STUDY ON CHARACTERISATION AND PROPERTY OF LOCAL SAND

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MECHANICAL ENGINEERING UNVERSITI TEKNOLOGI PETRONAS JANUARY 2008

CERTIFICATION OF APPROVAL

STUDY ON CHARACTERISATION AND PROPERTY OF LOCAL SAND

by

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A project dissertation submitted to the Mechanical Engineering Programme Universiti Teknologi PETRONAS In partial fulfillment of the requirements for the BACHELOR OF ENGINEERING (Hons) (MECHANICAL ENGINEERING)

Approved by,

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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 haven not been undertaken or done by unspecified sources or persons

(TEH CHOON CHI)

ABSTRACT

Glass is an amorphous solid. They are low tensile strength and brittle so that fracture occurs suddenly due to structure, manufacturing process, and properties, without prior plastic deformation. They often fail at a sudden temperature change. The objective is to study on local sand property when exposed to high temperature as well as looking into possibilities of developing a high temperature polymer based on local sand. The microstructure and characteristics of the sample is analyzed by using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). As a conclusion, the sand samples heated at high temperature can be enhanced after running with mechanical ball milling process. This shows that the mechanical milling treatment on the silica sand not only causes size reduction but also structural changes from crystalline phase to distorted structure which are activated due to the high surface energy. From the result, the sand samples were found having better properties after mechanical milling where the samples were found to be more amorphous.

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CHAPTER 1

INTRODUCTION

1.1 Background

Glass, ceramics and glass-ceramics represent a very broad class of materials differing in composition, structure, manufacturing process, and properties. However, they have many features in common. They usually have good chemical resistance, low thermal conductivity, high hardness and compressive strength, and often also good resistance to creep at high temperatures. [1] In this case, their tensile strength is usually lower, with scatter of individual values, and decreasing with time of loading in some cases. They are brittle so that fracture occurs suddenly, without prior plastic deformation. They often fail at a sudden temperature change.

Glass is an amorphous solid. A material is amorphous when it has no long-range order, that is, when there is no regularity in the arrangement of its molecular constituents on a scale larger than a few times the size of these groups.[2]

Silica glass contains a single component, SiO₂. It might therefore be assumed that its properties would vary within very narrow limits, regardless of the origin of the sample. However, the choice and purity of the initial raw materials and the manufacturing method are responsible for considerable differences between the samples. These differences are generally associated with defects in the structure of silica glass.[1]

1.2 Problem Statement

As we know, there are abundant of sand all around the area of Universiti Teknologi PETRONAS and TRONOH area. The sand is not being fully utilized for commercial purpose.

Most polymer made by sand are low tensile strength, brittle and easily fracture due to the high crystalline structure and properties. However, the most important factor of low tensile strength, brittleness and easy fracture of polymer is due to the sudden temperature change.

If the structure and properties of local sand can be improved, there will be more potential and commercial applications.



Figure 1.1: Sand Hill



Figure 1.2: Brittle And Easily Fracture Due To The High Crystalline Structure And Properties

1.3 Objective

The objectives of this project are:

- To study on local sand property when exposed to high temperature.
- To study possibilities of developing a high temperature polymer based on local sand.

1.4 Scope of Study

The scope of study of this project is to study on the local sand properties, structure and viscosity when exposed to high temperature and study possibilities of developing a high temperature polymer based on local sand. Some mechanical properties have to be determinate to ensure the toughness and strength of the high temperature polymer.

This project involves study, sieving, heating, testing and analysis. In this case, this project is executed in two phases, where phase one (FYP 1) is mainly concentrates on the preliminary research, studies on the local sand, sieving and experiment on the local sand by heating in different temperatures. While for the second phase (FYP 2) will continue with the polymer structure studies after heating at particular temperature, testing on the strength and will be more focus on the analysis and the final documentation.

For the project planning and progress of this project, in phase 1 (FYP 1) and phase 2 (FYP2) are cover in the chapter three under methodology section.

