated. From the analysis of particle size and its distribution done, it can be conclude that when the bentonite loading increases, the wetting of epoxy droplets on bentonite particles will

decreases. Hence, the epoxy droplets become smaller, so the average particles size decreases (Micromeritics, 2014).

4.3.5 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was done on the raw bentonite, epoxy/polyamine and produced porous epoxy/clay (PEB) particles where the functional group, chemical compound and chemical bond exits in each sample were identified. FTIR also provides quantitative and qualitative analysis of the materials. Epoxy/polyamine was prepared through simple mixing mechanisms, where the small amount of epoxy mixed with small amount of polyamine and the resulting mixture is cured into thin transparent film for the FTIR analysis. For this analysis, IR spectrum of porous epoxy/clay particles with 10wt%, 30wt% and 50wt% was chosen to made comparison with IR spectrum of raw bentonite and epoxy/polyamine. Comparison was made in order to study the effect of bentonite loading on PEB particles through structural information of the molecules. Tables 4.3 and 4.4 shows FTIR analysis of raw bentonite and epoxy/polyamine respectively, besides that, Figures 4.20, 4.21 and 4.22 shows IR spectra of raw bentonite, epoxy/polyamine and combination of PEB10, PEB30 and PEB50.

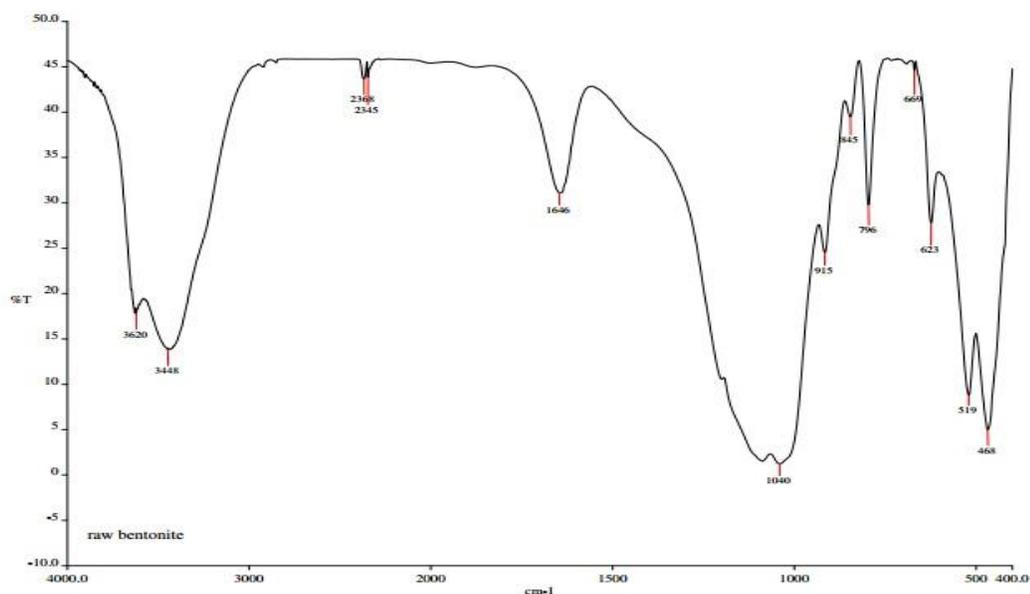


Figure 4.20: IR spectra of raw bentonite

Table 4.3: FTIR Analysis of Raw bentonite

Frequency (cm ⁻¹)	Types of vibrations
3620	O-H stretch
3448	Free O-H stretch
2368	FE ³⁺ - O stretch
2345	Mg-O stretch
1646	H-O-H
1040	Si-O
915	OH bending stretch
848	Al-O-Mg stretch
796	FE ³⁺ - O-Mg stretch
669	Si-O-Mg stretch
623	Si-O-Al stretch
519	Si-O-Si stretch
468	Si-O-Si stretch

According to the observation made, at IR absorption peak at 3620cm^{-1} and 3448cm^{-1} are identified the presence of O-H functional group stretch at the both peak, where at 3620cm^{-1} has medium intensity while at 3448cm^{-1} has strong intensity because of free O-H molecules where the rate of change of the dipole increases which results strong intensity. Absorption peak at 2368cm^{-1} and 2345cm^{-1} indicates the presence of Fe^{3+} - O stretched and Mg-O stretched, for both of the peaks have weak intensity where the stretching is narrower. Absorption peak at 1646cm^{-1} indicates the high absorption of water with strong intensity with the presence of water vibration H-O-H and hydrophilic characteristic of bentonite. The absorption peak at 1040cm^{-1} indicates Si-O functional group with symmetric stretching (strong and broad). The absorption peak at 915cm^{-1} indicates O-H bending vibration with medium intensity. Meanwhile, the absorption peak at 848cm^{-1} , 796cm^{-1} , 669cm^{-1} , 623cm^{-1} , 519cm^{-1} , and 468cm^{-1} indicates stretched vibration of Al-O-Mg, Fe^{3+} - O - Mg, Si-O-Mg, Si-O-Al, Si-O-Si, Si-O-Si respectively with medium intensity (Zuzana, et al., 2012).

