FINAL YEAR PROJECT

FINAL REPORT

Mineral Derived from Fine Clay Isolated from Potter Wasp Nest Collected from Universiti Teknologi PETRONAS (UTP) Main Hall

by

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CERTIFICATION OF APPROVAL

MINERAL DERIVED FROM FINE CLAY ISOLATED FROM POTTER WASP NEST COLLECTED FROM UNIVERSITI TEKNOLOGI PETRONAS (UTP) MAIN HALL

By

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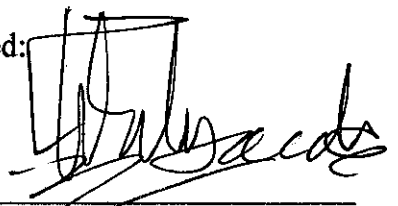
Approved:



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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 specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMAD ASRI BIN ABU SAMAH

Abstract

This project is mainly about researching and experiment on a clay use by an insect to build their nest. There are several types of clays; assumption has been made that this insect called potter wasp has an instinct to choose fine clay to build their nest. Thus the idea of this research is to prove that either the clay use by the potter wasp is the fine clay from the hypothesis. This project will be focusing on experiment and analyzing on the clay isolated. There are also several step involve before the experiment will start such as identification, sampling, washing and lastly analyzing. This report will represent in details on the introduction about the project which is to briefly about the study, the problem statement, the objectives and methodology used in conducting the experiment.

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CHAPTER 1
INTRODUCTION

1.1 Background of Study

Technology of ceramics is rapidly rising in today's science world. As a matter of fact, there is devoted competition among the industrial nations to exploit this knowledge to the fullest. Ceramic is define as any various hard, brittle, heat and corrosion-resistant materials made by firing clay or other minerals and consisting of one or more metals in combination with one or more nonmetals. Ceramics are primarily crystalline in nature. Ceramic materials can be subdivided into traditional and advanced ceramics. Traditional ceramics include clay-base materials such as dinnerware, brick, tile, sanitary ware, electrical porcelain and clay pipe.

Ceramic is an art whose beginnings are shrouded in the past. This is not a unique situation because it is easy to say the same thing about many of our new and revolutionary technologies. The industrial revolution was made possible by advance furnaces and heat engines, and ceramic materials were essential for thermal insulation of the various types of furnaces and engine. Figure 1 shows one of the applications of the ceramic.

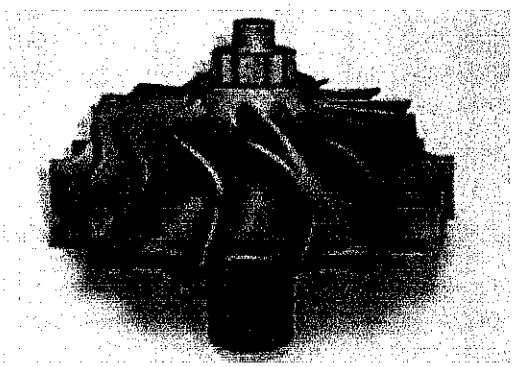


Figure 1: Ceramics turbine

Clay minerals make up the vast majority of the earth's crust. It remains the most widely used raw material for producing traditional ceramic products. Silicate and aluminum silicate mineral are widely available to form the back bone of the traditional high-volume products of the ceramic industry. However, they are expensive. Therefore, the porcelain made from the kaolinite as the essential ingredient along with feldspar and quartz which is the far finest is mostly highly prized form of ceramic.

The raw materials for most of the ceramic came from mines and quarries, and these raw ingredients were prepared for the thermal processes needed to convert them to useful articles by crushing, washing, sieving and mixing appropriate formulations. Usually, these naturally found materials were not pure, and the formulas had to take into account the small fractions of naturally occurring and often variables impurities and minor fractions.

Basically, there is an insect call as potter wasp, building its nest by using the clay. The potter wasp is named based on the shape of the nest. The nest is build based on the mixture of water and clay. There are around 3000 species worldwide. Potter wasps are medium wasps which is about 9 to 20 mm (0.35 to 0.79 in) long. They are black with white, yellow, orange, or red markings. Potter wasps comprise the subfamily Eumeninae in the family Vespidae.

1.2 Problem Statement

1.2.1 Problem Identification

Clay is the element in deciding the quality of the tri-axial ceramic. The finest clay used in forming the ceramic will exaggerate the price and certain properties of the ceramic. In relation, the unique insect nest, potter wasp, build their nest by using clay. Therefore, a hypothesis is made that the potter wasp nest is build by using very fine clay which is very costly to obtain. This study is to identify do the potter wasp build their nest by using the fine clay and also to classify the type of the clay extracted from the nest.

1.2.2 Significant Of the Project

Through this project, microstructure of the clay will be observed. Apart, composition of the clay will also be examined. With this, it will be possible to discover the type of the clay extracted from the nest.

1.3 Objective

The main purpose of this project is to analyze the clay extracted from the potter wasp nest. In order to complete the main objective, the clay extracted will be analyzed on the microstructure and the composition of the clay. The result will be compared with the kaolinite.

1.4 Scope of Study

The scope of study is mainly on analyzing the clay derived from the potter wasp to gather information. The study will also give a basic knowledge and understanding the properties of the tri-axial ceramic form by the finest clay.

1.5 The Relevancy of the Project

This project will require lot of test and knowledge. As a mechanical engineering, this project will give an extra understanding regarding the material study during the process to form, phase and also the properties of the ceramic. This study highlight the testing method to identified the composition of the material, thus adding an extra knowledge regarding the testing and the equipment used.