CHAPTER 2

LITERATURE REVIEW AND THEORY

Sand is a naturally occurring granular material composed of finely divided rock and mineral particles. The most common constituent of sand, in inland continental settings and non-tropical coastal settings, is silica (silicon dioxide, or SiO₂), usually in the form of quartz, which, because of its chemical inertness and considerable hardness, is resistant to weathering. The composition of sand is highly variable, depending on the local rock sources and conditions.[3]

Silica sand is usually colorless or white but is frequently colored by impurities, such as iron, and may then be any color. Sand may be transparent to translucent, hence its use in glassmaking, and have a vitreous lustre. Sand is a hard mineral owing to the strength of the bonds between the atoms and it will scratch glass. It is also relatively inert and does not react with dilute acid. These are prized qualities in various industrial uses. [4]

Silica sand has considerably low thermal expansion and fairly high melting point. It is because of the strong covalent bonding. During the melting and boiling, covalent bonds between atoms are broken. Much more energy required to break covalent bonds than to separate the molecules from each other. [5] In addition, silica sand is resistant to creep making it a good refractory material. It tends to be used in acid environments if used on its own or used as a starting material for the synthesis of other refractory products. Due to the fact that silica is insoluble in the majority of acids, it is used as a refractory material in acidic environments. Silica is classified as an acid refractory as it behaves like an acid at high temperatures reacting with bases. The majority of its applications are in the glass industry. [6]

The commonest use of quartz and glass-sand, also referred to as silica-sand, is in the manufacture of glass. Great advancement has been made in the manufacture of translucent, transparent, colored and clear glass in sheets or in glassware. The size of the sand grains is important in glass industry. It should be between 40 to 80 mesh (BSS). It should be of high purity containing a minimum of 98% SiO₂. In the manufacture of colorless glass the iron content (Fe₂O₃) should not exceed 0.04%. for optical purposes, the presence of iron oxide (Fe₂O₃) more than 0.008% is not tolerated.

Iron and chromium are both objectionable impurities in glass-sand. The minutest presence of these impurities gives color effect in the glass melt. Glass is manufactured by melting a mixture in suitable proportion of feldspar, dolomite, limestone and soda ash together with glass-sand at 1400° - 1500°C in the furnace when clear molten glass is formed. [7]

Sand glass is a combination of various chemical materials. It is because various chemical materials possess the capability for forming a vitreous structure. The most important among the inorganic materials are the oxides of silicon (Si), boron (B), germanium (Ge), phosphorus (P), and arsenic (As). When cooled quickly after melting, they solidify without crystallization, forming glass. [8] Sand samples were subjected to water and chemical washing (hot and cold) and grading to remove the ferrogeneous claying fraction and undesirable coarse and fine particles.

Semicrystalline polymers constitute the largest group of commercially useful polymers. These polymers exists as viscous liquids at temperatures above the melting point of the crystals. Upon cooling, crystals nucleate and grow to fill the available volume. The reason these materials are called "semicrystalline" is that some fraction of the polymer remains un-crystallized, or, amorphous when the polymer is cooled to room temperature. The amorphous polymer becomes trapped between the growing crystals.

As a result of the highly entangled nature of the polymer chains, the movement of the amorphous polymer becomes restricted. [9]

Solids may be either amorphous or crystalline in their molecular structure. In amorphous solids, the molecules are arranged haphazardly. Glass is an example of an amorphous material. Like other amorphous materials, glass does not melt at a particular temperature, because the long, randomly intertwined glass molecules cannot easily become disentangled. As a result, glass softens bit by bit as the temperature is raised, eventually becoming liquid. Crystalline materials, on the other hand, have a definite orderly array of atoms, ions, or molecules.[10]

For the polymer has amorphous structure on an atomic level. It will has a very good mechanical properties because amorphous structure does not has a slip plane. [11] It is mean that polymer with amorphous structure will have higher tensile strength and more ductile compare to polymer with crystalline structure.



Figure 2.1: Deformation Characteristics Of Crystallite Structure And Amorphous Structure

2.1 Scanning Electron Microscope (SEM) [12] [14]

The scanning electron microscope (SEM) is a type of electron microscope that creates various images of a specimen that placed into a high vacuum chamber by focusing a high energy beam of electrons is forced onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample's surface. It permits the observation and characterization of heterogeneous organic and inorganic materials on a nanometer (nm) to micrometer (μ m) scale.