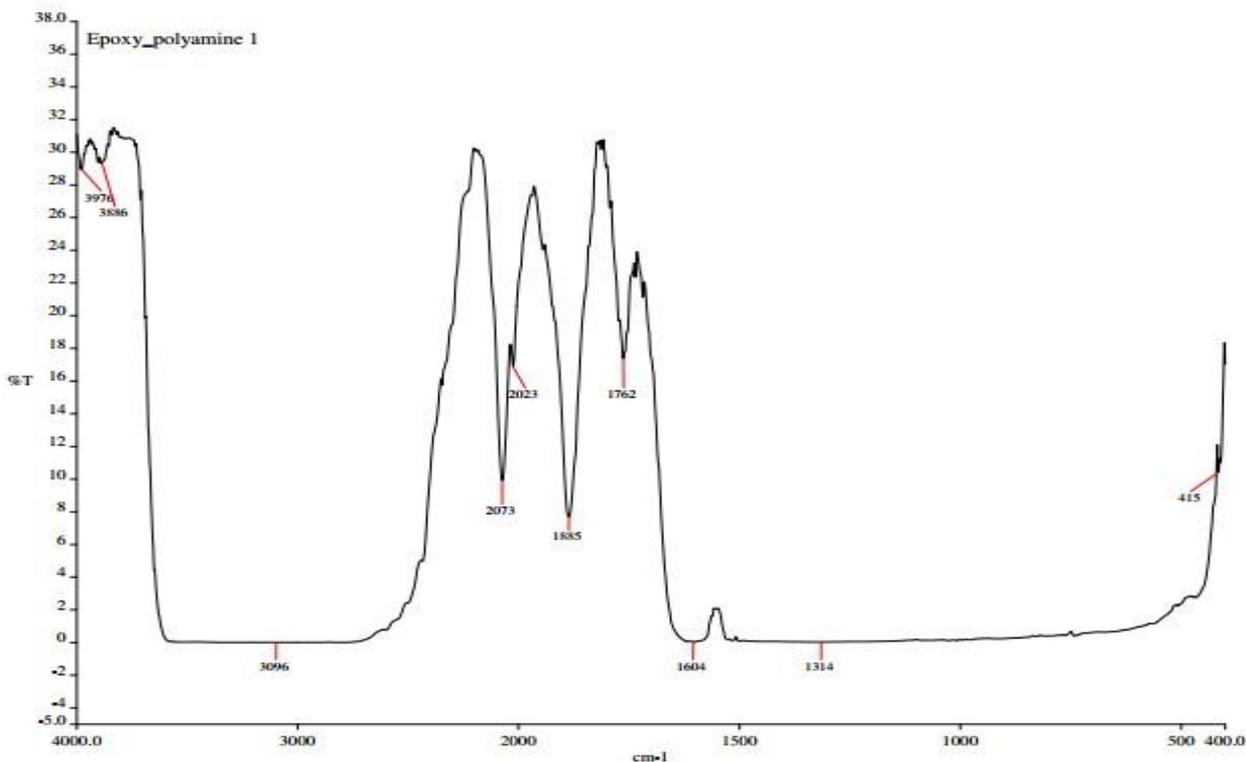


Figure 4.21: IR spectra of epoxy/polyamine

Table 4.4: FTIR Analysis of epoxy/polyamine

Frequency (cm ⁻¹)	Types of vibrations
3976, 3886	Ph-N stretch
3096	C-H stretch
2073, 2023	N-CH ₂ stretch
1885	C-O-C stretch
1762	C=O stretch
1604	N-H stretch
1314	C-O stretch
415	-

The Figure 4.21 shows the IR spectra of epoxy/polyamine. The absorption peak at 3976cm⁻¹ and 3886cm⁻¹ indicates Ph-N stretch vibration, the Ph represents (phenyl) with weak intensity which also indicates presence of tertiary amine. Absorption peak at 3096cm⁻¹ indicates C-H symmetric stretching vibration where it has strong intensity because the peak is broader. Meanwhile, absorption peak at 2073cm⁻¹ and 2023cm⁻¹ indicates same functional group which is N-CH₂ stretch with medium intensity. Absorption peak at 1885cm⁻¹ indicates C-O-C stretch with medium intensity, where the anhydride group presence from epoxy. Meanwhile absorption peak at 1762cm⁻¹ indicates the presence of C=O stretch ketone functional group with weak intensity. The presence of C=O at lower frequency indicates resonance, where a ketone that is conjugated with C=C double bond absorbs at a lower frequency. Absorption peak at 1604cm⁻¹ indicates the N-H stretch vibration with medium intensity, where the N-H actually presence from polyamine. Absorption peak at 1314cm⁻¹ indicates C-O group with weak intensity. C-O atom indicates heavier atom because as the atom bonded with carbon increases in mass, hence it vibrated lesser and presence at lower frequency. Lastly, absorption peak with very low frequency which at 415cm⁻¹ occur due to the moisture absorption of potassium bromide (KBr) where the KBr presence during pellet preparation (González, et al., 2000).

