Investigation on Coating Materials to Enhance Sand Proppant Performance for Sand Production Control

by

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CERTIFICATION OF APPROVAL

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ABSTRACT

Disclosed herein is review/research for investigations on coating materials to enhance sand proppant properties exploiting local sand supply, reported methods of forming resin coated particulates and present invention provides sand production control using resin coated sands. The objectives of this project are to determine the desired behavior of local resin coated silica with desired mesh size of 40/70 sand particles as well as to investigate and analyze the characteristics and physical properties of the obtained sample of resin coated silica using different types of resin; **Epikote628, Epoxy, Polyester** and **Vinyl Ester**. This research has been done as a result of the extremely high well maintenance cost due to abrasive mechanical erosion of downhole equipment during operation by massive sand productions; meanwhile the limitation of natural sand and delivery period for current proppant are time consuming which cause a significant delay in oil/gas yield production. Likewise, current proppants application has met with limited success due to the reaction of the resin coating in the wellbore. For this particular project, the scope of study will widely covers but are certainly not limited to the investigation of coating material for sand proppant, its types and properties, even so include studies on local sand potential for sand proppant, coating methods, types of resin and its properties, testing for physical properties of the sample that will be obtained will be predetermined in this paper. Basically, background of study of the project covers the feasibility of the project – sufficient supply of local sand and resins, equipments needed/availability for sample(s) testing, potential of local sand to be commercialize. Findings trough research/review of books, journals, patented articles, web site, etc. are been inferred throughout this paper. The method is applying a coating including the continuous phase resin and non-reactive sand particles embedded or adhered to the continuous phase. The final finding is that vinylester resin shows the high potential characteristic of good resin for application.

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(ROHAIZREEN IRDAYU BINTI MOHD RADZI)

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CHAPTER 1

INTRODUCTION

Loose or incompetent sand is being produced along with formation fluids such as hydrocarbon and/or water (Todd, Slabaugh, Powell, & Yaritz, 2001). Therefore, sand control method has been applied in order to prevent unwanted event in future such as erosion of tubing/other equipment, plugging, production depletion and etc. Hydraulic fracturing operations utilize resin coated particulates, normally employed as proppants, to boost, keep open fractures and maintain the relative permeability of the hydraulically induced fracture with respect to the natural permeability of the hydrocarbon bearing formation (Harry & Sharif, 1996) (R.McDaniel, McCrary, W.Green, & Xu, 2009). The term proppant is indicative of particulate material which is injected into fractures in subterranean formations surrounding oil wells and gas wells to provide support to hold or prop these fractures open and allow gas or liquid to flow through the fracture to the bore hole or from the formation (McCrary, Barajas, & McDaniel, 2009). Brief definition by Schlumberger Oilfield glossary, described that, proppant is sized particles mixed with fracturing fluid to hold fractures open after a hydraulic fracturing treatment. In addition to naturally occurring sand grains, man-made or specially engineered proppants, such as resin-coated sand or high-strength ceramic materials like sintered bauxite, may also be used. Proppant materials are carefully sorted for size and sphericity to provide an efficient conduit for production of fluid from reservoir to the wellbore (Schlumberger Oilfield Glossary). Examples of particulates used as proppants in various hydraulic fracturing operations include particles of sand, glass beads, ceramics and nutshells/walnut shells (Harry & Sharif, 1996) (McCrary, Barajas, & McDaniel, 2009) (Grood & D.Baycroft, 2010). It is preferable to use sand as a medium of propant by reason of the advantage of sand is that it is cheap whereas the disadvantages are its relatively low strength/high crush values and lower flow capacities than sintered ceramic particles.

Nonetheless, the ceramic particles are disadvantageous in that the sintering is carried out at high temperatures, resulting in high energy cost (McCrary, Barajas, & McDaniel, 2009). Pilato (2010) writes each particulate has been resin coated and cured at one time or another to enhance its performance.

Natural sands and synthetic proppants are exploited and produced, respectively in various place in the world. While traditionally both types of materials chiefly derived from the USA, in the last decade increasing contributions are also coming from other countries and continents e.g. China. According to Detlef Mader (1989), replacing sintered bauxite by resin coated sand can result in about 50% total proppant expense savings due to both cheaper pound prices and higher volume of the end product (p. 32). Proppant package stability improvement by application of resin coated grains is a common and efficient means of sand production control and prevention of fracturing operation failure by proppant flowback.

Sand sample from Meraga, Terengganu, East Coast of Peninsular Malaysia is to be tested with four different types of resin for this project. The massive sources of approximately 1.5 meters depth in hundreds hectares of sandy area were found by Malaysian Mineralogy Department. The reason of choosing this sand is due to the abundant resource of the sand approximately 45.6Mt sand reserves. Besides, the sand also showing desired characteristics of good quality sand for resin coated silica purpose such as sphericity and roundness (Universiti Teknologi PETRONAS, 2011).

1.1 Problems Statements

Proppant flowback does not only result in fracture damage by removal of the support of the crack and thereby triggering its partial closure, but similarly as does sand production also leads to borehole operational problems such as stuck pumps plungers, eroded pump valves, sucker rod failures, stuck tubing and proppant fill (Pope, Willes & Pierce 1987) (Grood & D.Baycroft, 2010). Proppant flowback can also fill surface equipment with sands which gives rise to inaccurate fluid

measurements and eventually requires extensive cleaning. In many cases, the cost to return a well to oil production after failure by proppant flowback has been so excessive that the well had to be prematurely abandoned (Mader, Hydraulic Proppant Fracturing and Gravel Packing, 1989, p. 32).

Harry and Sharif (1996) reported that the practice in the oil field pumping services and production industry of using resin coated particulate to enhance the conductivity of fractures in hydrocarbon bearing formation has met with limited success due to the reaction of the resin coating with the additives which comprise the hydraulic fracturing fluid.

1.2 Objectives and Scope of Study

The objectives of the project are:

1. To determine the desired behavior of local resin coated silica of 40/70 mesh size sand particles.
2. To investigate and analyze the characteristics and physical properties of the obtained sample of local resin coated silica using different types of resin.

For this particular project, the scope of study widely covers but are certainly not limited to the investigation of coating material for local sand proppant, its types and properties, even so include local sand potential for sand proppant, proper and efficient proppant sample coating methods, types of resin, testing for physical properties of the obtained sample are briefly discussed in this paper.

1.3 Background of Study

Basically, background of study of the project covers the **feasibility of the project (within specified time frame)** – sufficient supply of local sand and resins, equipment needed/availability for sample(s) testing, potential of local sand to be commercialize and particle packing characteristics.

1.3.1 Local Sand as Sand Proppant

Sand samples from Meraga, Terengganu are coated using four different types of resin namely Epikote628, Epoxy, Polyester and Vinyl Ester. Accordingly the samples obtained are subjected to three types of physical testing; porosity test, permeability test and shear stress test. Consequently, their property is compared.

In order to further with the project progress, several factors that need to be considered have been studied in details. Thus, the requirements must be met for sand feasibility as specified by American Petroleum Institute, API (API RP 58, 1995):

- Enough sand resource for supply.
- Sand used is qualified by Authority Board.
- Sand used suitable for coating.
- Skin (thickness) of the applied resin coating.

1.3.2 Desired Characteristics of the Resin System

Resins are polymers made by repeatedly linking discrete molecules also known as monomers, together to form chains or networks (Phenolic Novolac and Resol Resins, 2011). Any resin system for use in a composite material will require the following properties:

1. Good mechanical properties.
2. Good adhesive properties.
3. Good toughness properties.
4. Good resistance to environmental degradation.

1.3.3 Particle Packing Characteristics

An understanding of particle packing characteristics is vital in many diverse fields of technology (German, Particle Packing Characteristics, 1989, p. 16). However, insufficient attention is often given to the particle packing characteristics. From studies by German (1989), it is proven that more advantage can be made from a better understanding of particle packing. The overall success in fabricating high performance structures depend on the ability to work with high packing density particulate systems. A high compaction force could be applied to the particle to overcome poor packing (p. xiii).

German (1989), in his book writes that the key characteristics of a particle mass which are especially important to processing include packing density/porosity, strength, surface area, packing coordination, permeability, pore size and connectivity (p. 9). The desire is to predict flow behavior in terms of simple structural parameters such as the pore size. In turn, the flow rate can be estimated if the pressure drop is known, or the pressure drop can be estimated for a needed flow rate (p. 354). The two fundamental types of fluids; liquids and gasses, differ in compressibility. The flow rate in the pore structure, as a further subdivision, determines the interaction between the fluid and pore, thereby controlling the flow resistance. It is noted that a fluid passing through a pore will momentarily stick to the pore surface, leading to drag. In this case, the interaction between the pore wall and the fluid combines with the

fluid viscosity to determine the flow resistance. Viscous flow occurs with both liquids and gasses (German, 1989, p. 354).

Various relations linking the pore size to the particle size and packing density have been proposed. Efforts to relate the particle packing features to the particle size are generally prone to error. Combinations of techniques can be potent in characterizing packing structures; for example, mercury porosimetry and gas adsorption measurements can be cross-calibrated to pore volume, surface area and pore size. These provide an excellent basis for assessing the packing characteristics of very small particles (German, 1989, p. 419). Therefore, to obtain high performance proppant design, particle packing characteristics is one the crucial part to look at.

CHAPTER 2

LITERATURE REVIEW

A gravel pack is a term commonly used to refer to a volume of particulate materials placed into a wellbore to at least partially reduce the migration of the unconsolidated formation particulates; sand/fines into the wellbore (D.Weaver & D.Nguyen, 2010) (McCrary, Barajas, & McDaniel, 2009). In gravel packing operations, the coated and/or uncoated particles suspended in a carrier fluid are pumped into a wellbore in which the gravel pack is to be placed. The carrier fluid leak off into the subterranean zone and/or is returned to the surface while the particles are left in the annulus between the production string and the casing or outside the casing in the formation adjacent to the wellbore (McCrary, Barajas, & McDaniel, 2009). The resultant gravel pack acts as a filter assembly to support and retain the particles placed during the gravel pack operation (McCrary, Barajas, & McDaniel, 2009) as well as to separate formation sand from produced fluid while permitting the produced fluid to flow into the wellbore. Typically, gravel pack operations involve placing a gravel pack screen in the wellbore and packing the surrounding annulus between the screen and the wellbore with gravel designed to prevent the passage of formation sands through the pack (D.Nguyen, D.Weaver, & D.Desai, 2007). Gravel packing may also be used to protect the wellbore wall production integrity by employing a tightly packed deposit of aggregate comprising sand, gravel or both between the borewall and the production pipe thereby avoiding the time and expense of setting a steel casing from the surface to the production zone which may be many thousands of feet below the surface. The gravel packing is inherently permeable to the desired hydrocarbon fluid and provides structural reinforcement to the borewall against an interior collapse of flow degradation. It is recognized as open hole completions. In some situations the processes of hydraulic fracturing and gravel packing are combined into a single treatment to provide stimulated production and an annular gravel pack to reduce formation sand production. Such treatments are often referred to as frac pack

operations (McCrary, Barajas, & McDaniel, 2009). McCrary et al (2009) also point out that, the application of proppant particles has become common practice and highly desirable that the proppant particles are of high performance and can be produced in highly efficient processes; economically attractive. Like a gravel pack, a resin coated sand pack is sized to hold back the formation sand. However, a resin coating, rather than a screen, holds the sand pack in position. Working through tubing, gravel pack sand is usually pumped via coiled tubing into the perforation tunnels and void spaces outside the casing. The resin coating hardens and bonds the gravel together. Excess resin coated sand is removed from inside the casing, usually by drilling it out. Like all chemical sand consolidation treatments, productivity will be reduced by the treatment (Bellarby, Well Completion Design, p. 223).

The treatments should be performed on a live well, that is, through tubing with pressure control. As closure stresses are unlikely to be high, placement will be eased by low-density proppants. Consolidation relies on temperature rather than stress (Bellarby, p. 224). According to McDaniel and Robert,R. (2010), resin coated proppant comes in two types: precured and curable. Precured denotes the resin coating is already cured before it is introduced into the well. Thus, even under extreme conditions, the proppant does not agglomerate and this is reverse with the curable type. McCrary, et al. (2009) also describes the similar definition in their writing.

2.1 Resin Types

The resins that are used in current industries can also be referred to as polymers. All polymers exhibit an important common property in that they are composed of long chain-like molecules consisting of many simple repeating units. Man-made polymers are generally called synthetic resins or simply resins. Polymers can be classified under two types, thermoplastic or thermosetting, according to the changes of its properties on applied heat (Net Composites, 2011).

Thermoplastics are like metals, soften with heating and eventually melt, hardening again with cooling. This process of crossing the softening or melting

point on the temperature scale can be repeated as often as desired without any significant effect on the material properties in either state (Net Composites, 2011).

Thermosetting materials, or thermosets, are formed from a chemical reaction in situ, where the resin and hardener or resin and catalyst are mixed and then undergo a non-reversible chemical reaction to form a hard, infusible product. In some thermosets, such as phenolic resins, volatile substances are produced as by-products. Other thermosetting resins such as polyester and epoxy cure by mechanisms that do not produce any volatile by products and thus are much easier to process. Once cured, thermosets will not become liquid again if heated, although above a certain temperature their mechanical properties will change significantly. This temperature is known as the Glass Transition Temperature (Tg), and varies widely according to the particular resin system used, its degree of cure and whether it was mixed correctly (Net Composites, 2011).

When the substrate comprising the thermosetting polymer, it is desirable for the organic materials to undergo curing or otherwise known as crosslinking upon the application of either thermal energy, electromagnetic radiation or a combination comprising at least one of the foregoing. Initiators may be used to induce the curing. Other additives that promote or control curing such as accelerators, inhibitors or the like can also be used (McCrary, Barajas, & McDaniel, 2009). As in this project, methanol is been choose as additive.