The type of signals gathered in a SEM varies and can include secondary electrons, characteristic x-rays, and back scattered electrons. In a SEM, these signals come not only from the primary beam impinging upon the sample, but from other interactions within the sample near the surface.

The SEM is capable of producing high-resolution images of a sample surface in its primary use mode, secondary electron imaging. Due to the manner in which this image is created, SEM images have great depth of field yielding a characteristic threedimensional (3-D) appearance useful for understanding the surface structure of a sample. This great depth of field and the wide range of magnifications from 10 times up to 10000 times are the most familiar imaging mode for specimens in the SEM.

Characteristic x-rays are emitted when the primary beam causes the ejection of inner shell electrons from the sample and are used to tell the elemental composition of the sample. The back-scattered electrons emitted from the sample may be used alone to form an image or in conjunction with the characteristic x-rays as atomic number contrast clues to the elemental composition of the sample.



Figure 2.2: Scanning Electron Microscope (SEM)

2.2 X-Ray Diffraction (XRD) [13][14][15]

X-ray Powder Diffraction (XRD) is an efficient analytical technique and nondestructive for analyzing, identify and characterize various crystalline phases of materials, including fluids, metals, minerals, polymers, catalysts, plastics, ceramics, pharmaceuticals, thin-film coatings and semiconductors. Throughout industry and research institutions, XRD has become an indispensable method for materials investigation, characterization and quality control. Example areas of application include qualitative and quantitative phase analysis, crystallography, structure and relaxation determination, texture and residual stress investigations, controlled sample environment, micro-diffraction, nano-materials, lab- and process automation, and high-throughput polymorph screening.

XRD is a basic tool in the mineralogy evaluation of rock especially fine-grains sediments and clay-rich rocks. Monochromatic x-rays are used to determine the interplanar spacings of the unknown materials. Samples are analyzed as powders with grains in random orientations to insure that all crystallographic directions are "sampled" by the beam. When the Bragg conditions for constructive interference are obtained, a "reflection" is produced, and the relative peak height is generally proportional to the number of grains in a preferred orientation.

The X-ray diffraction pattern of a pure substance is like a fingerprint of the substance. Every crystalline substance gives a pattern, the same substance always gives the same pattern, and in a mixture of substances each produces its pattern independently of the others.

The x-ray spectra generated by this technique, thus, provide a structural fingerprint of the unknown. Mixtures of crystalline materials can also be analyzed and relative peak heights of multiple materials may be used to obtain semi-quantitative estimates of abundances. A glancing x-ray beam may also be used to obtain structural information of thin films on surfaces. In addition, changes in peak position that represent either compositional variation (solid solution) or structure-state information (e.g. order-disorder transitions, exsolution, etc.) are readily detectable. Peak positions are reproducible to 0.02 degrees.

Data reduction routines rapidly determine peak position, relative intensities, and calculate intracrystalline d-spacings. The complete American Society of Testing Materials (ASTM) powder diffraction file is available on-line for identification of unknown crystalline materials with an internationally recognized database containing for more than 70000 phases. The advantages of the X-Ray powder diffraction (XRD) are rapid identification of materials, ease of sample preparation, computer-aided material identification, large library of known crystalline structures and multi-sample stage.



Figure 2.3: X-Ray Diffraction (XRD)

CHAPTER 3

METHODOLOGY

3.1 Procedure Identification

The procedures of identification that involved in this project can be split down into several main sections. They are literature review, experiment, result analysis and conclusion for the overall project methodology flow.

However, this project is mainly on the studies about property and characterisation of local sand, so this project required a lot of experiments. First of all, the writer needed to collect samples (local sand) from Universiti Teknologi PETRONAS area and TRONOH area. As the particle size of the raw local sands vary from 63µm to 2mm, so mechanical sieve shaker is required to ensure the finest and equally size of the sand will be used throughout the whole process of the project and this process has been carried out according to the British Standard (BS1377). In addition, dry sieving method was chosen for this process, because the soils contain insignificant quantity of silt and clay. [16] In this case, the raw local sand had been sent to drying oven for heating at 100°C for at least 24 hours to ensure the sands are free from moisture before the mechanical sieving process was carried out.