CHAPTER 2
THEORY AND LITERATURE REVIEW

2.1 Potter wasp

Wasps are different from bees. Compared to bees, wasps create a nest out of wood or mud where bees make their nests from honey. They are normally found in their nest. They are very sensitive to light. Mostly, they eat caterpillars.

Mud-dauber wasp, mason wasp or known as potter wasp is classified in *Eumeninae* subfamily under *Vespidae* family [1] [2]. The potter wasp is different with the other wasp in building their nest. They are using mud or clay instead of mash woody fiber or pulp [2]. The potter wasp forms a blob of clays by using its mandibles before carrying it to the nest [3] [4]. Research shows that the potter wasp does not appear to mix its saliva with the collected soil. The soils collected are also does not mix with the any organic substance when building their nest [4].

Figure 2 below show the image of the potter wasp nest

Figure 2: Image of Potter wasp nest [5]

Mostly, the nest can be found under the roof space of outhouse, on walls of houses, under piles of wood, or fire-place cowls. Based on research, the location of the nest does not effecting the material of the nest. Researchers have an assumption that the wasp collected the material far from the location of its nest. The location of globules is being collected indicates that the wasps have some constancy in their direction of flight [4]. Figure 3 and figure 4 below shows the image of the male and female potter wasp.

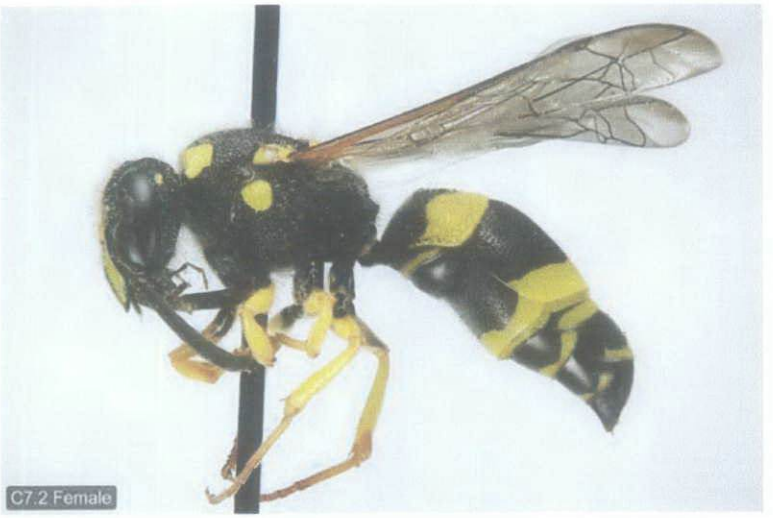


Figure 3: Image of Female potter wasp [5]

Figure 4: Image of male potter wasp [5]

2.2 Kaolinite [$AL_2Si_2O_5$ (OH) $_4$]

Natural clay minerals are well known and familiar to mankind from the earliest days of civilization. Because of their low cost, abundance in most continents of the world, high sorption properties and potential for ion exchange, clay materials are strong candidates as adsorbents[9]. Clay materials possess a layered structure and are considered as host materials. They are classified by the differences in their layered structures. There are several classes of clays such as smectites (montmorillonite, saponite), mica (illite), kaolinite, serpentine, pylophyllite (talc), vermiculite and sepiolite[7] [8].

Clay minerals are among the most important minerals we know or have ever known since they form the basis of pottery and building bricks [6] [8]. The properties of clays are determined by the fact that they are layer materials. They are a subgroup of the layer silicates [8]. In general, the clay minerals are hydrated aluminum silicates based on $(Si_2O_5)n$ sheets. Figure 5 exemplify the DTA curve for the kaolinite and montmorillonite which is one of the clay types.

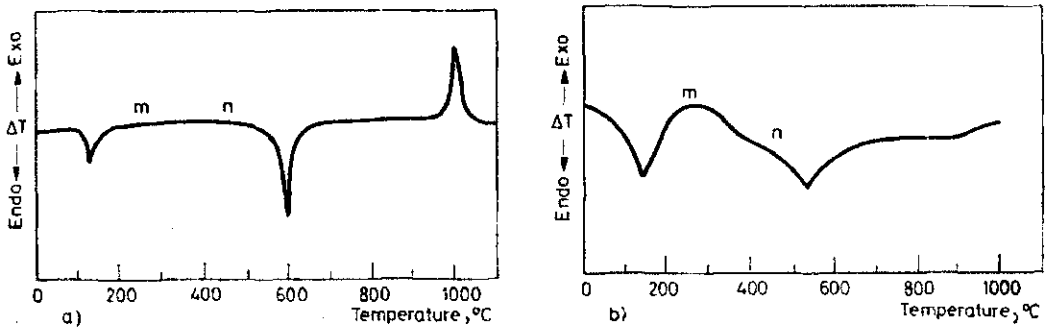


Figure5: DTA for a) kaolinite b)montmorillonite[12]

Kaolinite clay, one of the most versatile industrial minerals, is mostly used as ceramic raw material, coating and filler pigment for paper, filler for paint, rubber, insecticide and also been used in manufacture formulation of medicine [9]. Mined kaolin usually contains silica (quartz) as major contaminant and Fe- and Ti- bearing minerals that impart color as other contaminants [10]. Figure 6 will shows the sheet forming a layer of the kaolinite.