Although there are many different types of resin in use in the composite industry, the majority of structural parts are made with three main types, namely **polyester, phenolic and epoxy.** For some reason, in this project, the resins that will be employed are **Epikote628, Epoxy (Quickmount 2), Polyester and Vinyl Ester**. These four resins will be utilized for further investigation using local sand (Oniyanagi, Wada, Oowada, & Saikawa, 2011). Basically, Epikote628 and Epoxy

(quickmount 2) are from the same family of epoxy resin. Studied below are the comparisons of above-mentioned types of resin.

2.2 **Resins Comparisons** (Net Composites, 2011) (Glass-Evercoat, 2011)

POLYESTER RESIN VERSUS EPOXY RESIN

Characteristics	Polyester Resin	Epoxy Resin
Flexural Strength	Good	Best
Tensile Strength	Good	Best
Elongation %	Good	Lowest
Water Absorption	Good	Lowest/Excellent
Hardness	Good	Best
Pot Life	4 – 7 Minutes	14 – 20 Minutes
Working Time	20 – 30 Minutes	½ - 6 Hours
Above Waterline	Yes	Yes
Below Waterline	Yes	Yes
Major Construction	Yes	Yes
General Repairs	Yes	Yes
Shelf Life	18 –24 Months	2 Year +
Catalyst	MEKP	2-Part System
Cure Time	6 – 8 Hours	5 –7 Days

Table 2.1: Polyester Resin vs. Epoxy Resin (Glass-Evercoat, 2011)

The polyesters, vinyl esters and epoxies discussed here probably account for some 90% of all thermosetting resin systems used in structural composites. In summary the main advantages and disadvantages of each of these types are (Net Composites, 2011):

Polyester

Advantages	***Disadvantages***
Easy to use	Moderate mechanical properties
Cheapest resin available	High styrene emissions in open moulds
	High cure shrinkage
	Limited range of working time

Table 2.2: Advantages and disadvantages of Polyester resin (Net Composites, 2011)

Vinylester

Advantages	*Disadvantages*
Very high chemical/environmental resistance	Postcure generally required for high properties
Higher mechanical properties than polyester	High styrene content
	Higher cost then polyesters
	High cure shrinkage

Table 2.3: Advantages and disadvantages of Vinylester resin

(Net Composites, 2011)

Epoxy

Advantages	*Disadvantages*
High mechanical and thermal properties	Expensive
High water resistance	Critical mixing
Long working times available	Corrosive handling
Temp. resistance can be up to 140^{o}C wet/ 220^{o}C dry	
Low cure shrinkage	

Table 2.4: Advantages and disadvantages of Epoxy resin

(Net Composites, 2011)

2.3 API Requirements

Since the proppant is being pumped into reservoir extreme condition, there are some parameters needed to be fulfilled according to the specification established by API. According to (API RP 60, 1995), the parameters are set as follows:

Properties	API requirements
Sphericity & Roundness	> 0.6
Acid Solubility	2-3%
Bulk Density	2.00g/cm^3
Crush Resistance (40/70 Mesh)	8% crushed sand @5000psi
Turbidity Test	250FTU

Table 2.5: API Requirements

Meraga's sand sample which been employed for this project was previously been analyzed for its properties to meet the API requirements. Since its properties have been verified to meet those requirements stated above, further researches are conducted throughout this project to investigate the coating materials to enhance its properties as a proppant. Below is the result of the analysis:

Properties	Data of Meraga's Sand Sample
Sphericity & Roundness	0.71 and 0.64
Acid Solubility	5.08%
Bulk Density	1.67g/cm^3
Crush Resistance (40/70 Mesh)	15% crushed sand @2000psi
Turbidity Test	319FTU

Table 2.6: Results of Meraga's Sand Sample

2.4 Package Stability Improvement by Resin Coated Proppant

Mader (1989) writes that, several alternatives method has been implied to control proppant flowback following fracture simulation comprising rising the pumping equipment above the perforations, using special sand pumps, installing gravel pack and running a tubing conveyed sand filter, but sufficient success was not achieve with any of these procedures. Satisfactory control of proppant flowback, however could be obtained by performing the fracturing treatment with a tail-in of curable resin coated sand of about 10%-20% of the total proppant quantity. (p. 689)

Under conditions of reservoir temperature and pressure, the resin chemically bonds together to form a consolidated permeable barrier against proppant flowback once carried out properly. Resin coated proppant tail-in effectively prevents fracture evacuations in the near wellbore area, because the consolidated proppant packages firmly couples boreholes and crack thus leading to optimum drainage of the productive formation (Sinchlair, Graham & Sinchlair 1983). In addition to inhibition of fracture evacuation, proppant flowback prevention greatly reduces well maintenance, wear, erosion and abrasion problems (Mader, 1989, p. 689)

Failure rates in fractures with a wedge of resin coated proppants in the terminal interval near the wellbore are much lower than in conventionally equipped boreholes. Failures of the resin coated sand to control proppant flowback can be attributed to failure of resin bonding and failure of the resin coated sand to cover the whole perforated interval. If the resin cures before the gel breaks and thus before the proppant grains come into contact, the ability of consolidation of the proppant package is lost. If the proppant is being banked near the wellbore during simulation, the curable resin coated sand in the tail-in will override the top f the

banked proppant and cover only the perforations that are taking fluids. (Mader, 1989, p. 689)

Advance studies by Mader (1989), subsequent acidizing does not affect the stability of the consolidated package due to resin inertness. Productivity losses due to fracture evacuation near the wellbore by flowback of uncoated proppants have turned out to be much higher than reductions of exploitation due to slightly lower flow efficiencies of resin coated proppant consolidated package with respect to those of cracks propped with conventional sand, thus underlining that a tail-in of resin coated sand is an effective method of controlling proppant flowback following fracture simulation. (p. 689)

Figure below shows the comparison of the advantage using coated proppant over uncoated proppant (Grood & D.Baycroft, 2010). The proppant volume means the end product of the proppant yielded. For coated proppant, the volume is large due to the amount of resin added to the particles, whereas the uncoated proppant are sole particles. High volume of coated proppant, indicates that only small amount of proppant are required yet sufficient to be injected into the wellbore compared to the uncoated proppant.

CONDITION	UNCOATED PROPPANTS	COATED PROPPANTS
Pumping rate	15-25 bpm	25-40 bpm
Proppant concentration	10-12 ppa*	12-15 ppa*
Proppant volume	250,000 lbs	400,000 to 800,000 lbs.
Flow Velocity	70-400 ft/sec.	70-800 ft./sec.

*Pounds proppant added (per gallon)

Figure 2.1: Advantages using coated proppants over uncoated proppants

(Grood & D.Baycroft, 2010)

CHAPTER 3

METHODOLOGY

3.1 Reported/Researched Methodology

Researched methodology is exceedingly vital yet is done in order to help the progress of the project in wider scope. Overview of the project is obtained through researches and studies. Thus, it can be preserve as the basic guideline in completing the assign project.

3.1.1 Forming Resin Coated Particulate Materials
(R.Murphey & D.Totty, 1989) (Grood & D.Baycroft, 2010)

After completing a lot of journals, patents and books review focus for coating method for this project is indicated by the red box in **figure 3.1**. This is based on patented journal by R.Murphey & D.Totty (1989)**. Figure 3.2** and **3.3** are used as references on how resin coated sand are being prepared (Pilato, 2010). R.Murphey & D.Totty (1989) shows that the present inventors found that resin-coated sand using spherical molding sand that is refractory particles having a specific composition and particle diameter with high sphericity and smooth surface reducing water absorption can exhibit excellent performance as molding sand. (Ina, 2011)

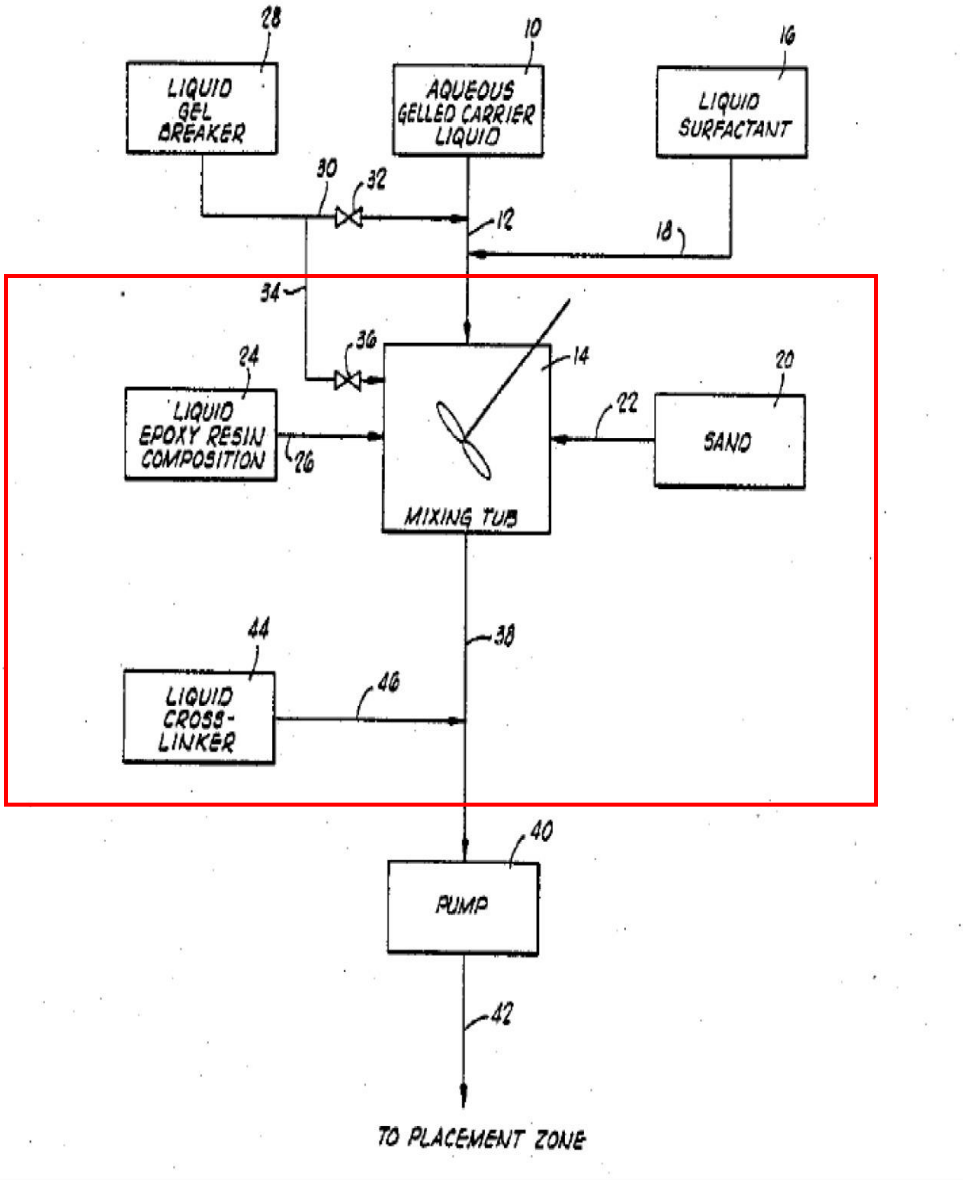


Figure 3.1: Review of coating method I

(R.Murphey & D.Totty, 1989) (Grood & D.Baycroft, 2010)

From figure 3.1, it can be clearly and simply noted that liquid epoxy resin composition and sand are added into the mixing tub. Liquid cross linker is then added into the mixer and the coated sand is pumped into the formation. This basic step is been implemented in the manufacturing the coated particles using different types of resin available.

3.1.2 References for Coating Method(s) Review

(Pilato, 2010) (Ina, 2011)