After collecting the finest sand samples from the process of mechanical sieving, the sand samples have been sent for heating in the furnace with 200°C, 400°C, 600°C, 800°C, 1000°C and 1200°C respectively. In this case, the changes of the structure and characteristic of the sand samples can be taken according to the specific elevated heating temperature by using scanning electron microscope (SEM) and X-Ray Diffraction (XRD). During the heating process, we have to ensure the sand samples are heating at the specific temperature for at least 30 minutes before the particular sand sample is being collected from the furnace. This step is to ensure every single particle of the sand samples are fully heat with the specific temperature.

After the heating process, approximately 20% of the sand samples have been sent to the scanning electron microscope (SEM) laboratory, 30% sent to the X-Ray Diffraction (XRD) laboratory and 50% sent to the mechanical material laboratory for running ball milling process before going through the SEM and XRD mineralogy evaluation. For the SEM process, the high-resolution topographic images of a sample surface is yielding a characteristic three-dimensional (3-D) appearance useful for understanding the surface structure of a sample. In this case, the sand samples microstructure surface can be analyzed easily.

Eventually, XRD process have been carried out for non-destructive analyzing, identifying and characterizing various crystalline phases of the specific sand samples. The procedures of the main sections are stated at the overall project methodology flow chart as below in Figure 3.1.



Figure 3.1: Overall Project Methodology Flow Chart

The experimental process in this project is showed in the experimental methodology flow chart as below in the Figure 3.2.



Figure 3.2: Experimental Methodology Flow Chart

3.2 **Project Planning (Key Milestone)**

Phase 1 or final year project1 (FYP 1) mainly concentrates on preliminary research, studies on the local sand, sieving and experiment on the local sand by heating in different temperatures. While phase 2 (FYP 2) of this project will continue with the polymer structure studies after heating at particular temperature, testing on the strength and will be more focus on the analysis and the final documentation.

| TASK | DESCRIPTION | PREDECCESSOR | NORMAL TIME |
|------|---------------------------------------|--------------|-------------|
| | | (WEEK) | (WEEKS) |
| 1 | Selection of project title | 1 | 1 |
| 2 | Preliminary research work | 4 | 3 |
| 3 | Mechanical Sieving | 5 | 2 |
| 4 | Heating process | 7 | 2 |
| 5 | Scanning Electron Microscope (SEM) | 8 | 1 |
| 6 | Ball Milling | 9 | 1 |
| 7 | X-Ray Diffraction (XRD) | 11 | 2 |
| 8 | Progress report 1 | 13 | 2 |
| 9 | Analyze | 17 | 4 |
| 10 | Progress report 2 & Seminar | 19 | 2 |

| 11 | Poster exhibition & Oral | 20 | 1 |
|----|--|----|---|
| | Presentation | | |
| 12 | Dissertation final draft & oral presentation | 23 | 3 |
| 13 | Submission of hardbound thesis | 24 | 1 |

Table 3.1: Project Tasks And Duration List For (FYP 1 & 2)

Based on table 3.1 as above, a project Gantt chart was developed to facilitate the project execution, which is attached in APPENDIX A .

3.3 Tools Required

3.3.1 Hardware Or Outsourced Tools And Equipments:

- Shovel
- Electronics balances
- Tray

3.3.2 Software:

- Microsoft offices XP (word, excel, power point, project)

3.3.3 Mechanical Engineering Workshop Facilities:

- Furnace
- Scanning Electron Microscope (SEM)
- X-Ray Diffraction (XRD)
- Ball Milling

3.3.4 Civil Engineering Workshop Facilities:

- Drying Oven
- Mechanical Sieve Shaker

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Findings And Discussion

4.1.1 Physical change of the sand after heating

The sand bodies were formed by pressed and fired at different temperatures (between 1100°C and 1300°C). From the observation of the sand samples physical, we can see that the colour of the sand samples after heat with the higher temperature were changing from darker colour to lighter colour. The colour of the sand samples after heating with 200°C and 1200°C are shown as below:



Figure 4.1: Sand After Heat With 200°C



Figure 4.2: Sand After Heat With 1200°C

4.1.2 Microstructure Of Sand After Heating With Different Temperature

From the observation by using Scanning Electron Microscope (SEM) to scan the sand samples by magnifying the size of the samples into 3000 times with Energy High Tension (EHT) 15kV, we can see that the microstructure of the sand samples are merging while heating with higher temperature.