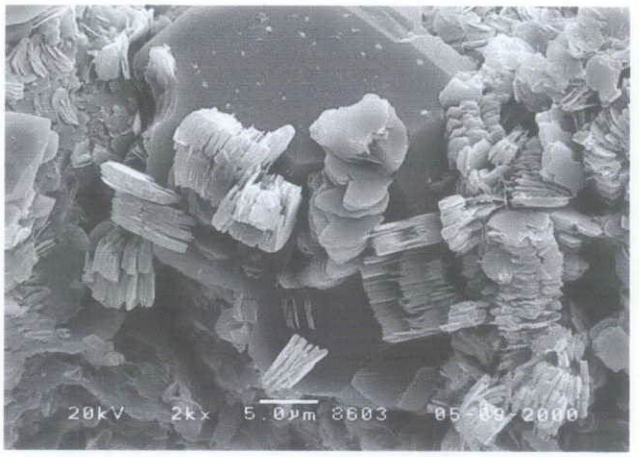


Figure 6: Kaolinite microstructure based on SEM image [11]

Kaolinite is the most common clays with 1:1 layer silicate with structure consist of alternating layer of $(SiO_4)^{4-}$ tetrahedral combined with octahedral coordinated aluminum ions [7]. Kaolinite can be seen in white, brownish white, Grayish white, Yellowish white, grayish green [9].

Figure 7 will illustrate the crystal structure of the kaolinite.

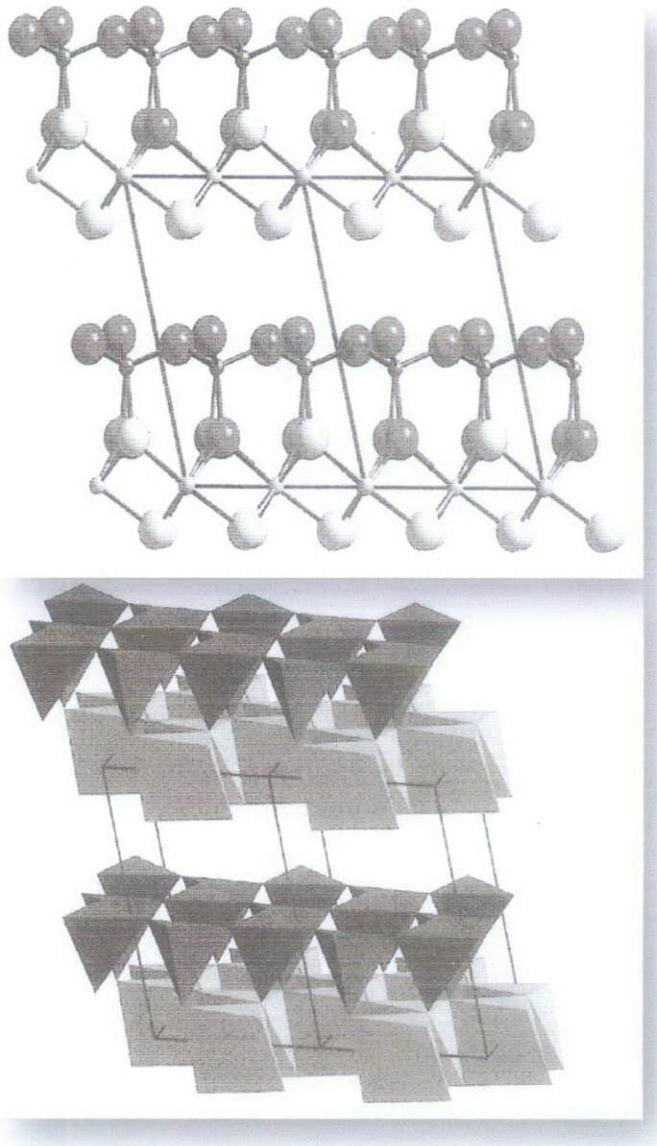


Figure 7: Crystal structure of kaolinite $[AL_2Si_2O_5\ (OH)_4]$ [7]

2.3 Water, H_2O

Water exists in liquid, solid, and gaseous states. At room temperature, water is tasteless, odorless and colorless liquid. Its melting point is at 0°C and boils at 100°C. The density of water is 1000 kg/m^3 [13].

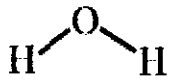


Figure 8: Structural Formula of water, H_2O [13]

2.4 Ethanol CH_3CH_2OH

Ethanol is an alcohol group a group of chemical compounds whose molecules contain a hydroxyl group. It is a colorless liquid. Ethanol has melting point at –114.1°C and boiling point at 78.5°C. The density of ethanol is ***0.789 g/mL*** [13].

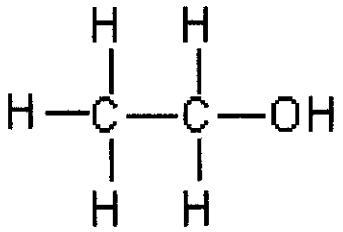
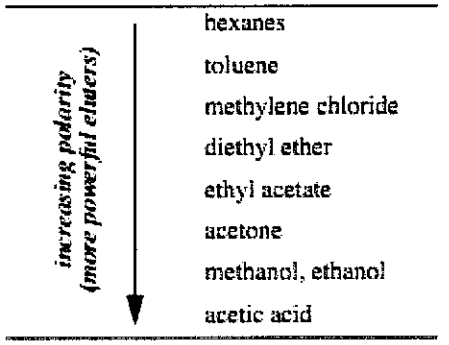


Figure9: Ethanol structural formula CH_3CH_2OH [13]

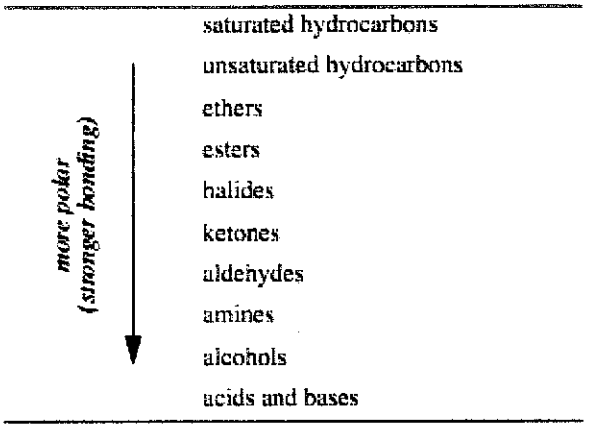
2.5 Acetone $(CH_3)_2C=O$

Acetone is an organic compound. Several properties of acetone are colorless, flammable, and mobile. Acetone is classified in ketones. Density of the acetone is ***0.791 g cm^{-3}***. Acetone has a low boiling point 56.5 °C [13].