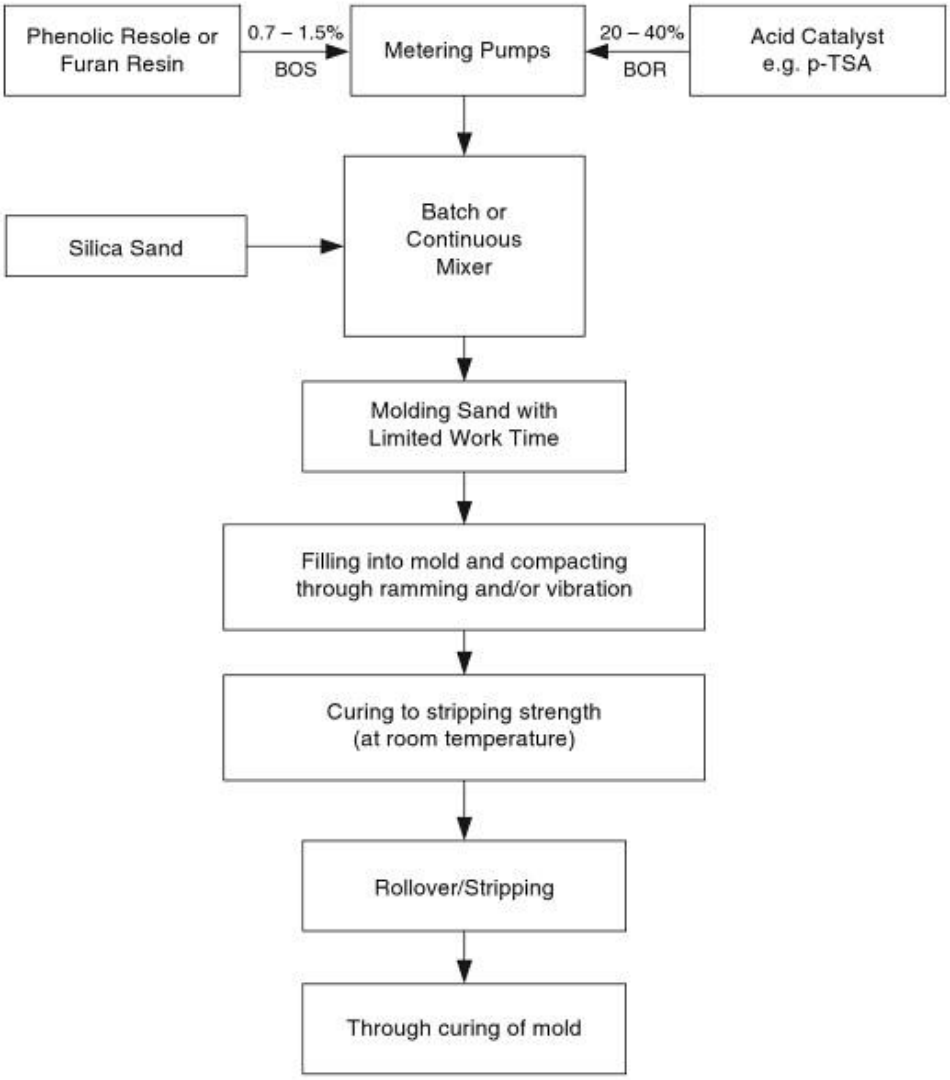
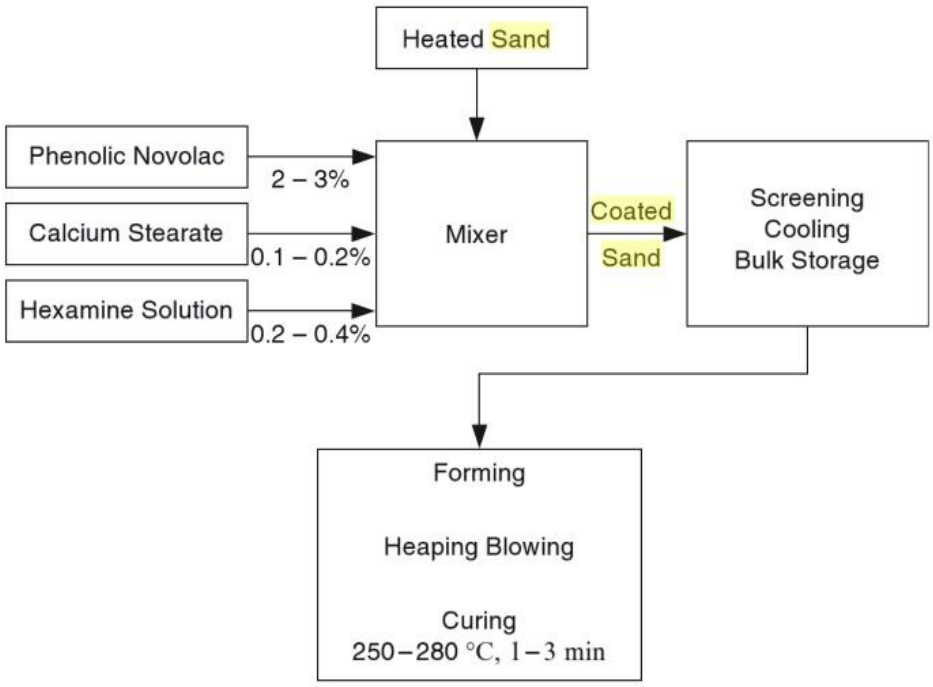


Figure 3.2: Review of coating method II (Pilato, 2010)

Figure 3.2 shows the coating method that is practical to be taken into consideration for manufacturing the coated particles. This method exploited similar step with the previous method in figure 3.1. First, catalyst, resin and sand are added into the mixer. Then, the sample is

molded and compacted. Next, the sample is cured at ambient temperature to boost it strength.

Schematic diagram below also shows the identical method of manufacturing the proppant sample. The sand is first heated to enhance its strength. Subsequently, the heated sand and resin together with the catalyst are adjoined into the mixer. After some time, the sample is cooled and shaped. Lastly, it is cured at high temperature 250-280^{o}C for 1-3 minutes. In one embodiment by Brannon, Wood, Rickards, & Stephenson (2010), recommend that curing temperatures for hardening ranging from about room temperature to about 200^{o}C., preferably 50^{o}C-150^{o}C. In another patented invention, the sand is heated at 150-160^{o}C. (Kawata, Takeshou, & Saikawa, Manufacturing Method for Phenolic Novolac Resin and Resin-Coated Sand, 2011)



Croning (also known as shell molding or resin coated sand) process for making cores and molds (schematic description)

Figure 3.3: Review of coating method III (Pilato, 2010, pp. 463-471)

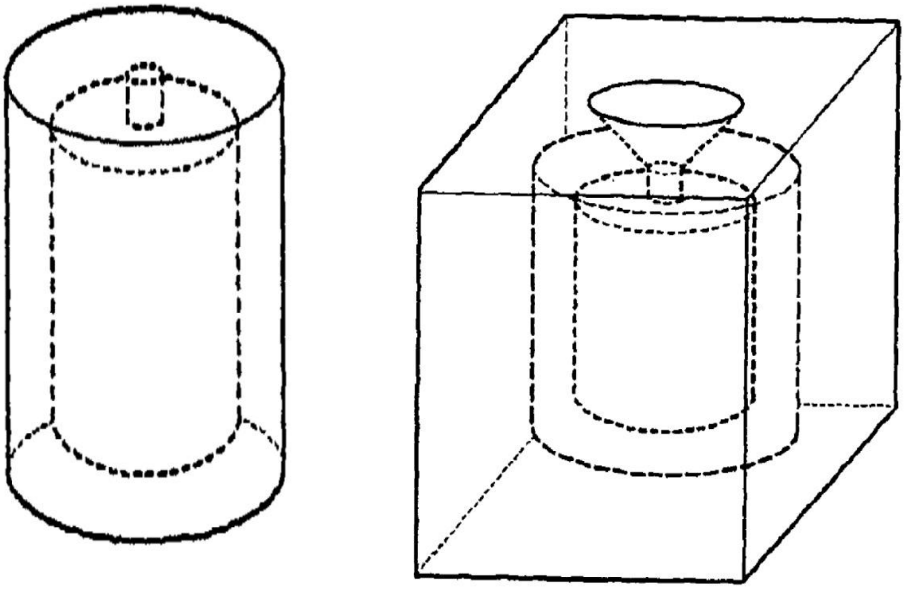


Figure 3.4: Spherical molding sand (Ina, 2011)

Study by Ina (2011) suggested that cylinder mold is one of the renowned and efficient method implemented in the industry. Thus, this suggestion will be relevant to be applied throughout the entire project.

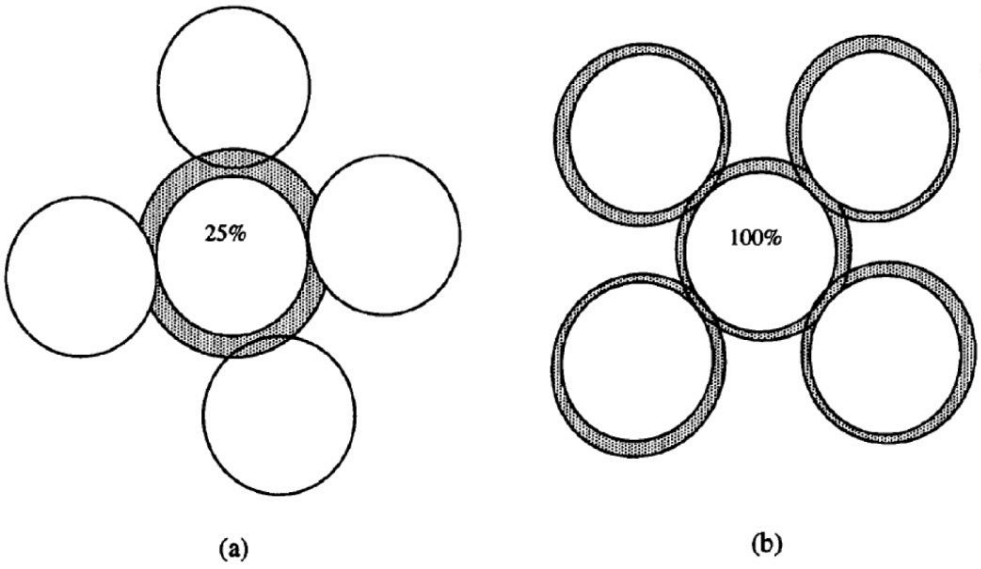


Figure 3.5: Illustration of resin coating particles

(D.Nguyen, D.Weaver, & D.Desai, 2007)

Figure illustrates a stylized view of the distinction between a traditional resins coating (b) and the resin coating of the present invention (a) (D.Nguyen, D.Weaver, & D.Desai, 2007). It is exemplified that present invention can reduce the amount of resin used for each sample while sustaining its linkage/bond. Yet, it is extremely economically attractive.

Therefore from the review that have been done, it can be conclude that the fundamental steps of manufacturing the proppant sample is admixing the powder particles to the resin coated substrate to embed or adhere to the continuous phase resin coating. Catalyst added act as cross linker for the mixing. Those methods of making utilize high temperature application of organic resin to sand in cycles requiring only a matter of minutes that yields high performance coated particles for the industry.

3.2 Methodology

Throughout this project completion, essential knowledge in performing the methodology of the sand coated resin sample is referred from the previous section and sub-sections which are **3.1 Reported/Researched Methodology, 3.1.1 Forming Resin Coated Particulate Materials and 3.1.2 References for Coating Method(s) Review.** Further understanding is attained from deep and thorough study of the subject matter.

Disclosed herein are coated particles methods of making the proppant sample. Each particle has a curable coating disposed upon a substrate. The substrate is a particulate substrate of an inorganic material, a substantially homogeneous formed particle of cured resin and filler (McCrary, Barajas, & McDaniel, 2009). The filler at this juncture is strictly refers to sand sample mentioned beforehand. Sand size selection is made after succinct study of few journals and patents. Brannon, Wood, Rickards, & Stephenson (2010) advocated that the preferable particle size are between from about 10-200 microns. Smaller scope patented by McCrary, Barajas, & McDaniel (2009) said that sand of 40/70 mesh is the most typical and frequent size utilized in the industry.

3.2.1 Sieve Analysis

The very first step in the project completion was begun with the sieve analysis. It is one of the crucial parts since the sand distribution of 40/70 mesh size particles is desired as stated earlier in the objective of the project. An appreciate amount of sand collected from Meraga, Terengganu is been washed and dried to remove the unwanted contaminants or substances. Then, the sieve analysis has been done using standard electric sieve shaker machine and sand of 40/70 mesh size is assembled since the best desired size of the sand particles is decided to be of the size 40/70 mesh size. The decision of employing 40/70 sand is suggested by

(McCrary, Barajas, & McDaniel, 2009) (Brannon, Wood, Rickards, & Stephenson, 2010). The corresponding sieve size/mesh size and the grain size are as follows:

Mesh Size (num. of holes/inch)	Grain Size (mm)
4	4.750
6	3.350
8	2.360
12	1.860
16	1.180
20	0.850
30	0.600
40	0.425
50	0.300
70	0.280
80	0.180
100	0.150
140	0.106
200	0.075
270	0.053

Table 3.1: Mesh size and grain size distributions

3.2.2 Preparation of the Sand Coated Resin Sample(s)

The core sample(s) is/are a randomly packed structure since the fabrication process is done manually. The design/shape of the samples are predetermined to be in cylindrical form due to the reference of the conventional core sample and the easiness of performing the testing afterward since the permeability test apparatus require size of 10cm height and 5.5cm diameter of core sample to be fixed in the apparatus's sleeve and the pressurized chamber. Cited beforehand, the cylinder shape core

sample is highly recommended through investigation by Ina (2011). As precaution, the mould dimensions used during the laboratory sessions are 15cm in height and 5.5cm in diameter. The method of preparing the proppant sample in the laboratory is based on combinations of approved journals study and is listed as below:

1) 420g of sand of 40/70 mesh size (McCrary, Barajas, & McDaniel, 2009) (Brannon, Wood, Rickards, & Stephenson, 2010) is weighted and separated into half; 210g each tray.
2) 20% of resin amount (McCrary, Barajas, & McDaniel, 2009) (Brannon, Wood, Rickards, & Stephenson, 2010) is calculated from sand weight; which yielded in 84g of resin.
3) Resin and its hardener are weighted appropriately to its fix ratio and premixed using an automatic mixer for approximately two minutes. (Grood & D.Baycroft, 2010) (R.Murphey & D.Totty, 1989) (Pilato, 2010)
4) First half of the measured sand is added into the mixer.
5) After 5 minutes, the remaining sand is added into the mixer.
6) 20ml of methanol (McCrary, Barajas, & McDaniel, 2009) is then added into the mixture to act as a catalyst and dilute the resin. (Grood & D.Baycroft, 2010) (R.Murphey & D.Totty, 1989) (Pilato, 2010)
7) After 5 minutes, the sand coated resin is ready to be moulded.
8) Once the sand coated resin is compacted into the mould, it is then positioned into the oven for 20 hours at 162^{o}C. (Brannon, Wood, Rickards, & Stephenson, 2010)
9) Steps 1 to 8 are repeated using different types of resin.

The ratios of the resin to the hardener used in the research/project are indicated as below:

Type of Resins	Ratio Resin : Hardener	Resin, weight (g)	Hardener, weight (g)
Epikote628	1:1	42.00	42.00
Epoxy	10:1	76.64	7.36
Polyester	2:1	56.00	28.00
Vinyl Ester	99:1	83.07	0.93

Table 3.2: Resin and hardener ratio

Attach below are the equipment used for the manufacturing of the sand coated resin sample(s).

Figure 3.6: Mixer

Figure 3.7: Sand coated sample

Figure 3.8: Sample is compacted into the mould

Figure 3.9: Proppant sample is placed into the oven

After four samples of sand coated resin have been successfully prepared, using Epikote626, Epoxy, Polyester and Vinyl Ester, the proppant samples are subjected to four types of tests with the aim of obtaining the desirable data to achieve the objectives of the project. The tests are namely, photomicrograph using scanning electron microscope (SEM), permeability test, porosity test and shear strength test. Flow chart below illustrates the project planning and activities. Brief experimental procedures will be discussed at the end of this section.

