SILICON DIOXIDE RECYCLING FROM PALM OIL MILL FUEL ASH WASTE (POMPAW) BY USING ACID LEACHING ULTRASOUND WAVE (ALUW)

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Thesis is submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

JANUARY 2017

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DEDICATION

By the name of Allah, the most Gracious and the most Merciful

I humbly dedicate my thesis to both of my beloved parents,
Rusli Bin Norani & Siti Sawinah Binti Abd Tassin

To my supportive supervisor,
PM.Dr.Ir Said Nurdin

To every each of my siblings,

To my sweety pie,
Zulaikha Mohd Subri,

To all FKKSA's Staffs and lecturers,

Thank you for all your prayers and encouragements all the way

ACKNOWLEDGEMENT

I would like to express my special appreciation and thanks to my supervisor, PM Dr. Ir. Said Nurdin .You have been a brilliant mentor for me. I would like to thank you for your never ending support during my tenure as research student under your guidance, for giving insightful comments and suggestions of which without it, my research path would be a difficult one . Your advice on my research has been valuable.

A special thanks to my family. Words cannot express how grateful I am to my mother, father and also my beloved siblings. Your prayer for me was what sustained me thus far.

I am also indebted to the Ministry of Higher Education and Universiti Malaysia Pahang for funding my study.

I would also like to thank all of my friends who supported me in writing, and motivate me to strive towards my goal. I am sincerely grateful to the staffs of Chemical Engineering and Natural Resources Faculty who helped me in many ways and made my stay in UMP pleasant and unforgettable.

ABSTRACT

Palm oil mill fuel ash waste (POMFAW) is highly potential renewable source of silicon dioxide. Silicon dioxide is also well-known as silica has been applied widely in many industries these days. Therefore, it is important to use this approach to utilize the POMFAW since Malaysia nowadays continuously producing biomass wastes. This paper demonstrates experimentally the method on silicon dioxide recycling from palm oil mill fuel ash waste (POMFAW) by using acid leaching ultrasound wave (ALUW). In this study, there are 3 parameters has been investigated which are the treatment time, acid concentration and irradiation power in order to identify the optimum condition to extract silica from POMFAW by using ultrasonic-assisted acid leaching method. Thus, the maximum amount of silica that can be extracted from POMFAW is up to 84.12% by using the sulphuric acid at 2M concentration at 450W of sonication power at the treatment time of 30 minutes.

ABSTRAK

Abu kelapa sawit (POMFAW) merupakan sumber yang boleh diperbaharui yang berpotensi tinggi untuk menghasilkan silicon dioksida. Silikon dioksida yang juga dikenali sebagai silika diaplikasikan secara meluas dalam industry pada hari ini.Oleh yang demikian, pendekatan untuk meghasilkan silica berasaskan POMFAW ini amat sesuai diaplikasikan kerana Malaysia kerap menghasilkan biomass pada masa kini. Kajian ini menunjukkan kaedah bagi mengitar semula silicon dioksida daripada abu kelapa sawit (POMFAW) dengan menggunakan teknik asid larut lesap yang dibantu oleh gelombang ultrasound. Penyelidikan ini merangkumi 3 parameter untuk dianalisa iaitu tempoh rawatan, kepekatan asid dan juga kuasa ultrasound bagi mendapatkan keadaan yang optimum dalam proses pengkitaran semula silikon dioksida menggunakan kaedah ini.Oleh itu, jumlah maksimum silikon dioksida yang boleh dikitar semula adalah sebanyak 84.12% pada kepekatan asid sulfurik sebanyak 2M dan sebanyak 450 W kuasa ultrasonic pada tempoh rawatan selama 30 minit.

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M	Molarity
W	Watt

LIST OF ABBREVIATIONS

POMFAW	Palm Oil Mill Fuel Ash Waste
ALUW	Acid Leaching Ultrasound Wave
EFB	Empty Fruit Brunches
PKS	Palm Kernel Shell
MF	Mesocarp Fiber
POME	Palm Oil Mill Effluent
OPT	Oil Palm Trunks
OPL	Oil Palm Leaves
OMF	Oil Palm Fronds
SEM	Scanning Electron Microscopy
XRF	X-Ray Fluorescent