In addition, we can observe that while the heating temperature on the sand samples were getting higher, the pores on the sand samples surface is getting bigger and more significant as well. From this point of view, we can conclude that while the heating temperature is getting too high, the better and stronger microstructure of the sand samples are not formed and the properties of the sand samples are not being enhanced as well, but will start to crack.

As we can see from the microstructure image from the sand samples heated at high temperature at 1200°C can be enhanced after running with mechanical ball milling process. This shows that the mechanical ball milling treatment on the silica sand not only causes size reduction but also structural changes from crystalline phase to distorted structure which are activated due to the high surface energy.

The sand samples had been heated with 200°C, 200°C run with mechanical ball milling process, 400°C, 600°C, 800°C, 1000°C, 1200°C and 1200°C run with mechanical ball milling process respectively. The microstructure of the sand samples are shown as below:



Figure 4.3: Microstructure Of Sand After Heating With 200°C



Figure 4.4: Microstructure Of Sand After Heating At 200°C And Processed With Mechanical Ball Milling



Figure 4.5: Microstructure Of Sand After Heating At 400°C



Figure 4.6: Microstructure Of Sand After Heating At 600°C



Figure 4.7: Microstructure Of Sand After Heating At 800°C



Figure 4.8: Microstructure Of Sand After Heating At 1000°C



Figure 4.9: Microstructure Of Sand After Heating At 1200°C



Figure 4.10: Microstructure Of Sand After Heating At 1200°C And Processed With Mechanical Ball Milling

4.1.3 Characteristics of crystalline substances for sand after heating with different temperature

From the result by using X-Ray Diffraction (XRD) in room temperature,25°C with peak positions are reproducible to 0.02 degrees, we discover the characteristics, atomic and molecular structure of crystalline substances.

Throughout the analysis of XRD results by referring to the range from 20 to 30 and 40 to 60 2-Theta-Scale in the XRD graphs for the density of crystallization according to the ASTM, we can observe that the characteristics and molecular structure of the sand samples is getting better by reducing the crystalline density of the sand samples while the heating temperatures were getting higher respectively. In these cases, sand samples were getting more amorphous.

Furthermore, we can observe that the density of montmorillonite is reducing while the heating temperature is increasing. In this case, montmorillonite is shown in the range from 30 to 40 and 60 to 70 2-Theta-Scale according to the ASTM for the standard sand.

In addition from the XRD results, we can analyze that the crystallisation of the sand properties can be improved by running the sand samples with mechanical ball milling. It means that the sand samples will have better property after going through the process of mechanical ball milling. The property of the sand samples will be more amorphous and less crystalline.

The sand samples in room temperature and had been heated with 200°C, 400°C, 600°C, 800°C, 1000°C and 1200°C, respectively. The characteristics of crystalline substances of the samples are shown as below:



Figure 4.11: XRD Result For Sand Without Heating.



Figure 4.12: XRD Result For Sand After Heating At 200°C



Figure 4.13: XRD Result For Sand After Heating At 200°C And Processed With Mechanical Ball Milling



Figure 4.14: XRD Result For Sand After Heating At 400°C



Figure 4.15: XRD Result For Sand After Heating At 600°C



Figure 4.16: XRD Result For Sand After Heating At 800°C







Figure 4.18: XRD Result For Sand After Heating At 1200°C



Figure 4.19: XRD Result For Sand After Heating At 1200°C And Processed With Mechanical Ball Milling

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Since this project has achieved all of the objectives for the project plan for the final year project 1 (FYP 1), it can be concluded that the status of the project is realized as scheduled. However, at certain stages there is some delay and caused behind of the planned schedule. The delay is mostly caused by the lab equipment breakdown and unavailable of X-Ray Diffraction (XRD) machine and Scanning Electron Microscope (SEM).