3.3 Flow Chart and Activities

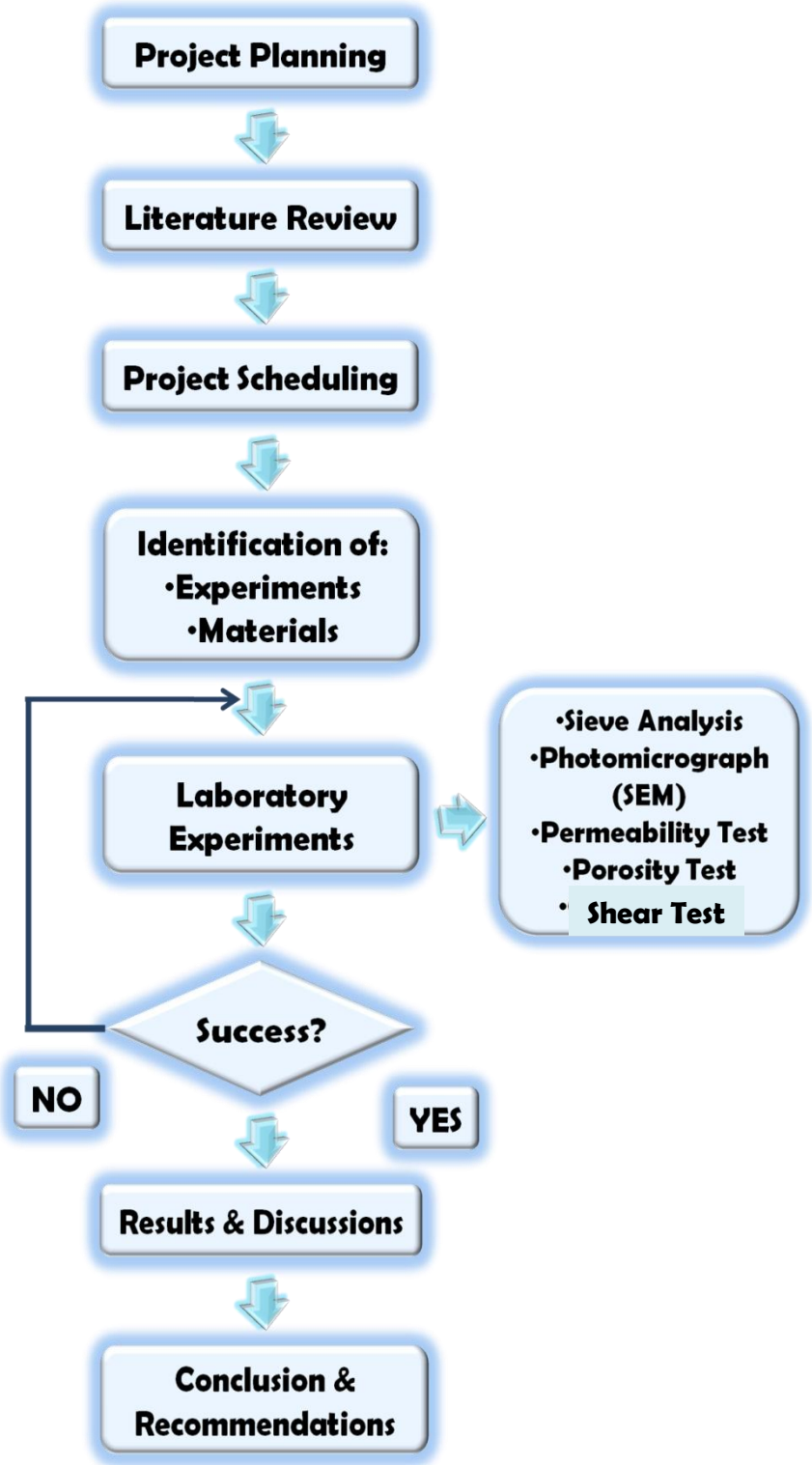


Figure 3.10: Project Flowchart

3.4 Project Gantt chart

	FYP 1				FYP 2			
	Sept	Oct	Nov	Dec	Jan	Feb	Mar	Apr
Topic Selection	X							
Background Studies		X						
Resin Types & Sand Assessment Technique		X	X	X				
Preliminary Report Preparation			X					
Submission of Prelim Report			X					
Proposal Defense			X					
Interim Report Preparation			X	X				
Submission of Interim Report				X				
Proppant Preparation & Testing					X	X	X	
Submission of Progress Report							X	
Results Analysis & Final Report Preparation						X	X	X
Poster presentation (Pre-EDX)								X
Submission of Final Report								X
Oral Presentation (viva)								X

Table 3.3: Project Gantt chart

3.5 FYP 1 Project Timeline

Project Timeline For Semester 1																
NO	**DETAILS/WEEK**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	Project Topic Selection															
2	Preliminary Research Work															
3	Submission of preliminary report															
4	**Project Work Continues**															
	a. Literature review on proppant and its usage in hydraulic fracturing and gravel packing															
	b. Checking on equipment availability															
5	Submission of interim report															
6	**Project work continues**															
	a. Literature review on sand															
	b. Collecting sands sample															
	c. Lab test planning															
7	Submission of interim report															
8	Proposal Defense															

Table 3.4: FYP 1 Project timeline

3.5.1 FYP 2 Project Timeline

Project Timeline For Semester 2																
NO	**DETAILS/WEEK**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	Sand cleaning and drying															
2	Sieving analysis															
3	Proppant manufacturing															
4	Porosity test															
5	Permeability test															
6	Photomicrograph															
7	Report and work researches															
8	Submission of progress report															
9	**Project work continues**															
10	Crushing test															
11	Pre-EDX presentation															
12	Preparation of final report															
13	Submission of final report															
14	Oral presentation															

Table 3.5: FYP 2 Project timeline

3.5.2 Experiments Purpose

FYP EXECUTION PLAN (EXPERIMENTAL)			
No.	**Experiment**	**Equipment Used**	**Experiment Purpose**
1	Sieve Analysis	Standard Electric Sieve Shaker	To obtain the desired 40/70 mesh size sand particle.
2	Photomicrograph	Scanning Electron Micrograph (SEM)	To have close view/clear image(s) that linking the resin and sand.
3	Permeability Test	Permeability Apparatus Setup (Matest)	To verify the permeability of the proppant core sample.
4	Porosity Test	Mercury Pressure Porosimetry (Thermo Scientific)	To measure the porosity of the proppant core sample.
5	Shear Test	Crush Test Equipment (Matest)	To determine the shear strength of the proppant core sample.

Table 3.6: Experiments purpose

3.6 Laboratory Experiments Conducted

As above mentioned in the project planning/activities, there are five forms of experiments that been conducted throughout the whole project. It is in order to achieve the objectives of the project which are to determine the desired physical behavior of local resin coated silica of 40/70 mesh size sand particles along with to investigate and analyze the characteristics/behavior and physical properties of the obtained sample(s) of local resin coated silica using different types of resin namely **Epikote628, Epoxy, Polyester** and **Vinyl Ester.**

3.6.1 Sieve Analysis

Standard electric sieve shaker device has been run during sieve analysis task so as to obtain an appropriate sand distribution. Before the chore is done, the sand sample is properly washed to ensure it is uncontaminated. Trays arrangement is set. Mesh size become smaller as it goes to the bottom. A significant amount of sand is poured uniformly on the top tray. The switch is on and after running the device for 5 minutes; the desired particle of 40/70 mesh size is then collected. The sand distribution size and mesh size has been tabulated in **section 3.2.1**. Shown is the image of electric sieve shaker equipment set.

Figure 3.11: Standard Electric Sieve Shaker

3.6.2 Photomicrograph

Equipment called scanning electron microscope (SEM) is run in order to perform this particular experiment. The procedure is incredibly simple. First, a small piece of the proppant sample of each resin type are vacuumed and coated with specific coating material known as aurum palladium. The vacuum and coating process are done simultaneously using the equipment. The process took approximately 30 minutes to be completed. Next, the coated and vacuumed sample is placed into the SEM chamber. The particle view of each sample is then clearly visualized on the computer screen. Hence, images of the scanned electron at the desired degree of view are captured. Illustrated below is the SEM equipment set available in UTP.

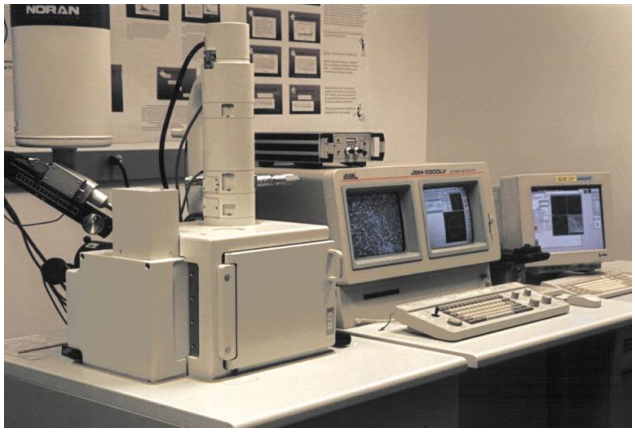


Figure 3.12: SEM Device

3.6.3 Permeability Test

Permeability test is done using the permeability apparatus by Matest. The core sample sized 10cm length and 5.5cm diameter is inserted into the plastic sleeve provided. The reason for the sample dimension to be 10cm long and having 5.5cm diameter is actually based on this equipment size. The core is then sealed in the pressure chamber surrounded with hydraulic fluid. On each side of the chamber, there are two openings. One is subjected

to water flow tube; inlet and the other one is free opening; outlet. The chamber is pressurized up to ≈5000kN and the pressure is known as confined pressure and denoted as ω^3. Water is pumped and injected through the core sample at 1000kPa. The whole core is flooded with water to pass through. The amount of water passed through the core sample for one minute is collected at the outlet end and the accumulated water volume is recorded. The experiment is repeated using the four core samples and the result for each core sample is recorded. The calculation to determine the permeability is done by applying Darcy Equation. Darcy's law is generally assumed to be valid for pressure driven flow of fluids through packed particles (German, Particle Packing Characteristics, 1989, p. 361). Illustrated below is the permeability apparatus set.

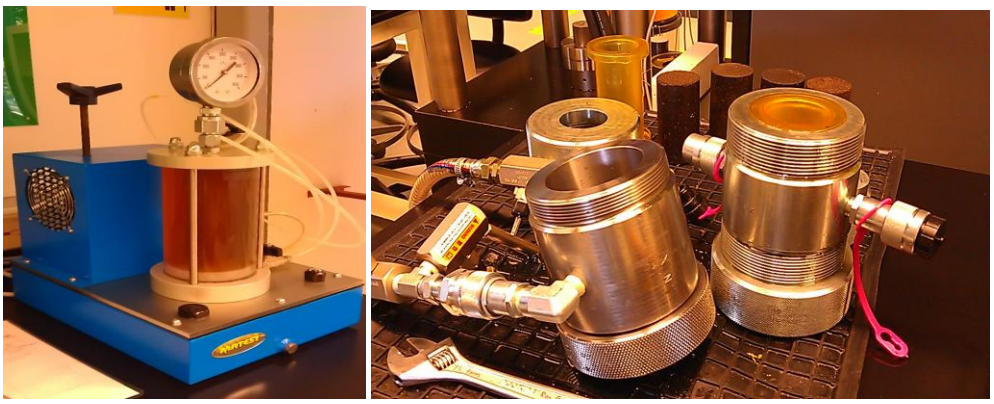


Figure 3.13: Permeability apparatus set

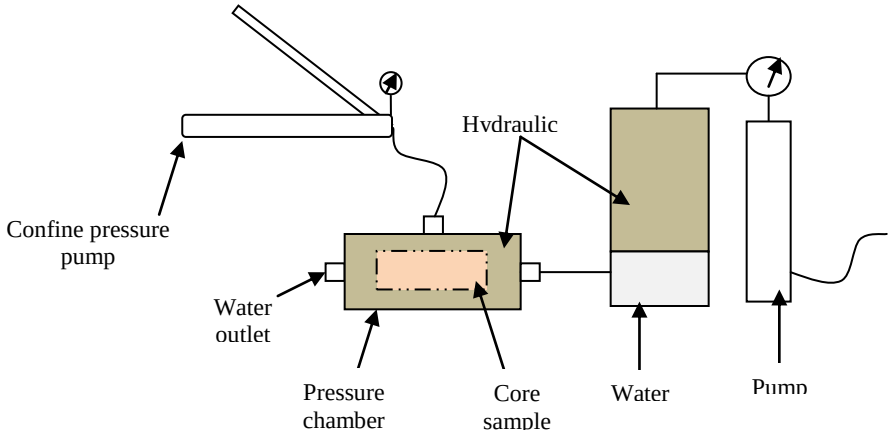


Figure 3.14: Schematic diagram of permeability apparatus set

3.6.4 Porosity Test

As for the porosity test, mercury pressure porosimetry apparatus set is set up and run to obtain the required readings. The equipment will automatically generate the results once the simulation completed. The experiment procedures only require a few steps. First, each of the proppant sample's density is determined using method and apparatus as pictured below.