Chromatography solvents eluting order.



Usual eluting order of organic compounds.



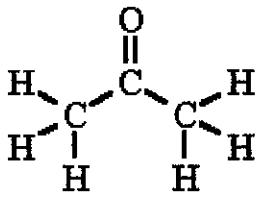


Figure 10: Structural Formula of Acetone [13]

CHAPTER 3

Methodology

3.1 Research Methodology

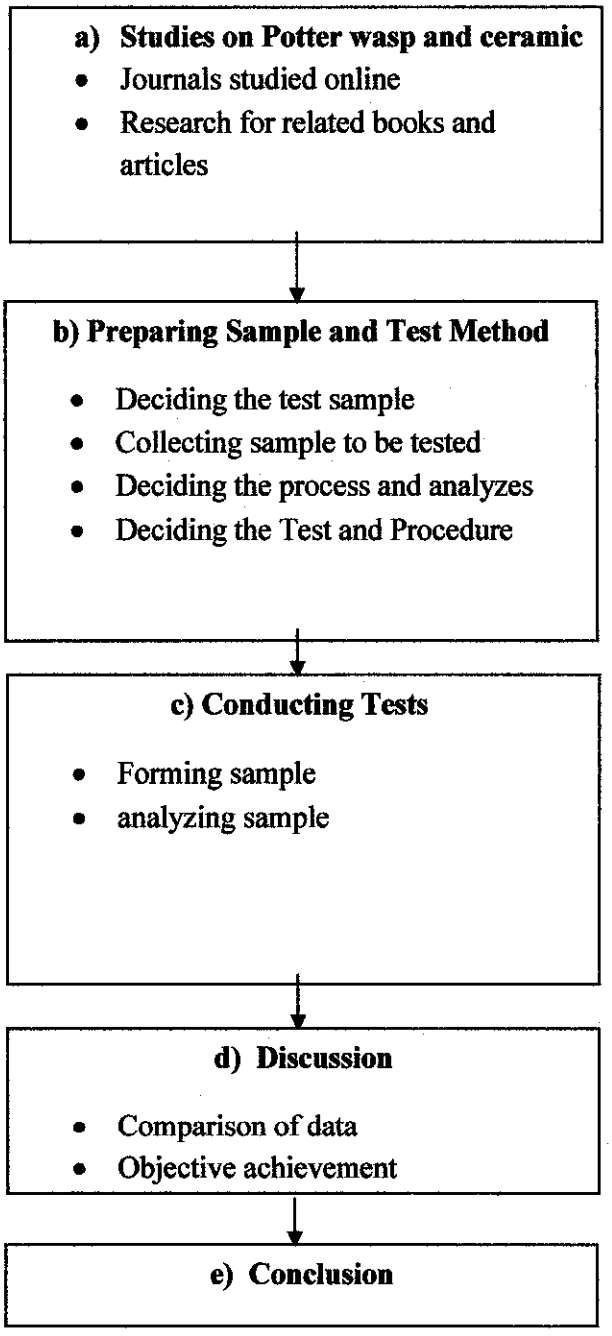


Figure 11: Flow chart of FYP methodology

3.2 Process Flow

3.2.1 Nest Collection

The potter wasp nest has been identified at the UTP main hall. Therefore, in order to ensure that the consistency of the result and sample, the nest will be gathered at the same location.

3.2.2 Physical Selection

Basically, the nest is contaminated with an organism or other type of substances. The physical selection is to remove the contamination as much as possible before proceeding with the next step.

Apparatus;

Magnifying glass, Firing plate / cover plate, Tweezers, 150ml beakers, weighing scale

Procedure:

1. The sample collected is weighted and the data is recorded.
2. Next, the sample collected is placed on a white plate (Firing/cover plate).
3. Magnifying glass is used to magnify the sample to identify the contamination.
4. Tweezers is used to remove the contamination and the clean samples are placed in the beakers.
5. Finally, the sample is weighted and the data is recorded.

3.2.3 Physical separation based on color differentiation

The third part is separating the clay from the nest into several parts by its color and will examine in separate experiment and both type of clay will be compared through specific characteristic with the kaolinite and with the current clay use in the industry.

Procedure:

1. The clean sample again is placed on a white plate (Firing/cover plate).
2. Magnifying glass is used to magnify the sample to identify the color differential between the samples.
3. Tweezers is used to separate the sample based on the color.
4. The separated sample collected is placed in the beaker.
5. Again, the sample is weighted and the data is recorded.

3.2.4 Washing

Washing part is a process to clean up the clay in order to produce pure clay. This part will undergo several processes such as dissolving, filtration with water dissolve and filtration with non-polar solvent.