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Malaysia is one of the world's second largest producer of palm oil after Indonesia. In 2011, it was recorded that 5 million hectares of the land in the country were yielded by the palm oil plantations (Islam et al., 2015). Thus, Malaysia plays an important role to fulfil the developing global demands for oil and fats constantly. There are a few wastes generated after the oil palm fruits harvesting, palm oil processing or during oil palm trees replantation, such as empty fruit branches, palm kernel shells, mesocarp fiber, palm oil mill effluent , palm oil leaves and palm oil fronds. Foo & Hameed (2009), stated that the biomass wastes produced by the palm oil which yield about 90% and constitute only 10% of oil from the overall production of palm oil plantation were specified as the highly potential agro waste which can be utilized as the renewable source of energy or feedstock of downstream product variously. According to Awalludin et al., (2015), as this industry has expanding widely, the significant amount of palm oil waste has been generated and has led to the problem of biomass waste overload. Limited utilization of POMPAW which were disposed in a huge amount annually could lead to health hazard and environmental problems (Islam et al., 2015). Therefore, pollutions of air, river, sea and groundwater have increased in recent years due to the large amount of waste produced.

1.2 Motivation

- Silica has wide range of applications in industry such as food processing, oral care dentifrices and textile industries.
- The existing methods of silica synthesis in industry are time consuming and produced in low yield.
- Recovery of silica from POMPAW using ALUW are beneficial because the raw material is free of charge and the recovery process is simple and low cost.

1.3 Problem Statement

- Synthesis of silica from non-renewable resources such as phosphorus slag and soil creates an environmental issue.
- Malaysia produces a lot of wastes such as POMPAW that containing high silica.
- Eco-friendly silica synthesis from renewable material such as POMPAW.

1.4 Objectives

To recover silicon dioxide from palm oil mill fuel ash waste (POMPAW) using acid leaching ultrasound wave (ALUW) based on variations of treatment time, acid concentration and sonication power.

1.5 Scopes of Study

The following are the scopes of this research:

- Pre-treatment and characterization of POMFAW.
- Silica recovery using acid leaching ultrasound wave (ALUW).
- Yield determination and Silica analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 Silicon Dioxide (SiO_2)

2.1.1 Introduction of Silicon Dioxide

Silicon dioxide is also known as silica is the name of a group of minerals which exhibit the most abundant elements in the earth's crust which are silicon and oxygen. Silica is having chemical formula of SiO_2 as it composed of one atom of silicon and two atoms of oxygen.Silica mainly presents in crystalline form such as α, β-quartz, coesite, stishovite and others. Nevertheless, it is also rarely found in amorphous (non-crystalline) form such as opal, hyalite, natural silica glass and others (Lee et al., 2016). According to the Eurosil, crystalline silica appears about 12% of the earth crust naturally. It is involved in all mining and quarrying activities. In fact, crystalline silica exists in all naturally occurring materials that have been extracted from the ground since the past few decades such as sand, gravel, dimension stones, metallic and non-metallic mineral ores. The sand on beaches containing up to 99% of silica and it has been acts as the basic material in creating most of the rocks on earth. Silicon dioxide is a hard compound. It is inert chemically and has a high melting point. Those valuable properties of silica has fulfilled the various applications in industry.

2.1.2 Applications of Silicon Dioxide

Conferring to Ima Europe, Crystalline form of silica is the most common type used in industry such as quartz and cristobalite. They are usually sold in the form of granular materials with the grain size distribution lies in between the range 0.06-0.2 millimetres. It is known as silica sand. Silica has been widely employed as the basic raw materials supporting the industrial revolution as well as information technology revolution since the last few decades. The finest form of silica applied as the functional filler for paints, plastics, rubbers, polymer compounds, sealants and adhesives. It offers resistance against abrasive actions and chemical attack. The typical examples such as self-cleaning exterior wall coatings and heavy-duty offshore or the marine paints. The nature of silica provides its importance towards plastics as the encapsulating electronic

materials. In ceramic industry, Silica has been ground to the finer particles to produce a major constituent of ceramic glazes. Furthermore, in glass industry, silica exhibits as the major ingredient of all types of glass such as flat glass and optical glass. Since the crystalline silica has a higher melting point compared to iron, copper and aluminium, thus it qualifies castings to be formed by pouring molten metal into moulds made up from silica and a binder. It is necessary in the production of specialist products such as jewellery, dental bridges, aviation turbines and golf club. Other than that, silica sand generally used as the filtration medium in water industry to extract solids from wastewater. Besides, silica is significantly applied by metallurgical industry as the raw material for silicon metal and ferrosilicon metal. Furthermore, the special grades of silica sand with rounded to sub rounded particles is ordinarily employed in oil production industry as to stimulate the well production of oil. The sand is forced into the oil bearing strata and increases its permeability thereby supporting the flow of oil into the well. Moreover, silica performs as soil conditioner or carrier fertilizer and also animal feed additives in agriculture industry. In information technology industry, silica is employed as the raw material in the manufacture of silicon chips, the heart of the computer world.