Figure 3.15: Electronic weighting scale

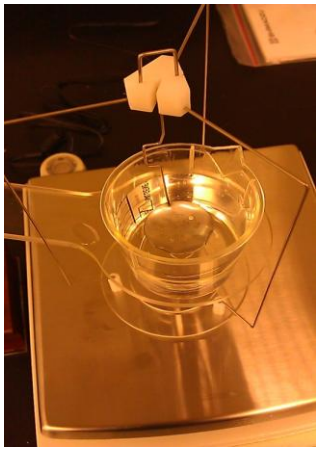


Figure 3.16: Top view of the apparatus

Density calculation steps:

1. Sample's weight in air is taken and recorded.
2. Sample's weight in water is measured and recorded.
3. Density of the sample is calculated.

$$\rho = \frac{weight\ in\ air - weight\ in\ water}{weight\ in\ air}$$

Subsequently, required data needed such as sample density and weight for the simulation to be accomplished is keyed in the software. The apparatus is set as below:

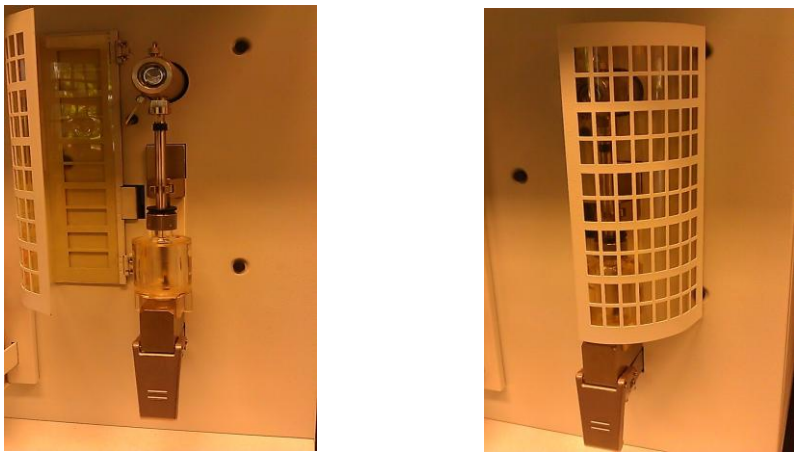


Figure 3.17: Sample is put into the dilatometer

Figure 3.18: Front view of the equipment; 400MPa pressure chamber (left), 200kPa pressure chamber (right)

3.6.5 Shear Test

The provided white cement is mixed and poured into the specific mould. Proppant core sample is placed as illustrated half buried in the mould. The sample is then sunbaked until it completely dry. Another considerable amount of cement is shaped on top of the sample and as well sunbaked. The final sample is placed into the equipment chamber and the sample is sheared. The equipment is then generated the shear stress value of the particular proppant core sample. Experiment is repeated to acquire all four sample strength value. Rendered below is the illustration of the experiment method.

Figure 3.19: Shear test apparatus set

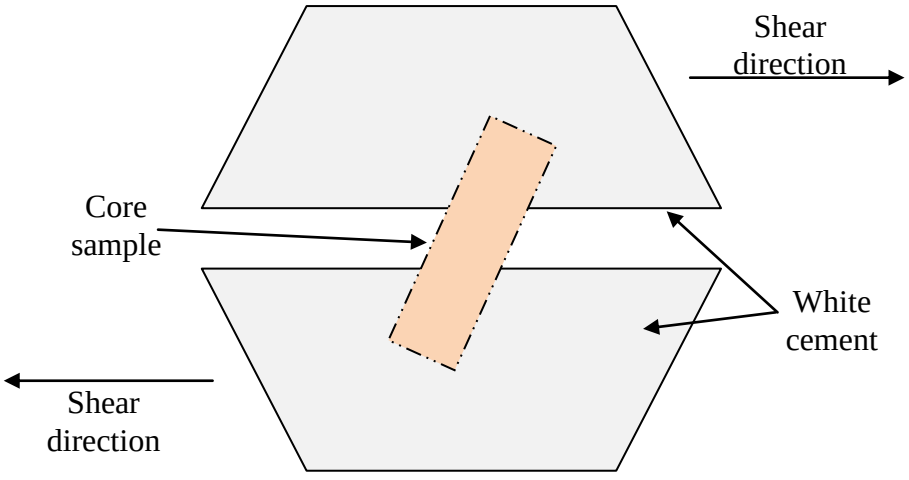


Figure 3.20: Illustration of shear test

CHAPTER 4

RESULTS & DISCUSSIONS

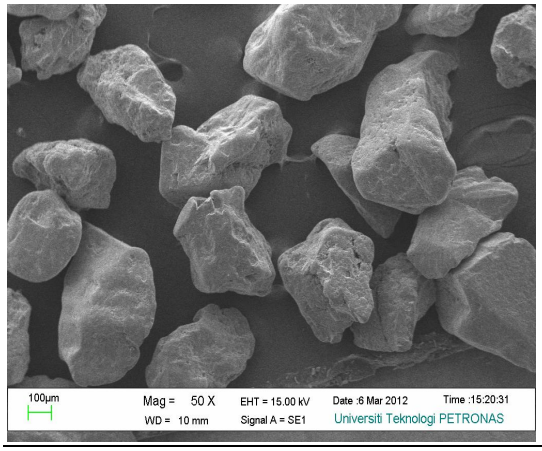
Theories and concepts applied for each experiments performed will be briefly discussed in this section. Results are calculated and tabulated. Final result for sample testing is to be competent to point out a core; resin coated sand sample using suitable type of resin with specific predetermined composition which possesses desired characteristic e.g. high permeability and porosity values to act as a filter to enhance the properties of sand proppant besides providing a cheap supply of sand proppant for local field applications.

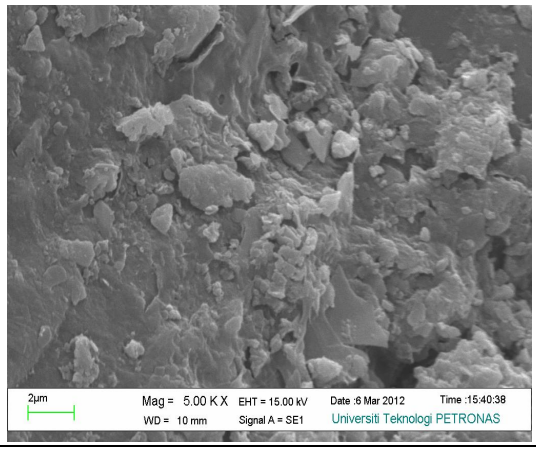
4.1 Photomicrograph

During compaction, there are permanent deformations or fracturing of the particles (German, Particle Packing Characteristics, 1989, p. 400). Thus, the SEM analysis will help to visualize the deformation so that it can be clearly observed. Fracture in this case has occurred along the particle contacts yet, they remain the weak links in the compact. Images captured using SEM device provided by UTP.

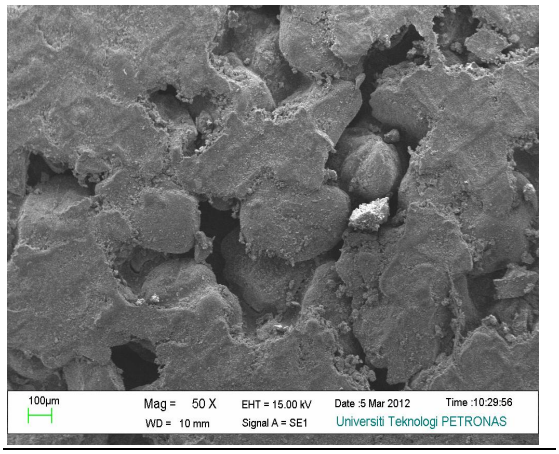
Each resin sample images show the measured view of 100 μm and 1 μm. The images captured have been viewed thoroughly. From the images obtained, it can be observed and compared to the other resin type, that **vinyl ester** resin exhibits the highest potential of good coating properties. Vinyl ester image showed the best particle bonding with little interlocking between particles which allow the sample to have high porosity and permeability value. Images generated by vinyl ester resin sample is more likely resemble the suggested sand coating as mentioned in section 3.1.1 figure 3.5.

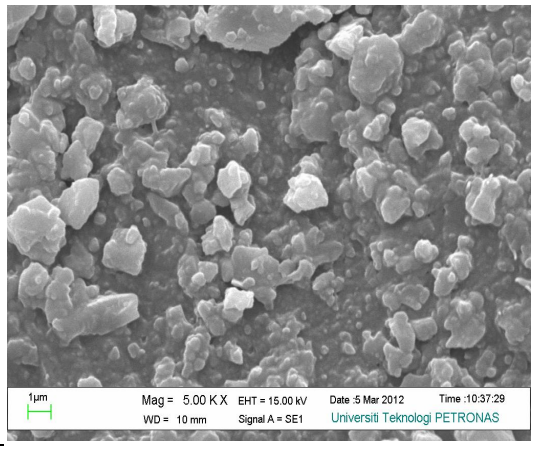
Raw sand



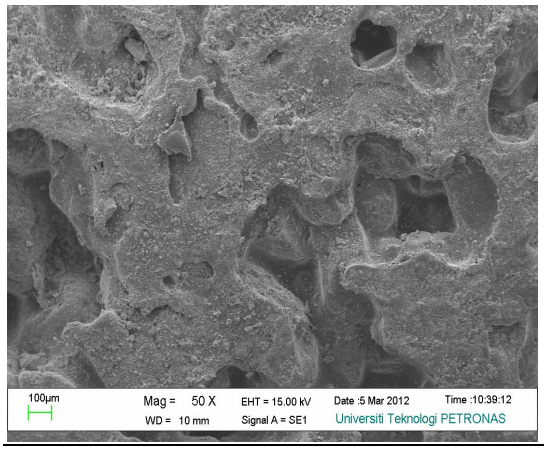


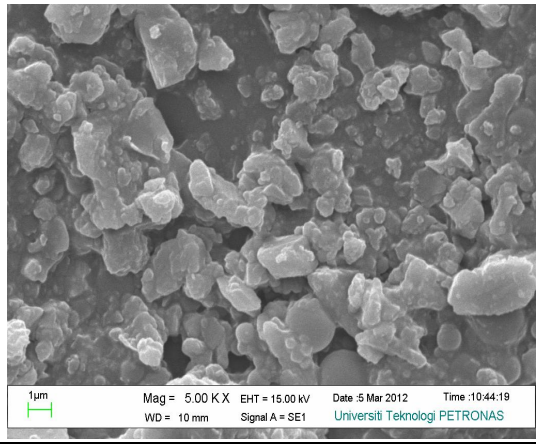
Epikote628



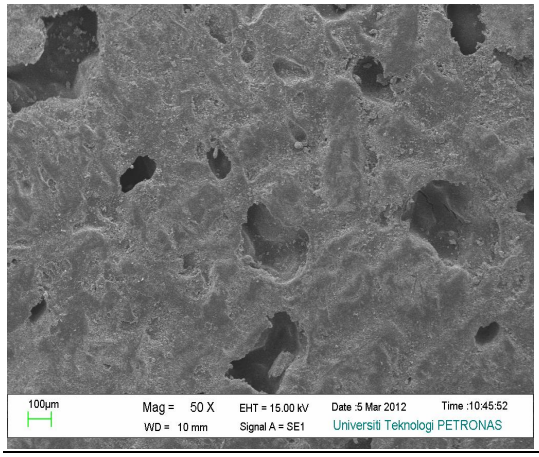


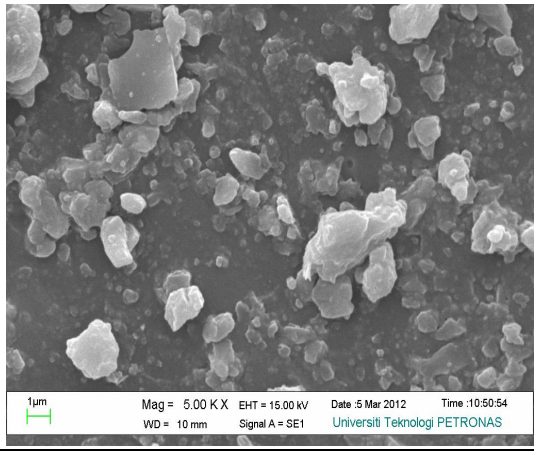
Epoxy



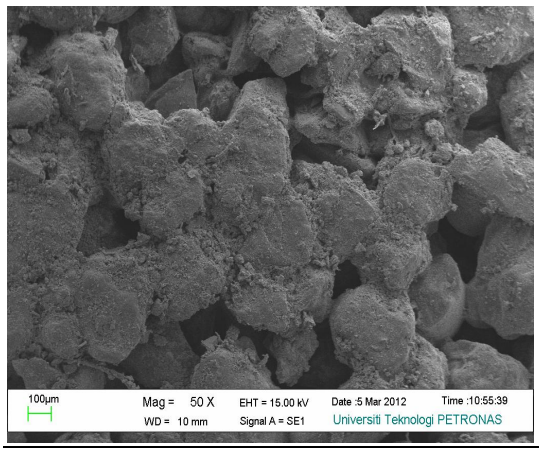


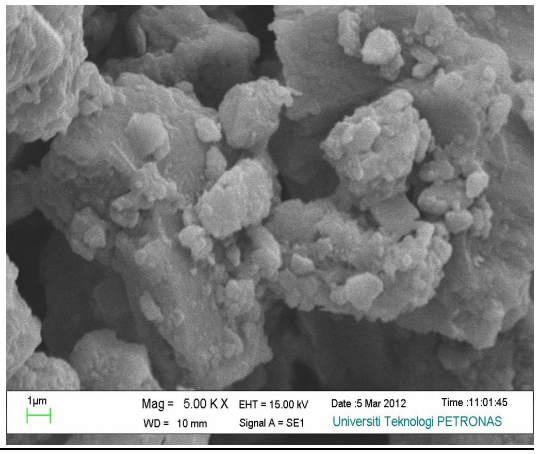
Polyester





Vinyl Ester





4.2 Porosity Test

Many solid materials both natural and manufactured contain a certain internal volume of empty space. This is distributed within the solid mass in the form of pores, cavity and cracks of various shapes and sizes. The total sum of these void volumes is called porosity. Porosity strongly determines important physical properties of materials such as durability, mechanical strength, permeability, adsorption properties etc. Mercury Pressure Porosimetry by Thermo Scientific provide accurate porosity measurement, pore size distribution – total pore size; intra pore size, particle size distribution, external surface area distribution, etc.

Mercury porosimetry technique involves very high pressure generation, up to 400MPa. Despite being a liquid, at such high pressure mercury is submitted to a certain compression degree. Additionally, the glass of the sample holder, the dielectric oil and other parts under high pressure, change also their properties. The sum of all these side effects generates the so called blank curve that is similar to a penetration curve. Therefore, the blank must be measured with a complete analysis without the sample and then subtracted from the penetration curve. The resulting corrected spectrum now represents the real sample porosity. In case the blank is not properly measured and subtracted, the mercury porosimetry results can lead to big mistakes in pore volume and surface area determination, especially in case of narrow pores with a small pore volume.