In the first half semester, the literature review and researches had been done for getting a better picture and understanding of developing high temperature polymer and indentify the main problems. The purpose of this study is to develop an understanding on structure changes due to heating of sand and to discover the possible ways of developing a high temperature polymer based on local sand.

From the analysis of the results from X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM), we can conclude that the structure and property of the local sand can be improved by heating with high temperature. Although the high temperature can improve the structure and property of the local sand by reducing the crystallization of the local sand samples and increasing the amorphous property, but with the extreme high temperature above 800 $^{\circ}$ C , the structure of the sand samples start to crack by getting bigger and significant pores on the structure surface.

In this case, the problem of cracking can be resolved by running the sand samples with mechanical ball milling. In addition, the property of the sand samples can be enhanced as well by increasing the level of amorphousness and reducing the crystalline of the local sand samples.

5.2 **Recommendation For Future Work**

The completion of this project is viewed as a completion of a stage only out of many stages. Future expansion on the project is still possible and viable.

It is recommended that a further study to be carried out to develop a high temperature polymer properties, so that a better quality glass in terms of mechanical properties can be produced. In this case, adding chemical elements into the sand samples before heating is recommended to enhance the sand mechanical properties.

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NO. **DETAIL/WEEK** 2 4 5 10 11 12 13 14 15 16 17 1 3 7 8 9 6 **Selection of Project Topic** 1 -Propose Topic -Topic Assign to Student 2 **Preliminary Research** -Introduction -Objective -List of references/literature -Project Planning 3 **Mechanical Sieving Heating Process** 4 **Scanning Electron** 5 Microscope (SEM) **Ball Milling** 6 X-Ray Diffraction (XRD) 7 **Progress Report 1** 8 9 Analyze

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| 12 | Dissertation Final Draft & | | | | | | | |
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APPENDIX B

DETERMINATION OF PARTICLE SIZE DISTRIBUTION (MECHANICAL SIEVING LABORATORY MANUAL)

UNIVERSITI UNIVERSITI TEKNOLOGI PETRONAS

TEST 3 : DETERMINATION OF PARTICLE SIZE DISTRIBUTION

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Objectives: the abuse officient requires leave the same transmission and the same transmission of the second state of the

To determine the size distribution of soil using the dry sieving and hydrometer method.

Apparatus:

- a) Test sizes having the following aperture sizes may be used: 75mm, 63mm, 50mm, 37.5mm, 28mm, 20mm, 14mm, 10mm, 6.3mm, 5mm, 3.35mm, 2mm, 1.18 mm, 600µm, 425µm, 300µm, 212µm, 150µm, 63µm, lid and receiver.
- b) Electronic balances.
- c) Riffle box
- d) Drying oven.
- e) Tray.
- f) Scoop.
- g) Sieve brushes.
- h) Mechanical sieve shaker.

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General Discussion

Two methods of sieving are specified. Wet sieving is the definitive method applicable to essentially cohesionless soils. Dry sieving is suitable only for soils containing insignificant quantities of silt and clay. Two methods of determining the size distribution of the fine particles namely the pipette method and the hydrometer method, in both of which the density of the soil suspension at various intervals is measured. Combined sieving and sedimentation procedures enables a continuous particle size distribution curve of a soil to be plotted from the size of the coarsest particle down to the clay size. This method covers the quantitative determination of particle size distribution in a cohesionless soil down to the fine-sand size.

Procedure:

- 1. Weigh the oven dried sample to 0.1% to its total mass (m_1)
- 2. Stack 8 numbers of test sieves on the mechanical shaker with the largest size test sieve appropriate to the maximum size of material present at the bottom of the stack followed by the smaller size test sieves and a receiver at the bottom of the stack.
- Place the sample on the top sieve and cover the sieve with a lid. Agitate the test sieves on the mechanical sieve shaker for 5 minute. Weigh the amount retained on each of the test sieves to 0.1% of its total mass.

Calculations and Final Results:

- 1. Calculate the percentage by mass of material retained on each test sieve.
- 2. Calculate the cumulative percentage (by mass of total sample) passing each of the sieves.
- Report the results as a table showing, to the nearest 1%, the percentage by mass passing each of the sieves used.
- 4. Express the results obtained on a semi-logarithmic chart

Discussions:

Conclusions:

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