2.1.3 Silicon Dioxide Market Analysis In Asia

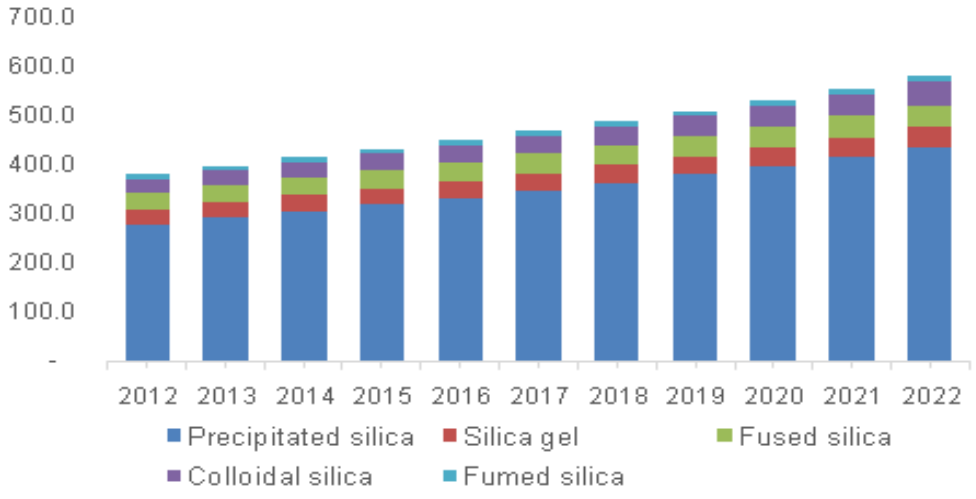

Figure 1. 1: silica market volume in US, 2012-2022 (Kilo Tons), Grand view research, (2016)

Since Silica exhibits the special properties in nature, it has met the global demand in various industries. According to Grand View Research 2016, silica market size globally was estimated at 3200 kilo tons in 2014. Silica demands in developing countries including India, China and Indonesia is expected to be increased over the estimated period since the rubber consumption in tire production in automotive industry has increasing significantly. Besides, Europe and North America as well is probably will bring the positive impact on the silica market demand over the estimated period as the demand for green tires has growing impressively. Fumed silica segment was valued at over USD 50.0 million in 2014 as it is widely applied in the paints & coatings manufacturing industry. In Asia Pacific, specifically in India and China is estimated to give the positive impact over the forecast period particularly in the growth of coatings industry on account of rising in construction and infrastructural development in the country.

2.1.4 Effect of Silicon Dioxide on Human Health

Normohammadi et al.,(2016**)** reported that, the occupational injuries and health hazards due to unsafe work environment mostly recorded from construction industries. The demolition of building leads to the emission of dust into the environment. Construction dust composed of hazardous compounds such as crystalline silica, lead and other toxic as well as carcinogenic agents. Thus, exposure to silica is considered as an important hazard in this industry. All sort of demolition activities especially such as breaking, cutting, crushing and grinding will produce are subjected to the silica exposure. Occupational exposure to silica dust may cause silicosis in construction workers. Besides, epidemiological studies approved that silica dust leads to chronic obstructive pulmonary disease and lung cancer in many workers in construction site.

2.1.5 Synthesis of Silicon Dioxide via Sol-Gel Method

Based on previous study, Li & Qiao (2016) explained that, the benefits of having unique properties such as large specific surface area and pore volume, easily modified surface, and good biocompatibility have made mesoporous silica as an exceptional choice over other mesoporous materials in many cases. Few years back, researches have tried hardly to enhance the utilization of mesoporous silica in industry and they have excellently prepared products by using variety of solid wastes as the low cost raw source.