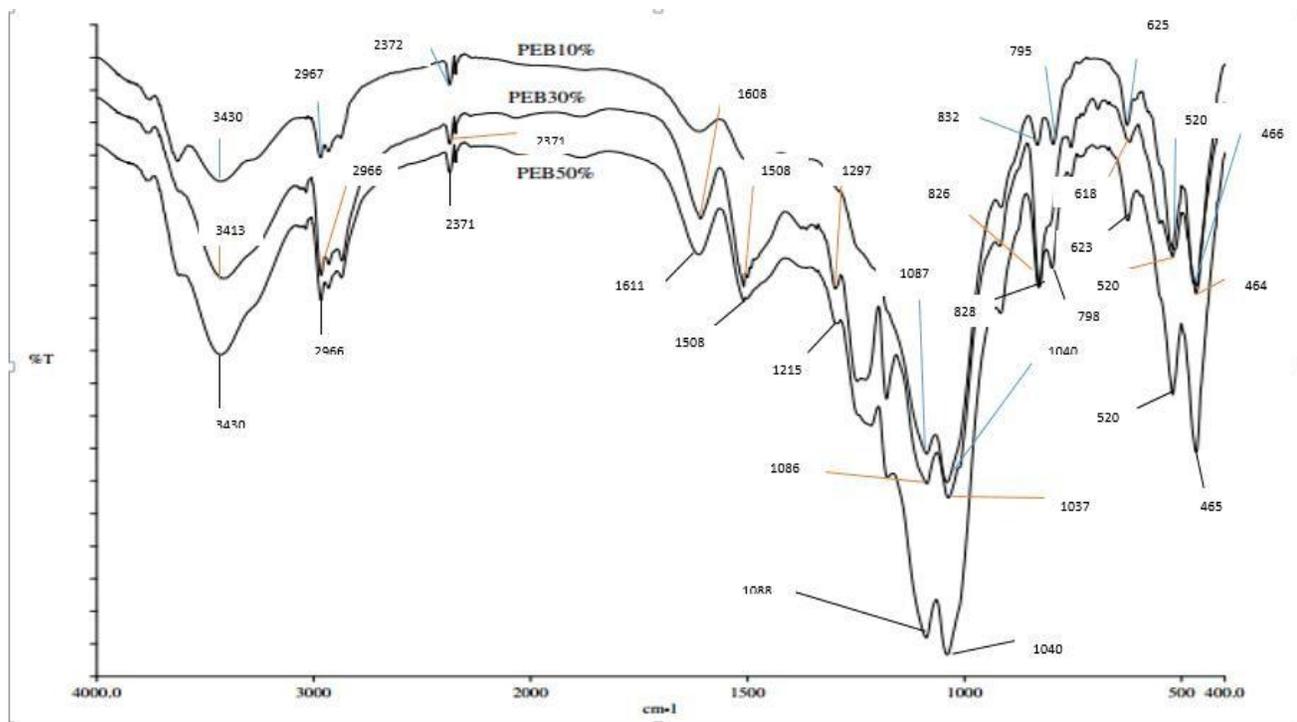


Figure 4.22: IR spectra of PEB10, PEB30, and PEB50

Figure 4.22 shows the IR spectra of PEB10, PEB30 and PEB50. These three samples are chosen for FTIR analysis except PEB20 and PEB40 because, bentonite loading of 10wt%, 30wt% and 50wt% shows obvious changes in terms of physicochemical properties such as surface morphology of the particles. Absorption peak at 3629cm^{-1} for PEB10 indicates O-H stretch vibration with medium intensity. The presence of O-H bond clearly proved the presence of epoxy because O-H bond is a main functional group of epoxy. This O-H bond shifted to higher frequencies due to opening of the epoxy ring. From the observation on PEB30 and PEB50 IR spectra, there is no O-H peak presence which proved that, as the bentonite loading increases the wetting of epoxy droplets decreases.

Absorption peak at 3430cm^{-1} for PEB10 indicates N-H stretch vibration with medium intensity. N-H bond proved that the presence of polyamine because, any atoms bonded with -N indicates the main functional group of polyamine itself. Absorption peak at 3413cm^{-1} for PEB30 also indicates the stretch N-H vibration with medium intensity.