Apparatus:

Filter Paper, Filter funnel, 150ml Beaker, Conical flask, weighing scales, measuring cylinder, stirring rod, Oven

Procedure:

1. Weight the sample and record.
2. 1:4 volume ratio of the sample will be diluted in the distilled water in a beaker. (Based on density of the solution)
3. The solution is completely stirred by using stirring rod.

Figure 12: Complete Stirred

4. The solution is filtered.

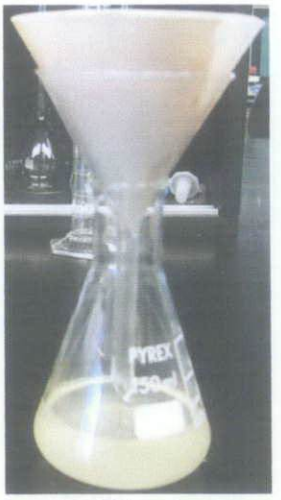


Figure 13: Filtration Process

5. The sample filtered will be heated 65°C to dry the sample.
6. 5ml of filtered solution in the flask is taken to dry and the sample is kept if there is change in the solution color.

Figure 14: 5ml of Filtered solution (From conical Flask)

7. The sample is weight again and weight loss recorded.
8. Then, the washing process is repeated by using ethanol and acetone. (Step 5 is repeated by using 55°C)

3.2.5 Examine

Examine is a part that the clay will undergo several testing and observation. The testing might use several tool and equipment such as XRE, XRD, SEM, EDX, Optical Microscope and other else that will be discuss in the tool and equipment part.

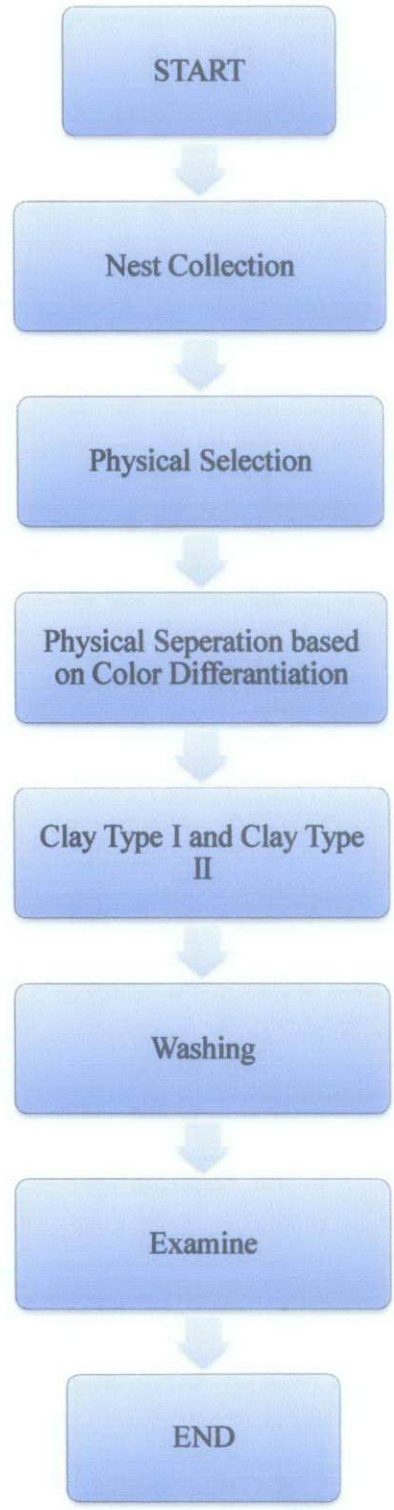


Figure 15: Flow chart for the experiment process

3.3 Tools/ Equipment

For this project, the tools and equipment use will only used at the washing part and testing. Most frequent equipment is the optical microscope which is use to magnify images of small samples after the washing part is completed. The Energy-dispersive X-ray spectroscopy (EDX) is use to determine the element of the compound in the pure clay. In order to emphasis on the structure of the clay, the Scanning Electron Microscope (SEM) is use to capture an images of the sample by scanning it with a high-energy beam of electrons. Differential thermal analysis (DTA/DSC) will measure relative changes in sample temperature during heating. The graph from the DTA will be used to compare and evaluate the result of the clay isolated with the commercial clay. For the firing process, an oven is use to supply the heat to the clay. Thermogravimetric analysis (TGA) is used in measuring weight loss during heating to be compared with the manually washing process. FT-IR stands for Fourier Transform Infrared, the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular finger prints of the sample. FTIR is used to identify the unknown compound (based on group).

3.4 Project Schedule

Table 1 and Table 2 indicate the timeline for the work to be completed.

Table 1: Project activities and timeline planned for Final Year Project 1(FYP1)

	WEEK													
Activity	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Research														
Extended proposal submission														
Proposal Defense														
Project Work														
Interim report submission														

Table 2: Project activities and timeline planned for Final Year Project 2 (FYP2)

	WEEK													
Activity	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Data Collection														
Analysis														
Progress report submission														
Pre-EDX														
Dissertation writing														
Draft Report Submission														
Dissertation Submission (soft bound)														
Technical Paper submission														
Oral Presentation														
Dissertation Submission (Hard Bound)														

3.5 Project Activities

Based on the project schedule there are several adjustments on the time frame due to scheduling the lab and equipment to be used. However, the experiment conducted is still on schedule as planned.

Table 3: Project activities

Activities	Start (week)	Finish (week)
Collecting Sample	Week 4	Week 4
Physical Selection	Week 5	Week 6
Physical Selection based on color differentiation	Week 7	Week 8
Washing by color	Week 8	Week 9
Testing, Examine, Firing	Week 9	Week 12
Report	Week 13	Week 14

CHAPTER 4

RESULT & DISCUSSION

4.1 Result

Table 4 and Table 5 will shows the data recorded based on the procedures.