The principle of the technique is based on the fact that mercury behaves as a non-wetting liquid toward most substances. This technique is not advisable when the sample contains metals reacting with mercury e.g. gold aluminum etc. and forming amalgam. Mercury is forced to enter into the pores by applying a controlled increasing pressure. As the sample holder is filled with mercury under vacuum conditions (mercury surrounds the sample without entering the pores due to the very low residual pressure), during the experiment, the pressure is increased and the volume of mercury penetrated is detected by means of a capacitive system. The

decreasing volume of the mercury in the sample holder represents the pore volume. The method is based on the capillary depressurization phenomenon.

The result for this experiment is attached as per **Attachment.** Nevertheless**,** the result is then simplified as below:

Sample Type	***Porosity,%***
Epikote628	18.46
Epoxy	22.00
Polyester	14.59
Vinyl Ester	**22.37**

Table 4.1: Porosity value

It can be deducted that **vinyl ester** resin sample shows the highest and most desired value of porosity.

4.3 Permeability Test

Permeability is defined as the ability of a porous media to effectively transmit fluid. The case of laminar flow of a viscous fluid through pores is the most widely encountered case in practice. Darcy's law proves applicable where the velocity of flow is directly proportional to the gradient in applied pressure. For an incompressible fluid the appropriate form of Darcy's law is (German, 1989, p. 355):

$$q = \frac{k\ A\ \ \Delta P}{\mu\ L}$$

Where,

q = flow rate, cc/sec

k = permeability, mD

A = cross sectional area, cm^2

ΔP = pressure difference over the distance L

μ = viscosity, cp

L = length, cm

The permeability characteristics depend on the particle packing. In a randomly packed structure, there is no order and the permeability is isotropic. In contrast, an ordered particle packing is not isotropic and the permeability is dependent on the direction of flow with respect to the packing orientation. The permeability is very sensitive to the porosity and pore size; small changes in either characteristic will induce major changes in the permeability. Several techniques are applicable to permeability measurements of packed particles. Most typically the permeability is measured by determining the fluid flow volume, mass or velocity versus the applied pressure differential (German, 1989, p. 356).

For this particular project, permeability apparatus which utilize water as a medium of flow is preferred over the apparatus is using helium,He gas as a medium of flow.

It is in order to avoid the slip flow or klikenberg effect that might occur during gas flow. Given that the medium of flow is water, though there is no need to apply the modified Darcy equation. Thus, to calculate the permeability,k the original Darcy equation is then rearrange:

$$k = \frac{q\ \mu\ L}{A\ \ \Delta P}$$

The experiment is conducted and the result obtained is tabulated as below:

Sample Type	***Accumulated Volume Of Water, ml***
Epikote628	15.0
Epoxy	21.0
Polyester	0.0
Vinyl Ester	51.0

Table 4.2: Volume of water collected

Parameters and calculations:

Viscosity,μ:

Viscosity is 1.0 cp since water is being used.

Length, L:

Length of each core is identical, 10.0 cm

Area, A:

$$A = \pi r^2$$

$$A = \pi(5.5)^2$$

$$A = 95.03cm^2$$

Pressure, P:

$5kN/m^2$ = 0.725188689 psi (ACCELWARE, 2009)

Flow rate, q:

$$q = \frac{volume\ of\ water, ml}{time, sec}$$

Sample Type	*Flow rate, q (cc/sec)*
Epikote628	0.25
Epoxy	0.35
Polyester	0.00
Vinyl Ester	0.85

Table 4.3: Flow rate value

The necessary value is inserted into the equation and **permeability value** obtained is calculated and recorded as follow:

Sample Type	*Permeability,k (mD)*
Epikote628	36.0
Epoxy	51.0
Polyester	0.0
Vinyl Ester	**123.3**

Table 4.4: Permeability value

From the result calculated, it can be conclude that **vinyl ester** sample shows the highest and best permeability value.

4.4 Shear Test

Shear strength test is done to measure the proppant strength whether it can stand the pressure exerted in the wellbore when injected. In German (1989) studies, it is reported that particle compaction creates bonding planes at the interfaces, greatly increasing the inter-particle strength. Initially, local deformation is caused by compaction as the result of flattening surface asperities. For compacted particles, the interfacial bonding becomes a key determinant of the strength (p. 400). Further study by German, it is found that, during compaction, shear and deformation are localized at these particle contacts. Deformation increases the bonding area and the number of bonds, and disrupts surface films that otherwise inhibit bonding. Thus, interparticle bonding improves as the compaction forces increase. (pp. 400-401). Strength is enhanced when particles are compacted as well as increasing the number of contacts and the interfacial bond area (German, 1989, p. 408).

The size and quality of the contact zones and the fractional density are the factors that affect the strength of compacted powders. Shear stresses at the interparticle contacts are important to attain high strengths due to surface contamination. The initial deformation improves strength in the proportion to the cube of the contact size (German, 1989, p. 401). The strength of the compacted particles is dictated by the strength of the interface between particles. In German's (1989) writings, he stated that the interparticle strength is most dependent on the shear stresses during compaction. But failure occurs when the stress at the interparticle bond reaches a critical value that varies with the combination of normal and shear stresses. Because of this effect, it is common to use wide particle size distribution to mix the benefits of large particles; good compactability and small particles; higher strength (German, 1989, p. 405). As such, selection of 40/70 particle size for this project is appropriate.

Tabulated below are the results obtained from the shear test performed:

Sample Type	*Shear Stress, kN*
Epikote628	38.0
Epoxy	36.0
Polyester	43.0
Vinyl Ester	36.0

Table 4.5: Shear stress value

Conventional reservoir pressure for local field is known to be around 2000 psi. Accordingly, local sand proppant sample must exceed this value in order for it to be compatible for local field proppant application. Converted force value obtained from the test to its corresponding pressure are listed as below: (Pressure Profile System Inc., 2010)

Sample Type	*Shear Stress, kN*	*Pressure, psi*
Epikote628	38.0	2319.793
Epoxy	36.0	2197.699
Polyester	43.0	2625.029
Vinyl Ester	36.0	2197.699

Table 4.6: Converted pressure value

Experimentally, all proppant samples strength value exceeds 2000 psi. Thus, it is clear that the manufactured proppant samples are able to stand the conventional reservoir pressure condition or in short, indicates that the local sand proppant is compatible to be injected into the wellbore.

CHAPTER 5

CONCLUSIONS & RECOMMENDATIONS

The research relates generally to methods and proppant coating materials for reducing mechanical erosion damage of the pumping components during downhole operations and for extending the life of the equipment used. As well to overcome those arising problems e.g. extremely high well maintenance cost due to massive sand productions, limitation of natural sand supply, and limited proppant current application. Broadly speaking, the proppant substrate may consist of any number of materials, including sand, ceramic, glass beads etc. Nonetheless, for this scheme, local sand from Meraga, Terengganu is being utilized. In particular aspects, the research relates to the composition of proppant used in sand control operations or productions and physical properties testing processes for resin coated sand using different types of resin. As for this final year project (FYP), all resin coated sand samples are prepared and tested using well equips facilities provided by Universiti Teknologi PETRONAS. FYP execution plan (experimental) have been conducted efficiently due to the easy access facilities.

As for this particular project, four core samples have successfully been manufactured. The manufacturing procedure was done manually using conventional cylinder mould yielding to randomly packed structures. The study of particle packing involves several simplifying assumptions concerning the nature of the particles. The packing can be influenced by secondary factors such as the container shape and size or the procedure used in assembling the packing (German, 1989, pp. 413,415). Though, it is highly recommended that in further studies of this subject matter, precise and proper particle packing method and characteristic should be prioritized and looks into details. Consequently, accurate properties testing result could be obtained, which leads to finer proppant invention. The two dominant features of

packed particles are the porosity and the particle size. The resistance to fluid flow increase rapidly as the porosity decrease.

With mixed particle sizes, the strength is dependent on the size of the smallest particle that forms a continuous matrix in the compact. Thus, the width of the particle size distribution is not a direct gauge of the strength. Consequently, connectivity concepts are important in understanding the strength of particulate systems with wide size distributions. The effect of the particle shape is seen in the degree of mechanical interlocking, as is evident in scanning electron micrograph section shows the fracture surface of the sand coated resin after compaction.

It is clear that the material and particle characteristics influence strength characteristics. In order to overcome the difficulties with low strength compacts of hard particles it is common to add cohesive fluids. In this project, is referring to the resin itself. As a good example of this, sand castles at beach are much stronger when constructed from wet sand. It is recommended to fabricate the core sand sample in a proper compaction and pressure applied.

Yet, due to tight schedule and time constraint, permeability test is been ran only once. Accordingly, it is recommended to repeat the permeability test at least trice to obtain extra accurate data and average readings could be rendered. Subsequently, the position of the core that been placed in the pressure chamber also needs to be put into consideration. The position of the core is meant by the position of the core being injected with water. Since the core is manually self fabricated, so the particle packing and compaction during the fabrication process might differ from side to side of the top and bottom of the same core. Recommendation for this permeability test is to compare the permeability value of the same core sample injected through its top or its bottom. Then only the comparison of permeability value of different sand coated resin sample is been made.

Continuous and further researches are required in order to develop deep understanding of the subject matter. New findings are extremely crucial in leading the project to a success.

Apart from upholding wise time management and frequent consultation with assigned supervisor for concise guideline, an effective two ways communications with laboratory technicians/demonstrators also had been put into practice to ensure the smoothness towards the project accomplishment. All together, the above-mentioned suggestions indicate a superior step towards a success in this final year project.

As a wrap, **Vinyl Ester** resin sample is highly recommended for application since the experimental results obtained, show the most preeminent bonding linkage between particles as well as most desirable value for porosity, permeability and shear stress. In view of the fact that the local sand tested is compatible with the resin, hence cheap proppant supply for local field can be commercialized. Prior to commercializing the local sand proppant for field applications, field pilot test for physical and chemical properties to support the feasibility of the completion are essential e.g. surface morphology and internal microporosity, crushing behavior and crystalline structure, friction angle, fluid salinity and mechanochemical stability, fluid temperature and hot brine aggressivity, equipment abrasion and grain hardness, specific gravity and fluid suspension properties, grain size and embedment, manufacturing process and pellet composition as well as conductivity discount.

CHAPTER 6

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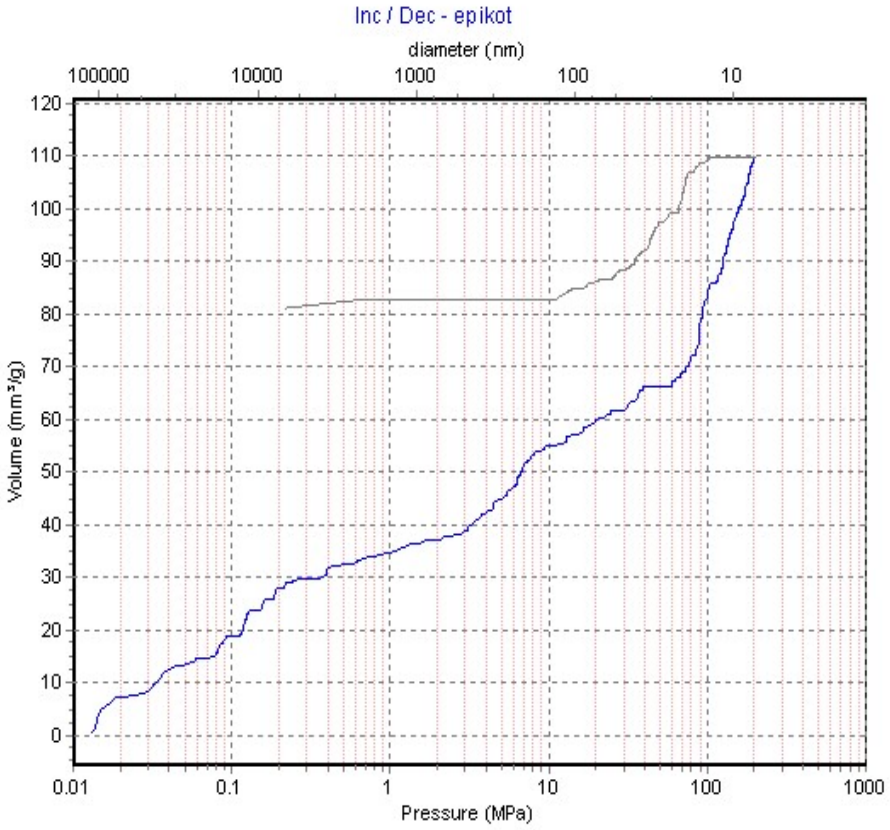
Attachments