From the observation, it shows that, the frequency of N-H bond in PEB30 is dropped low compared to PEB10 due to the high loading of bentonite. Absorption peak at 2967cm^{-1} of PEB10 indicates C-H stretch vibration. Meanwhile, the C-H stretch vibration for PEB30 and PEB50 presence at 2966cm^{-1} with weak intensity and low frequency compared to PEB10 which indicates the polyamine composition reduces with high bentonite loading. Absorption peak at 2345cm^{-1} and 2372cm^{-1} for PEB10 indicates C=N stretch vibration this due to the conjugation of nitrogen in polyamine.

For PEB30 and PEB50 the C=N bond presence at same frequency at 2345cm^{-1} and, 2371cm^{-1} and lower compared to PEB10, the lower frequency results from bond conjugation moves stretch to lower frequency. Absorption peak at 1087cm^{-1} and 1040cm^{-1} for PEB10 indicates C-N stretch accompanied by multiple bands. For PEB30 and PEB50 the absorption peak of C-N stretch occurs at lower frequency which at 1086cm^{-1} , 1037cm^{-1} and 1088cm^{-1} , 1040cm^{-1} respectively. Absorption peak at 832cm^{-1} of PEB10 indicates Al-O-Mg stretch vibration with weak intensity. Meanwhile for PEB30 and PEB50 the Al-O-Mg stretch vibration presence at 826cm^{-1} and 828cm^{-1} respectively (Djomgoue & Njopwouo , 2013).

Absorption peak at 795cm^{-1} for PEB10 indicates Fe^{3+} -O-Mg stretch vibration with weak intensity. For PEB50, Fe^{3+} -O-Mg stretch vibration presence at 798cm^{-1} , where for PEB30, no Fe^{3+} -O-Mg peak observed due to bonded to heavier elements. Absorption peak at 625cm^{-1} for PEB10 indicates Si-O-Mg stretch vibration with weak intensity. For PEB30 the Si-O-Mg stretch vibration presence at lower frequency compared to PEB10 which is at 618cm^{-1} . Absorption peak at 623cm^{-1} for PEB50 indicates Si-O-Al stretch vibration. Absorption peak at 520cm^{-1} and 466cm^{-1} indicates stretch Si-O-Si vibration. For PEB30 and PEB50, the Si-O-Si stretch vibration presence at frequency at 520cm^{-1} , 464cm^{-1} and 520cm^{-1} , 468cm^{-1} respectively (Djomgoue & Njopwouo , 2013).

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

Conclusions have been made from the results obtained from this research as follows:

1. Porous epoxy/bentonite clay particles (PEB) were successfully produced through water-oil homogenization method.
2. The effect of epoxy/bentonite/hardener mixture: water ratio has been identified by conducting preliminary test on the epoxy/bentonite/hardener mixture: water ratio to produced porous epoxy/bentonite clay particles. The mixture: water ratio of 1:2 is identified as most compatible ratio to produced porous epoxy/bentonite clay particles (PEB).
3. Porous epoxy/bentonite clay particles (PEB) were successfully developed at various bentonite loading through water-oil homogenization method at fixed epoxy/bentonite/hardener mixture: water ratio of 1:2.
4. Porous epoxy/bentonite clay particles (PEB) produced successfully characterized to identify the particles size and distribution, density, microstructure and morphological properties.
 - Particle size of porous epoxy/bentonite clay particles decreases with increasing of bentonite loading.
 - SEM analysis shows that the particles size of PEB particles decreases with increasing in bentonite loading. At lower bentonite loading PEB particles have more porosity compared to PEB particles with high bentonite loading. The shape of PEB particles produced become irregular as the bentonite loading increases.
 - Average density of PEB particles decreases with increasing of bentonite loading.

5.2 Recommendations

During this research work, some weakness have been recognized. Some recommendations have made in order to enhanced this research work with more accurate results. The recommendations as follow:

1. Various mixture: water ratio with different homogenization speed and time can be used to study the effect on the production of porous epoxy/bentonite clay particles.
2. More testing can be done to characterized the PEB particles such as Thermogravimetric analysis and water absorption test to provide information on thermal stability and swelling properties of PEB particles.
3. When conducting laboratory work, using of plastic ware during mechanical mixing and curing process is more preferable instead of glassware. Mechanical mixing will be carried out by using mechanical stirrer, where the shape blade with high speed can results in breakage to glass beaker. Using of glass beaker for curing the homogenized mixture, the cured products tend to stick on the glass beaker which results in difficulty in cleaning the glass beaker. PP (polypropylene) microwave Tupperware is recommended to use for curing process.

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