Table 4: Data collection for Red Color sample

Red		Before (g)	After (g)	weight loss (g)	Loss percentage
Distilled water	Sample	14.99	14.76	0.23	1.53%
	Filter paper	2.46	2.46	0	0
	Sample + Filter Paper	17.45	17.22	0.23	
Ethanol	Sample	13.76	13.74	0.02	0.14%
	Filter paper	2.37	2.37	0	0
	Sample + Filter Paper	16.13	16.11	0.02	
Acetone	Sample	13.7	13.57	0.14	0.95%
	Filter paper	2.34	2.34	0	
	Sample + Filter Paper	16.04	15.91	0.14	

Table 5: Data collection for Brown Color sample

Brown		Before (g)	After (g)	weight loss (g)	Loss percentage
Distilled water	Sample	16.73	16.45	0.28	1.67%
	Filter paper	2.36	2.36	0	0
	Sample + Filter Paper	19.09	18.81	0.28	
Ethanol	Sample	15.43	15.4	0.03	0.19%
	Filter paper	2.4	2.4	0	0
	Sample + Filter Paper	17.83	17.8	0.03	
Acetone	Sample	15.07	14.94	0.13	0.86%
	Filter paper	2.45	2.45	0	
	Sample + Filter Paper	17.52	17.39	0.13	