Nevertheless, most of these synthesis routes of silica are still based on sol-gel method. Sol-gel method was implemented to synthesis mesoporous silica with special morphology and different functional groups due to the important of producing ordered meso-structure. However, this existing method of silica synthesis has a difficulty on controlling the composition and homogeneity of the precursors in the larger scale of sol-gel process which limits the preparation of mesoporous silica in industry. Thus, in this study, the easy and low cost approach will be promoted to synthesis the silica from palm oil mill fuel ash waste.

2.1.6 Silicon Dioxide Recovery from Waste Based On Previous Study

Based on previous study, there were various raw materials used to extract silica from waste. However, none of the research about extraction of silica from palm oil mill fuel ash waste recorded.

Table 1: Silica Recovery from Waste Based on Previous Study

AUTHOR	FINDINGS
Bakar et al., (2016)	Production of amorphous silica from rice husk
Elineema et al., (2013)	Nanoporous silica recovery from waste products phosphate fertilizer industry
Karshigina et al.,(2015)	Silicon dioxide recovery from phosphorus slag
Kumar et al.,(2015)	Silica recovery from rice husk ash
Li & Qiao, (2016)	Preparation of mesoporous silica using fly coal ash

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2.2 Palm Oil Mill Fuel Ash Waste (POMPAW)

2.2.1 Palm Oil Plantation

As reported by Awalludin et al., 2015, Oil palm tree (Elaeis guineensis) is a tropical palm plant which was originated from Africa. Oil palm trees has becoming one of the world's most profitable agricultural sources. Back in 100 years before, oil palm tree plantations was in a relatively small-scale crop in Africa. Fortunately, oil palm trees can cultivate well in Malaysian climate. Thus, undoubtedly, it has been the most crucial agricultural crop in the country as well as the national economic sources. Malaysia is responsible to up rise the oil palm industry globally through the significant contributions and continuous commitment. For periods, Malaysia has appointed as the one of the most productive palm oil producers in the world. According to the bar chart below, the production of palm oil in Malaysia has increasing annually from 1975-2011 (MPOB).

2.2.2 Palm Oil Biomass

Oil palm tree is a multipurpose crop that brings the positive impact to the socio-cultural activities of the inhabitants of the area in which it cultivates. However, the cultivation of oil palm tree and its related industries will produce the biomass wastes massively. Biomass generally defined as all organic matters or compounds that produced either from crops, forestry or marine life. Other wastes such as sewage and municipal solid waste are also described as biomass. Biomass is a type of hydrocarbon material which contains carbon, hydrogen, oxygen and nitrogen. However, some of biomass containing a small amount of sulphur and other inorganic substances. The biomass plant is produced through photosynthesis during its lifespan. In palm oil industry, the biomass wastes generated after the oil palm fruits harvesting, palm oil processing or during oil palm trees replantation are including empty fruit brunches (EFB), palm kernel shell (PKS), mesocarp fiber (MF), palm oil mill effluent (POME), oil palm trunks (OPT),oil palm leaves (OPL) and oil palm fronds(OMF).

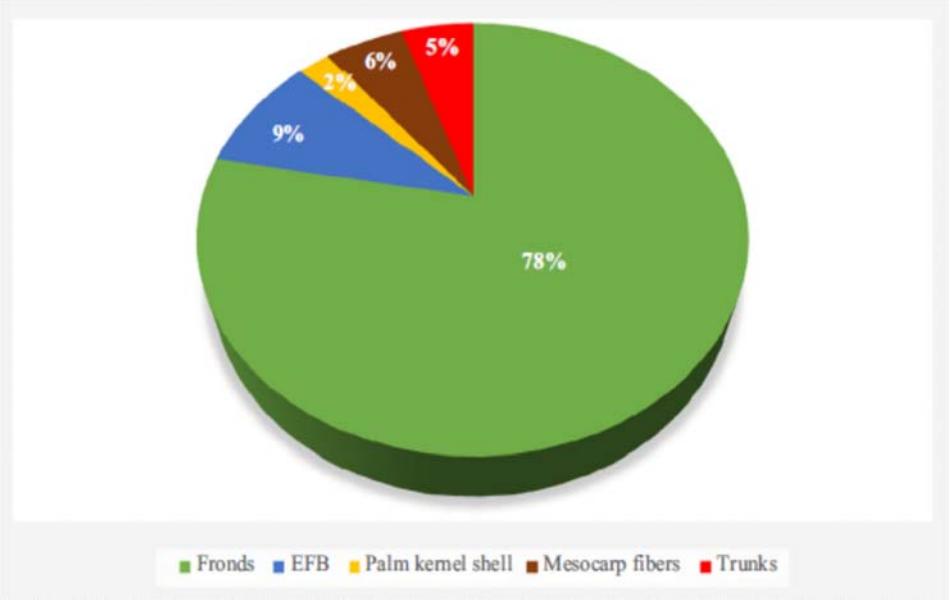

Figure 2.1: Availability of palm oil waste in Malaysia (Hosseini et al., 2015)