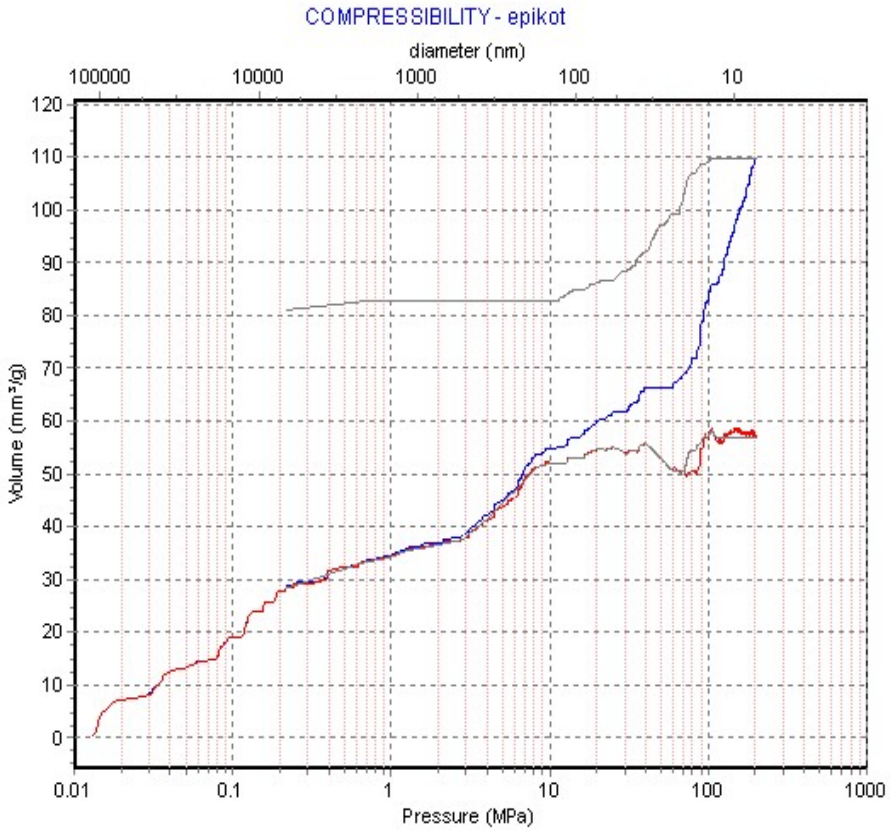
Epikote628

Source file:	C:\Thermo Scientific\Data\sample1(R).142
SOLID REPORTING	
Company name:	utp
Operator:	fifi
TEST INFORMATION	
Test date:	01-03-12
Sample name:	epikot
Comment:	meraga
Sample mass (g):	0.46
Sample real density (g/cm³):	1.77
Test file name:	C:\Thermo Scientific\Data\sample1_200MP_120301.P24
Combined with file (140):	C:\Thermo Scientific\Data\sample1_400kp_120301.P14
Mercury surface tension (N/m):	0.48
Mercury contact angle (°):	140.0
Test filling volume (mm³):	470.0
Starting hydr. press. of test (MPa):	0.0125
(Dil+Hg+Sample) weight (g):	243.21
Corrected weight (Dil.+Hg+Sample) (g):	243.467
ANALITICAL CONDITIONS	
Maximum test pressure (MPa):	200
Increase speed:	7
Increase method:	Pascal
Decrease speed:	7
Decrease method:	Pascal
Temperature of test (°C):	25.0
Mercury density @ test (g/cm³):	13.534
BLANK & DILATOMETER INFORMATION	
Blank date:	30-12-99
Blank filename:	C:\Thermo Scientific\Data\blank809\blank809_200MP_I7D7_110222.P24
Comment	
Blank Max pressure (MPa):	200
Blank Increase speed:	7
Blank Increase method:	Pascal
Blank Decrease speed:	7
Blank Decrease method	Pascal
Dil. number:	809
Dil. type:	CD3
Dil. Cone length (mm):	22
Dil. Electrode gap (mm):	5
Dil. stem radius (mm):	1.5
Dil. weight (g):	44
Temperature of blank (°C):	25
Mercury density @ blank (g/cm³):	13.534
Blank filling volume (mm³):	489



RESULTS WITHOUT COMPRESSIBILITY CORRECTION

Total intruded volume (mm³/g):	109.79	at pressure of (MPa):	200.6115
Intrud. Vol. (mm³):	11.95		
Envelope density (g/cm³):	1.6813		
Bulk density @ pressure (g/cm³):	1.7581	at pressure of (MPa):	0.174
Apparent density (g/cm³):	2.0619	at pressure of (MPa):	200.6115
Void volume by real density (mm³/g):	29.81		
Accessible porosity (%):	18.46		
Inaccessible porosity (%):	-13.45		



RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N):	Yes		
Linearity range:	From (MPa): 160.4892	To (MPa):	200.6115
Correction factor (mm³/g.MPa)	0.262908		
Compressibility factor (MPa.g/mm³)	3.8036		
Compressed volume (mm³/g):	52.74		
Sample compressibility (1/MPa):	2.395E-3		
Bulk modulus (MPa):	4.176E+2		
Total intruded volume (mm³/g):	57.05 at pressure (MPa):		200.6115
Intruded vol. (mm³):	11.93		
Envelope density (g/cm³):	1.6813		
Bulk density @ pressure (g/cm³):	1.7579	at pressure (MPa):	0.1735
Apparent density (g/cm³):	1.8597	at pressure (MPa):	200.6115
Real void volume by real dens. (mm³/g):	29.81		
Accessible porosity (%):	9.59		
Inaccessible porosity (%):	-4.58		

Epoxy

Source file:	C:\Thermo Scientific\Data\sample2(R).142

SOLID REPORTING

Company name:	utp
Operator:	fifi

TEST INFORMATION

Test date:	02-03-12
Sample name:	epoxy
Comment:	meraga
Sample mass (g):	0.22
Sample real density (g/cm³):	1.47
Test file name:	C:\Thermo Scientific\Data\sample2_200mp_120302.P24
Combined with file (140):	C:\Thermo Scientific\Data\sample2_400kp_120302.P14
Mercury surface tension (N/m):	0.48
Mercury contact angle (°):	140.0
Test filling volume (mm³):	484.0
Starting hydr. press. of test (MPa):	0.0127
(Dil+Hg+Sample) weight (g):	244.6
Corrected weight (Dil.+Hg+Sample) (g):	244.668

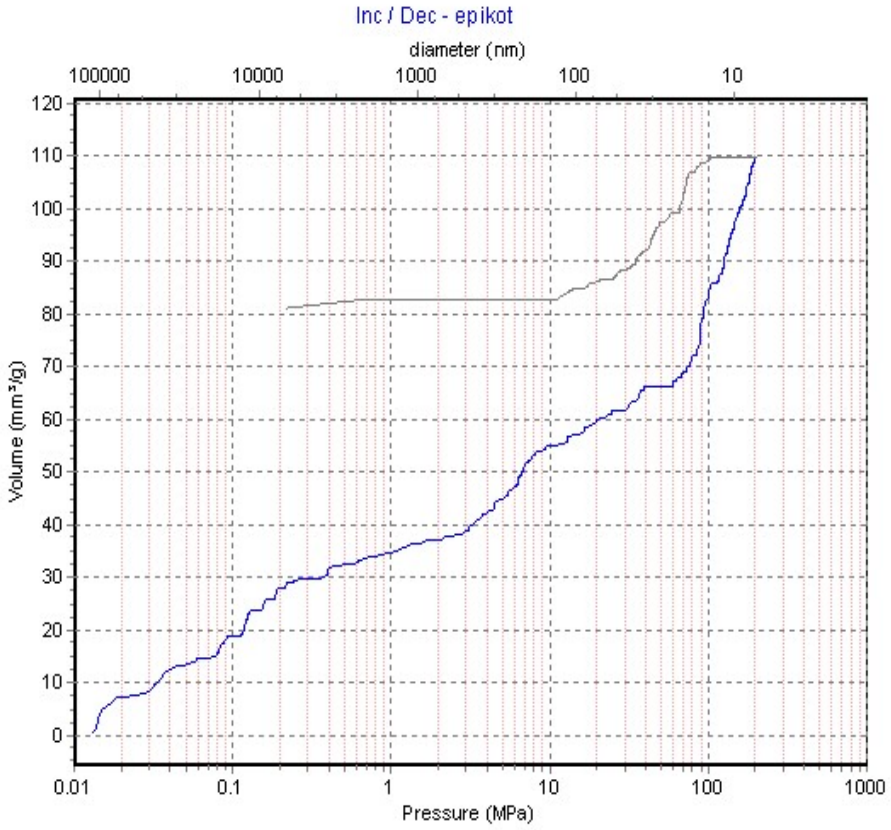
ANALITICAL CONDITIONS

Maximum test pressure (MPa):	200
Increase speed:	7
Increase method:	Pascal
Decrease speed:	7
Decrease method:	Pascal
Temperature of test (°C):	25.0
Mercury density @ test (g/cm³):	13.534

BLANK & DILATOMETER INFORMATION

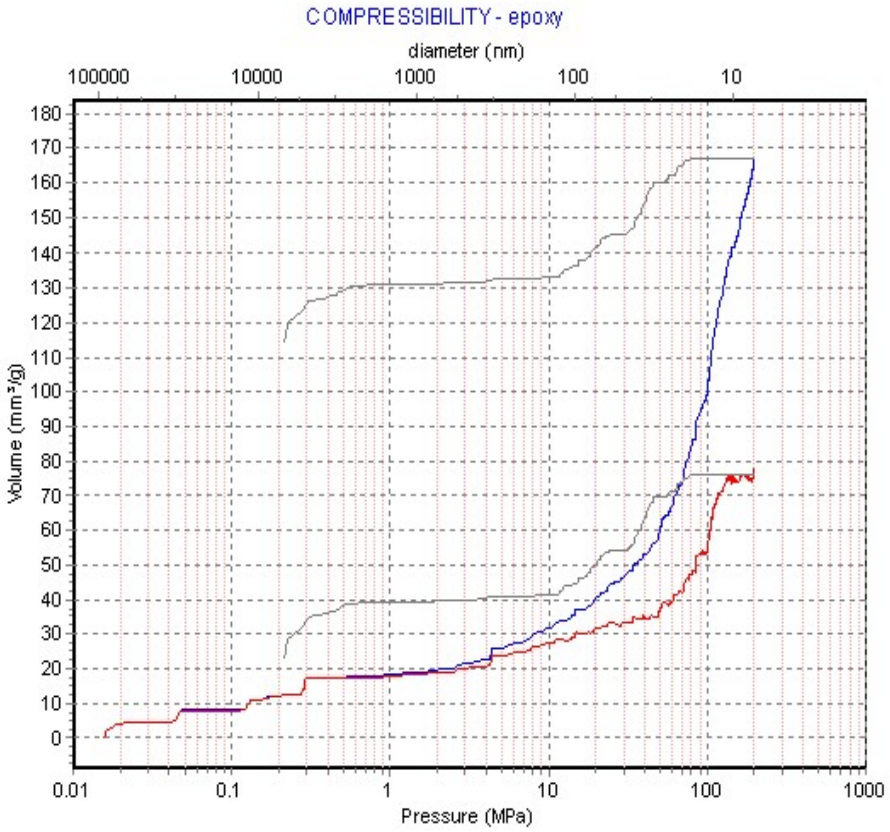
Blank date:	30-12-99
Blank filename:	C:\Thermo Scientific\Data\blank809\blank809_200MP_I7D7_110222.P24
Comment	
Blank Max pressure (MPa):	200
Blank Increase speed:	7
Blank Increase method:	Pascal
Blank Decrease speed:	7
Blank Decrease method	Pascal
Dil. number:	809
Dil. type:	CD3
Dil. Cone length (mm):	22
Dil. Electrode gap (mm):	5
Dil. stem radius (mm):	1.5
Dil. weight (g):	44
Temperature of blank (°C):	25
Mercury density @ blank (g/cm³):	13.534
Blank filling volume (mm³):	489

SAMPLE RESULTS



RESULTS WITHOUT COMPRESSIBILITY CORRECTION

Total intruded volume (mm³/g):	167.14	at pressure of (MPa):	200.5118
Intrud. Vol. (mm³):	2.62		
Envelope density (g/cm³):	1.3161		
Bulk density @ pressure (g/cm³):	1.3371	at pressure of (MPa):	0.174
Apparent density (g/cm³):	1.6873	at pressure of (MPa):	200.5118
Void volume by real density (mm³/g):	79.54		
Accessible porosity (%):	22.00		
Inaccessible porosity (%):	-11.53		



RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N):	Yes			
Linearity range:	From (MPa):	160.4094	To (MPa):	200.5118
Correction factor (mm³/g.MPa)	0.454870			
Compressibility factor (MPa.g/mm³)	2.1984			
Compressed volume (mm³/g):	91.21			
Sample compressibility (1/MPa):	2.721E-3			
Bulk modulus (MPa):	3.675E+2			
Total intruded volume (mm³/g):	75.94 at pressure (MPa):		200.5118	
Intruded vol. (mm³):	2.60			
Envelope density (g/cm³):	1.3161			
Bulk density @ pressure (g/cm³):	1.3369	at pressure (MPa):	0.1735	
Apparent density (g/cm³):	1.4623	at pressure (MPa):	200.5118	
Real void volume by real dens. (mm³/g):	79.54			
Accessible porosity (%):	9.99			
Inaccessible porosity (%):	0.47			

Polyester

Source file:	C:\Thermo Scientific\Data\sample3(R).142

SOLID REPORTING

Company name:	utp
Operator:	fifi

TEST INFORMATION

Test date:	05-03-12
Sample name:	polyster
Comment:	meraga
Sample mass (g):	0.58
Sample real density (g/cm³):	1.76
Test file name:	C:\Thermo Scientific\Data\sample3_200mp_120305.P24
Combined with file (140):	C:\Thermo Scientific\Data\sample3_400kp_120305.P14
Mercury surface tension (N/m):	0.48
Mercury contact angle (°):	140.0
Test filling volume (mm³):	479.0
Starting hydr. press. of test (MPa):	0.0126
(Dil+Hg+Sample) weight (g):	242.94
Corrected weight (Dil.+Hg+Sample) (g):	243.075