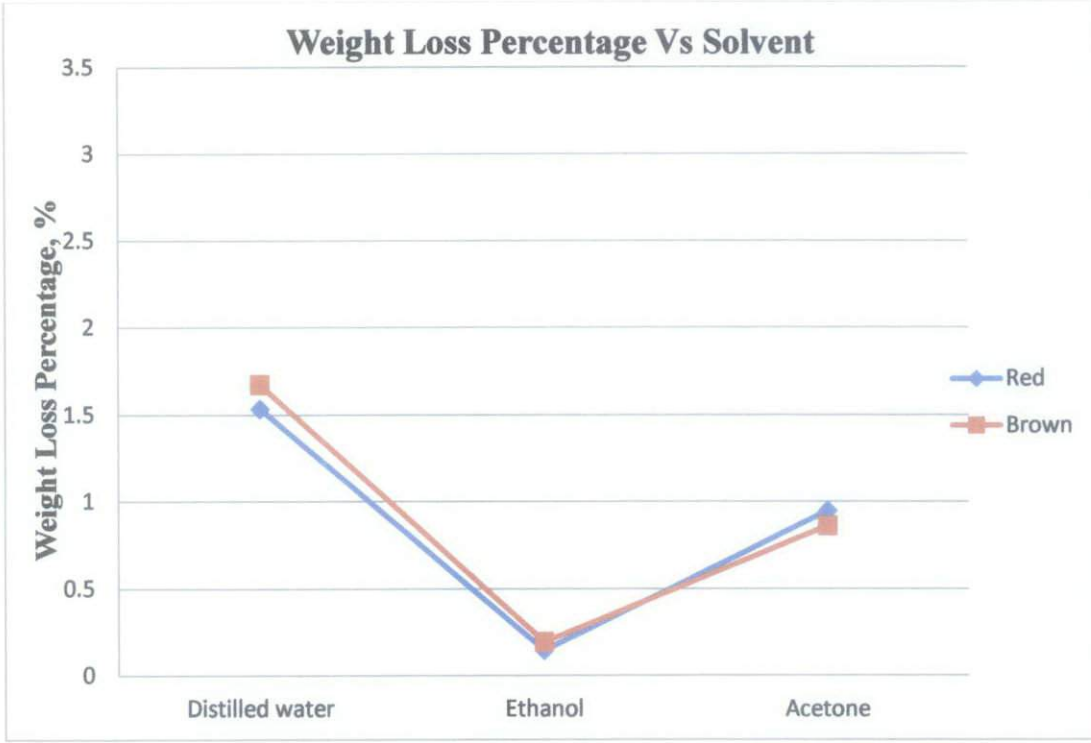


Figure 16: Weight Loss Percentage versus Solvent graph

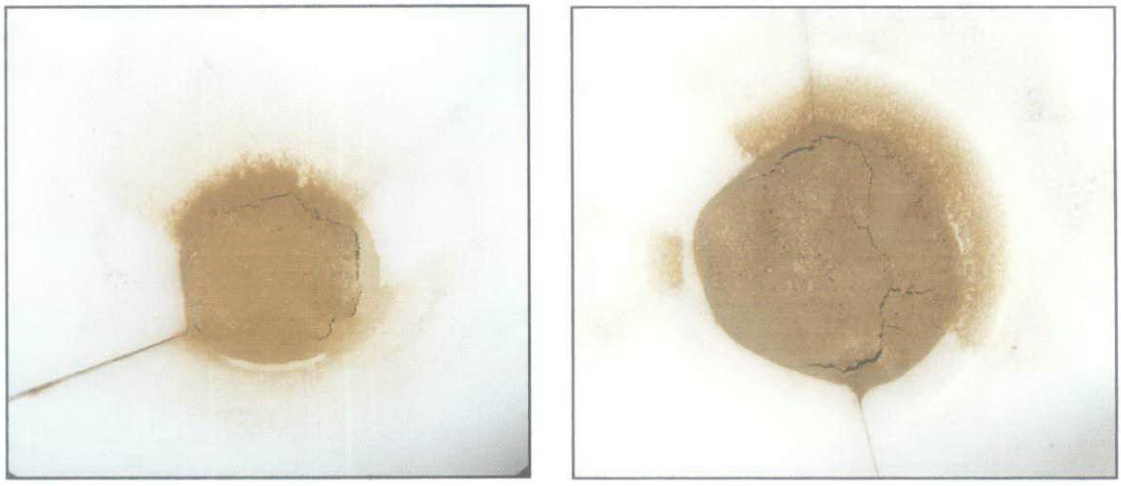


Figure 17: Picture of sample after the Filtration Process

SEM images for Red sample

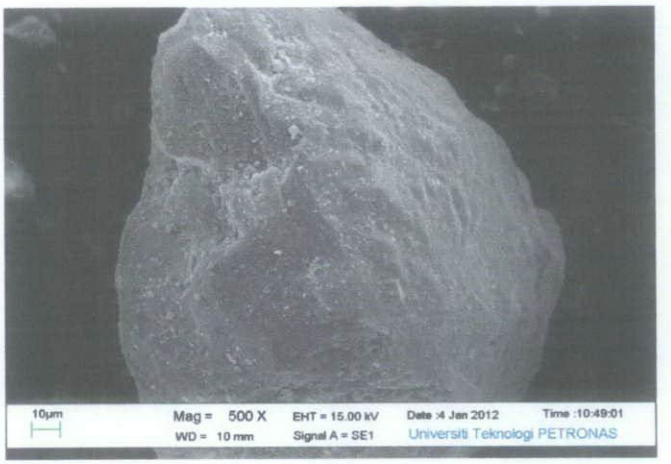


Figure 18: SEM image for the surface of the Red sample

Figure 19: SEM image for microstructure of Red sample

Figure 20: SEM image (Higher magnification) for microstructure of Red sample

SEM images for Brown Color Sample

Figure 21: SEM image for the surface of the Brown sample

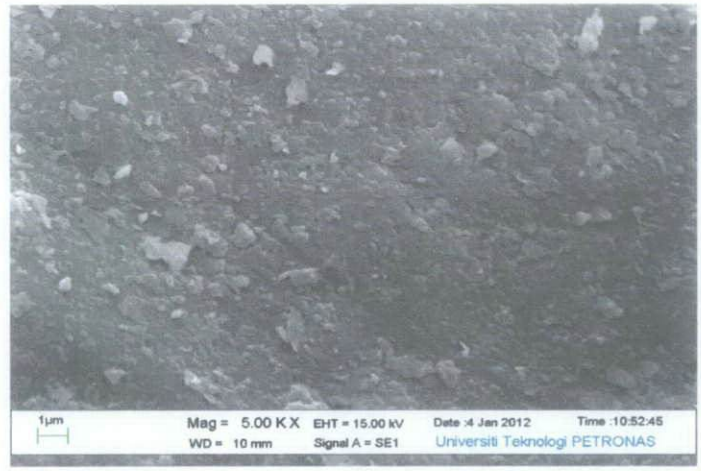


Figure 22: SEM image for microstructure of Brown sample

Figure 23: SEM image (Higher magnification) for microstructure of Brown sample

XRD Graph Red sample

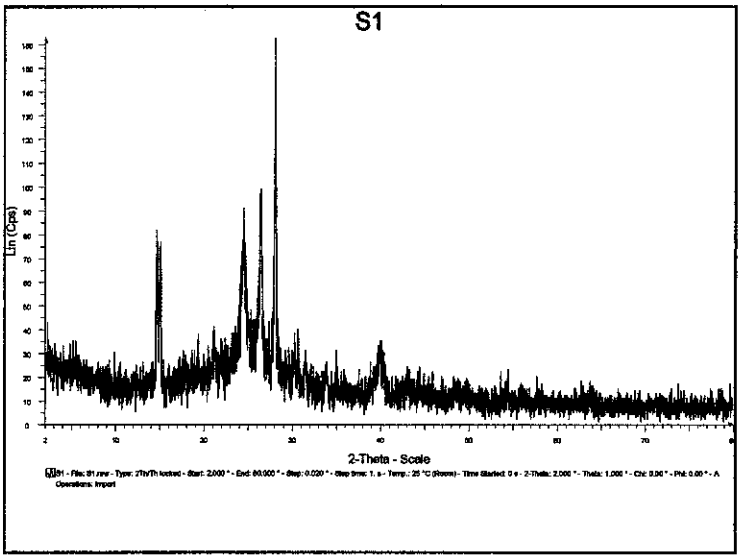


Figure 24: XRD curve for red sample

XRD Graph Brown sample

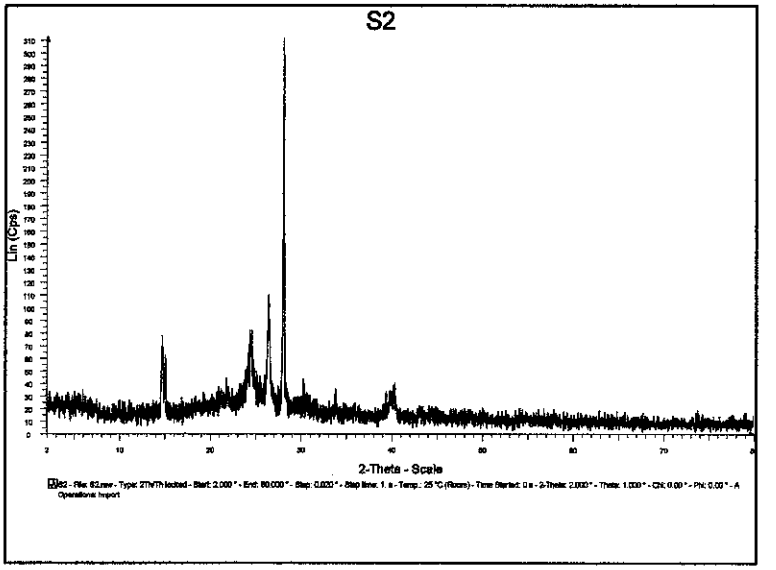


Figure 25: XRD curve for red sample

XRD Table of Composition

Table 6: Table of composition based on XRD graph

Sample	Mineral Composition	Possible Mineral
Red	SiO_2, Al_2O_3, Fe_2O_3,CaO, MgO, TiO_2, K_2O, MnO,SrO, Na_2O Ni, Zn Cr, Cu, P, V	Quartz,Alumina,Hematite, Kaolinite
Brown	SiO_2, Al_2O_3, Fe_2O_3,CaO, MgO, TiO_2, K_2O, MnO,SrO, Na_2O Ni, Zn Cr, Cu, P, V	Quartz,Alumina,Hematite, Kaolinite

FTIR Graph Interpretation Red sample

Table 7: Table of composition based on FTIR graph for Red sample

Band (cm^{-1})	**Transmittance (%)**	**Assingments**
3696.7	17.8	AL-O-H str
3433.68	1.3	O-H str (alcohols)
2066.29	16.6	C-triple bond (alkynes)
1637.72	7.2	N-H bend (primary amines)
1039.03	0.8	C-N (aliphatic amines)
916.86	14.7	O-H bend (carboxylic acid)
693.4	12.1	Si-O str
520.29	8.9	C-Br Str (alkyl halides)
468.75	7.6	Si-O-Fe str

4.2 Discussion

Washing

Based on the data collected, brown sample has greater weight loss compared to the red sample. This is due to the composition or group of the materials. The weight loss percentage for the red sample after been filtered by distilled water is 1.53%, while for the brown sample is 1.67%. The different between the values is approximately 0.14%. The result shows that the brown sample contain more polar compound compared to the red sample.

Figure 26: Final image of the Red (A) and Brown (B) sample

The weight loss graph indicates that the percentage of weight loss is decreasing while being filtered by using the ethanol and bounce to increase while being filtered by using acetone for both of the sample. It shows that the two samples have a compound which can dissolve in the most polar solution (acetone). However, based on the polarity and the result, its shows that both of the samples contain more organic compound which is less polar compared to most polar organic compound.

SEM Image

SEM images was done in 3 different magnify power. For the surface image, the magnification is 500. The second image is to focus on the microstructure for both samples. The magnification use is 5K. The third image is to highlight the microstructure of the sample by using 10K magnification. The higher magnification use is due to the blur image captured which is affected from the type of coating use to coat the sample. However, the images captured are successful.

Based on the observation, red sample show there are sheet staking to form a layer. While, the brown sample image show that there are several sheets in forming layer but the emphasis on the sheet is lack compared to the red sample. Its shows that the tetrahedral with octahedral combination is exist in the red sample; however the compound forming the bonding is yet to be determined. It is difficult to concluded that the sheet form is between $(SiO_4)^{4-}$ with aluminum ions.