In palm oil plantations, palm oil constitutes only 10% of the overall biomass. Another 90% are rejected as wastes. Averagely, in order to produce one ton crude palm oil, it requires five tons of fresh fruit brunches. Presently, Malaysia has approximately 417 productive palm oil extraction mills entirely. These mills collectively can generate more than 12.4 million tons of EFB and about 44 million tons of OPF annually. In 2012, it was recorded that about 83 million tons of oil palm biomass waste were available all around Malaysia. It is predicted, the generation of palm oil biomass wastes keep on increasing up to 100 million tons yearly at 2020.

2.2.3 Conversion of Palm Oil Biomass Waste into Valuable Products

Awalludin et al., (2015) described that, biomass waste can be transformed to valuable products through mechanical and thermal or thermochemical processes. The table below generally explain about the processes and its products.

Table 2: Conversion Process of Palm Oil Wastes into Valuable Products, (Awalludin et al., 2015)

Mechanical Processes	Thermal and Thermochemical Processes
1. Shredding and size reduction • Nutrient recycling • Mechanical pulping • Ruminant feed • Board making	1. Liquefaction/solvolysis • Bio-oil • Resin Precursor
2. Densification and drying • Pallets and briquetes	2. Pyrolysis • Fuel gases • Charcoal
	3. Gasification • Fuel gases (Hydrogen)
	4. Combustion • Heat and steam (fuel ash)
	5. Extraction • Extractives

2.2.4 Palm Oil Mill Fuel Ash Waste (POMFAW)

Based on the table above, direct combustion is the one of the thermal or thermochemical process of biomass waste conversion. Combustion is the process of releasing heat when any combustible materials are allowed to burn in the presence of air or oxygen. Thus, the biomass waste are combusted directly in the power plant as the source of energy to increase steam, which later spread through a turbo-alternator to create electricity. Combustion of biomass waste is able to produce hot gases at the temperature up to 1000℃ at the condition where the moisture content of the biomass is less than 50%. In order to produce 1 kg of the oil palm, it requires about 0.075-0.1 kWh of electricity and needs around 2.5 kg of steam for the process. Thus, this process is defined as one of the existing way of the utilization of the palm oil waste in Malaysia. Palm oil mill fuel ash waste (POMFAW) or else known as palm oil fuel ash (POFA) is a collected ash which formed from the combustion of fibers, shell and empty fruit branches in the palm oil mill boiler at the temperature between 800℃ – 1000℃ as a fuel to generate the electricity. It was recorded that on 2007, Malaysia produced about 3 million tons of POMFAW (Johari et al., 2012). The abundance of POMFAW which associates with silica rich properties paves the way of its usage in various industries especially for concrete and construction materials (Ranjbar et al., 2016) .According to Safiuddin et al., (2011), Silicon dioxide possesses the very high composition in POMFAW which yields about 65.3%.

Figure 2.2: Palm oil mill fuel ash waste (POMFAW)

2.3 Acid Leaching Ultrasound Wave (ALUW)

2.3.1 Separation Process

Referring to Geankoplis, 2014, in order to separate one or more components in a mixture, the mixture is brought into contact with another phase and the two-phase pair can be gas-liquid, vapor liquid, liquid-liquid, or fluid-solid. In this research, the separation process involves the separation of the silicon dioxide from the solid of palm oil mill fuel ash waste (POMFAW) by using solid-liquid separation process known as the leaching process.

2.3.2 Acid Leaching Extraction.

As stated by Tang &Steenari.,(2015), there are various approaches have been studied to utilize the solid waste ashes and the leaching process followed by the thermal treatment or solidification are the most common method used currently. Most of the treatment methods are functioning as to stabilize ash as the construction material. However, in some cases, leaching treatment employed as to recover specific metal from solution and then combined with further treatment. The leaching treatment of metals from ash is strictly influenced by the type of leachate, pH and liquid to solid ratio used as well as by the chemical compounds in the ash matrix also the surface morphology of the ash. Barati et al., (2011) stated that mostly of the impurities can be separated from silica by a simple acid leaching step because most of them are likely to condense at the temperatures different from the oxidation of silicon monoxide and they are not intimately attached to the silica particles.