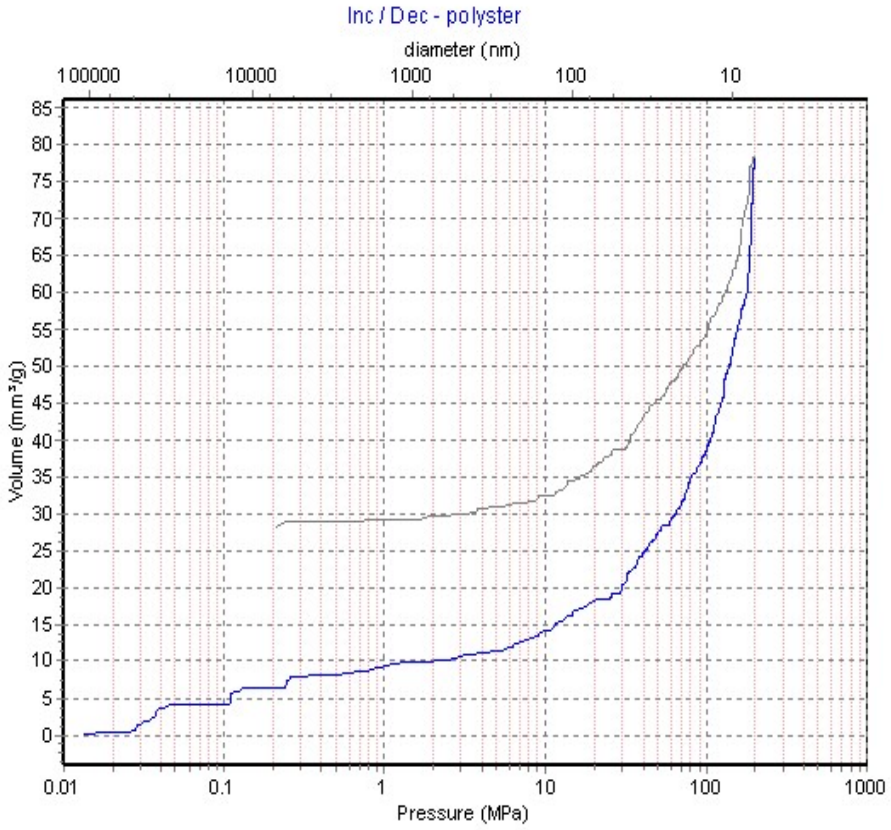
ANALITICAL CONDITIONS

Maximum test pressure (MPa):	200
Increase speed:	7
Increase method:	Pascal
Decrease speed:	7
Decrease method:	Pascal
Temperature of test (°C):	25.0
Mercury density @ test (g/cm³):	13.534

BLANK & DILATOMETER INFORMATION

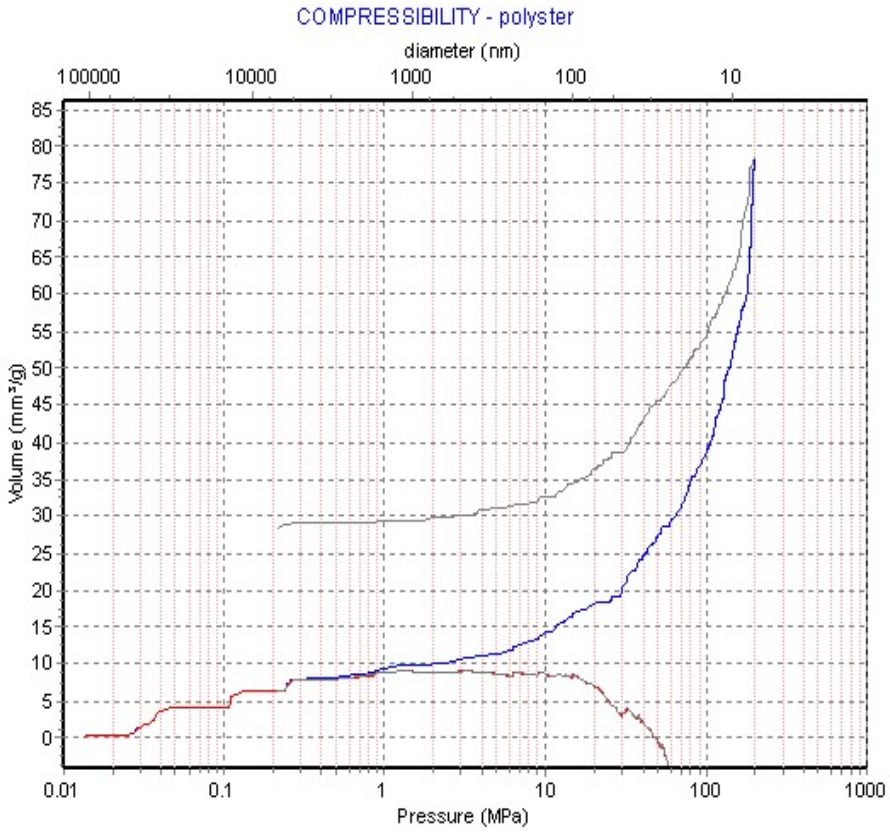
Blank date:	30-12-99
Blank filename:	C:\Thermo Scientific\Data\blank809\blank809_200MP_I7D7_110222.P24
Comment	
Blank Max pressure (MPa):	200
Blank Increase speed:	7
Blank Increase method:	Pascal
Blank Decrease speed:	7
Blank Decrease method	Pascal
Dil. number:	809
Dil. type:	CD3
Dil. Cone length (mm):	22
Dil. Electrode gap (mm):	5
Dil. stem radius (mm):	1.5
Dil. weight (g):	44
Temperature of blank (°C):	25
Mercury density @ blank (g/cm³):	13.534
Blank filling volume (mm³):	489

SAMPLE RESULTS



RESULTS WITHOUT COMPRESSIBILITY CORRECTION

Total intruded volume (mm³/g):	78.34 at pressure of (MPa):	200.6115	
Intrud. Vol. (mm³):	3.78		
Envelope density (g/cm³):	1.8625		
Bulk density @ pressure (g/cm³):	1.8854	at pressure of (MPa):	0.174
Apparent density (g/cm³):	2.1806	at pressure of (MPa):	200.6115
Void volume by real density (mm³/g):	-31.26		
Accessible porosity (%):	14.59		
Inaccessible porosity (%):	-20.41		



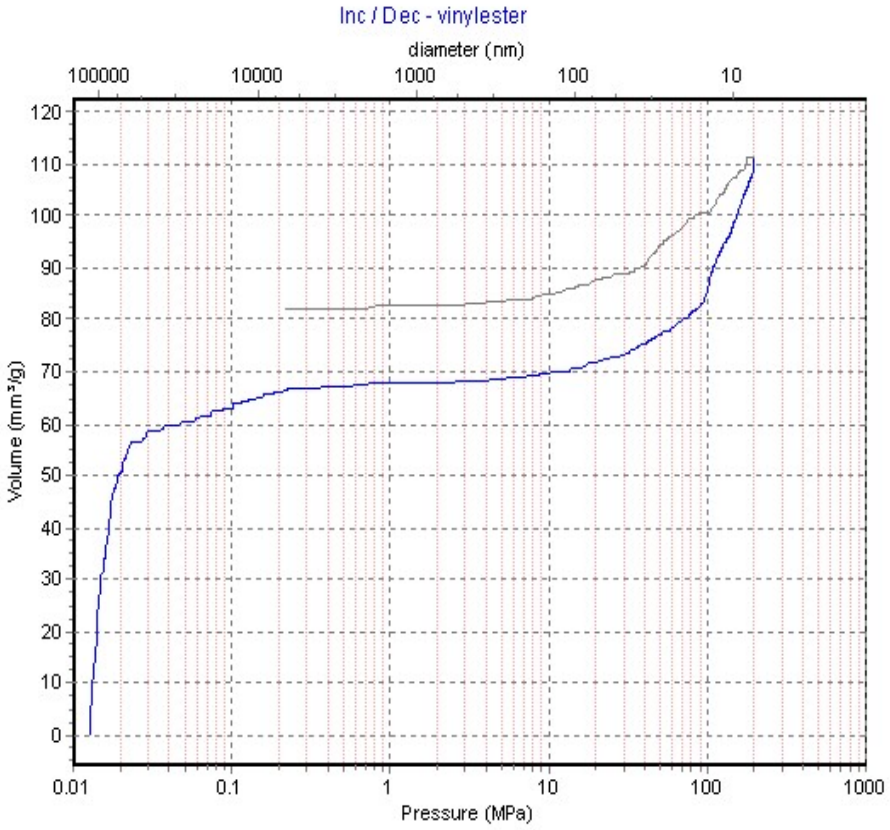
RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N):	Yes			
Linearity range:	From (MPa):	160.4892	To (MPa):	200.6115
Correction factor (mm³/g.MPa)	0.553885			
Compressibility factor (MPa.g/mm³)	1.8054			
Compressed volume (mm³/g):	111.12			
Sample compressibility (1/MPa):	7.070E-3			
Bulk modulus (MPa):	1.414E+2			
Total intruded volume (mm³/g):	-32.77	at pressure (MPa):		200.6115
Intruded vol. (mm³):	3.73			
Envelope density (g/cm³):	1.8625			
Bulk density @ pressure (g/cm³):	1.8850	at pressure (MPa):		0.1735
Apparent density (g/cm³):	1.7553	at pressure (MPa):		200.6115
Real void volume by real dens. (mm³/g):	-31.26			
Accessible porosity (%):	-6.10			
Inaccessible porosity (%):	0.28			

Vinyl Ester

Source file:	C:\Thermo Scientific\Data\sample4(R).142
SOLID REPORTING	
Company name:	utp
Operator:	fifi
TEST INFORMATION	
Test date:	05-03-12
Sample name:	vinylester
Comment:	meraga
Sample mass (g):	0.81
Sample real density (g/cm³):	1.72
Test file name:	C:\Thermo Scientific\Data\sample4_200mp_120305.P24
Combined with file (140):	C:\Thermo Scientific\Data\sample4_400kp_120305.P14
Mercury surface tension (N/m):	0.48
Mercury contact angle (°):	140.0
Test filling volume (mm³):	471.0
Starting hydr. press. of test (MPa):	0.0125
(Dil+Hg+Sample) weight (g):	241.82
Corrected weight (Dil.+Hg+Sample) (g):	242.064
ANALITICAL CONDITIONS	
Maximum test pressure (MPa):	200
Increase speed:	7
Increase method:	Pascal
Decrease speed:	7
Decrease method:	Pascal
Temperature of test (°C):	25.0
Mercury density @ test (g/cm³):	13.534
BLANK & DILATOMETER INFORMATION	
Blank date:	30-12-99
Blank filename:	C:\Thermo Scientific\Data\blank809\blank809_200MP_I7D7_110222.P24
Comment	
Blank Max pressure (MPa):	200
Blank Increase speed:	7
Blank Increase method:	Pascal
Blank Decrease speed:	7
Blank Decrease method	Pascal
Dil. number:	809
Dil. type:	CD3
Dil. Cone length (mm):	22
Dil. Electrode gap (mm):	5
Dil. stem radius (mm):	1.5
Dil. weight (g):	44
Temperature of blank (°C):	25
Mercury density @ blank (g/cm³):	13.534
Blank filling volume (mm³):	489

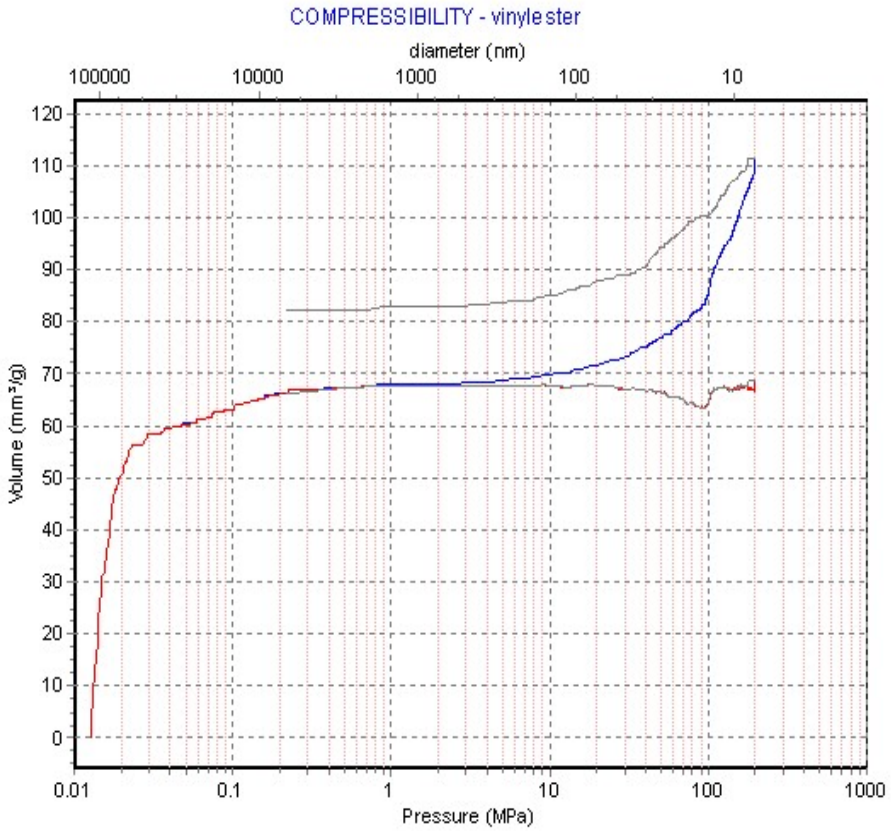
SAMPLE RESULTS



RESULTS WITHOUT COMPRESSIBILITY CORRECTION

Total intruded volume (mm³/g):	111.33	at pressure of (MPa):	200.5115
Intrud. Vol. (mm³):	53.33		
Envelope density (g/cm³):	2.0091		
Bulk density @ pressure (g/cm³):	2.3154	at pressure of (MPa):	0.174
Apparent density (g/cm³):	2.5880	at pressure of (MPa):	200.5115
Void volume by real density (mm³/g):	-83.66		
Accessible porosity (%):	22.37		
Inaccessible porosity (%):	-39.18		

COMPRESSIBILITY CORRECTION



RESULTS WITH COMPRESSIBILITY CORRECTION

Correction enabled (Y/N):	Yes		
Linearity range:	From (MPa): 160.4092	To (MPa):	200.5115
Correction factor (mm³/g.MPa)	0.212982		
Compressibility factor (MPa.g/mm³)	4.6952		
Compressed volume (mm³/g):	42.71		
Sample compressibility (1/MPa):	1.913E-3		
Bulk modulus (MPa):	5.227E+2		
Total intruded volume (mm³/g):	68.62 at pressure (MPa):		200.5115
Intruded vol. (mm³):	53.30		
Envelope density (g/cm³):	2.0091		
Bulk density @ pressure (g/cm³):	2.3152	at pressure (MPa):	0.1735
Apparent density (g/cm³):	2.3304	at pressure (MPa):	200.5115
Real void volume by real dens. (mm³/g):	-83.66		
Accessible porosity (%):	13.79		
Inaccessible porosity (%):	-30.60		