XRD

In order to identify the compound of the materials, XRD is used and the counts of the compound which is the amount can be identified. Based on the result, both of the graph shows that both of the sample contain the same compound which are SiO_2, Al_2O_3, Fe_2O_3,CaO, MgO, TiO_2, K_2O, MnO,SrO, Na_2O Ni, Zn Cr, Cu, Phosphate and Vanadium. The different between the red sample and the brown sample is the amount of the compound. The red sample contain high counts of the compound found compared to the brown sample.

FTIR

Result shows several groups of contain in the Red sample. The result is stated in the result part and has been analyze. However, due to the some troubleshooting on the equipment, the testing on the brown sample and the residue from the filtration cannot be completed. The equipment is waiting for the supplier for future inspection.

DTA, TGA & EDX

As planned, the equipment will be used to identify and to analyze the data gather from the sample. However, there are few problems occurs during the study. EDX machine was unavailable since the cooling gas was run out and the FeSEM-EDX at the central lab was under maintenance. TGA equipment is having a failure to the heat controller. Thus, making the heat supply and temperature of the TGA is fluctuated. The DTA machine is unavailable due to the specification of the equipment. The DTA at the lab is meant to use for the polymer sample type. Therefore, the equipment cannot be used for the future analysis of this study since the type of the sample is a non-polymer type.

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.0 Conclusion

The analysis XRD and FTIR shows that clay is mainly constituted of alumina and silica in major quantities and iron, calcium, magnesium oxide and other elements in minor quantities. The loss on ignition value indicates that clay has lower carbonaceous matter and higher mineral matter contents. X-ray diffraction study shows the presence of quartz as major phases. However, the presence composition for the kaolinite is unlikely to be confirming since the fraction is small compare to the other composition. The presence of above minerals was further confirmed by FTIR analysis. In short, the compounds of the kaolonite exist. However based on the result, the clay extracted from the potter wasp nest is not the finest clay which is kaolinite.

5.1 Recommendation

Based on the problems occur, several recommendation would like to be propose such that the further study for the research can be enhanced and additional result can be added to the study thus would be helpful for other researcher to gain an extra information from the study. An improvement that can be applied such as a data from DTA can be used to classify and identify the quality of the clay extracted. For future study, the data gathered from the DTA can be compared to recent commercial clay. In order to increase the reliability of the result for future work, more advance equipment should be used in order to reduce the error in data collecting. It would be great if the SEM is equipped with better coating for a better image and result. It is recommended that future analysis will have the data from TGA, FTIR and EDX.

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