2.3.3 Ultrasonic Assisted Acid Leaching

Applications of ultrasound in hydrometallurgy industry mainly as to assist the ore leaching process has expanding extensively in these recent era. Ultrasonic energy is a kind of reinforcement method that widely used for the prominence of leaching efficiency. As stated by Zhang et al., (2016), Ultrasonic consist of a series of longitudinal wave with various density and spread around through the medium. The formation of bubbles followed by the implosion resulting from the ultrasonic energy irradiating solution creates

an important “acoustic cavitation”. The high temperature and pressure differences generated caused the microscopic turbulence to be produced and the boundary layer becomes thinner around the particles with much bubbles collapsing. The importance of ultrasonic energy conveys the effects of optical, electrical, mechanical, thermal, chemical, and biological and many more. The acoustic cavitation and the heat effect are the special effect triggered by the mechanical action during reaction. It has clearly informed that ultrasonic energy is a very efficient tool to enhance reaction rates as it induces some mechanical and chemical effects. Xing et al., (2015) proved that cavitation bubbles formed upon the sonication increase the chemical reaction rate. According to Albero et al.,(2015), when the ultrasound radiation transmitted through the medium, it generates a disturbance that will creates expansion and compression cycle eventually. Bubbles or cavities are produced when the intensity of the ultrasound is higher. The mechanical effect of the sonication induces the penetration of solvent into solid matrices and it provides excellent contact between the solid and the extractant as well thus resulting in good recovery of the product. Ultrasonic wave has been effectively implemented to be added with dilute sulphuric acid which can be associated together to work at mild conditions(Rehman et al.,2013).

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter is about the material and methodology used in order to recycle silica from POMFAW by using acid leaching ultrasound wave. In this section, there are a few subtopics will be discussed such as the pre-treatment process and the characterization of POMFAW, the mixing process of POMFAW and the sulphuric acid followed by the ultrasonic-assisted process. The precipitation process of silica as well as the drying process until the crude silica formed also will be conversed in this section.

3.2 Materials and Apparatus

The raw POMFAW was collected from Palm Oil Mill at Felda Lepar Hilir 5, Gambang, Pahang. The chemical used to recycle Silicon Dioxide from POMFAW are sulphuric acid and sodium carbonate. The chemicals were purchased from Sigma Aldrich. The apparatus used were the beaker, hot plate, magnetic stirrer, pH meter and ultrasonic bath.

3.3 Methodology of the Experiment

3.3.1 Pre-treatment and characterization of POMFAW

The raw POMFAW was dried and calcined in the furnace at the temperature at 800℃ for 2 hours to remove foreign materials and unburnt fibres. The calcined raw POMFAW was sieved through a sieve 125 μm to possess the uniform particles size of the raw POMFAW. The pre-treated raw POMFAW was then characterized by Scanning Electron Microscope (SEM) for surface morphology and also XRF for elemental composition analysis of the pre-treated raw POMFAW.

Figure 3.1: Pre-treated POMFAW

3.3.2 Mixing Process of POMFAW and Acid

10g of pre-treated POMFAW was added with various concentration of Sulphuric acid H_2SO_4 (1M- 5M) and heated on the heating plate under the solution's boiling temperature of 100℃ with the constant stirring power of 300 rpm for 1 hour.

Figure 3.2: Mixing process of pre-treated POMFAW and acid solution

3.3.3 Ultrasonic-Assisted Process

The experiment was proceeded by exposing the sample with the ultrasound produced by ultrasonic bath working at (150-750 W) sonication power. The treatment timewas varied from 20 minutes to 60 minutes. The frequency of the ultrasonic bath was at 45 kHz filled with the sufficient amount of water at the room temperature at 25℃.

Figure 3.3: Ultrasonic-assisted process

3.3.4 Precipitation Process

After the ultrasound treatment, the sample was filtered by using the vacuum filter. The residue was removed and the clear solution was retained. The experiment was continued by heating the clear solution on the heating plate for 1 hour under the solution's boiling temperature, 100℃ and continuously stirred at 300 rpm. During the process, sodium carbonate was employed to increase the pH of the solution. The pH of the solution was retained at 2.4 to form the precipitation.

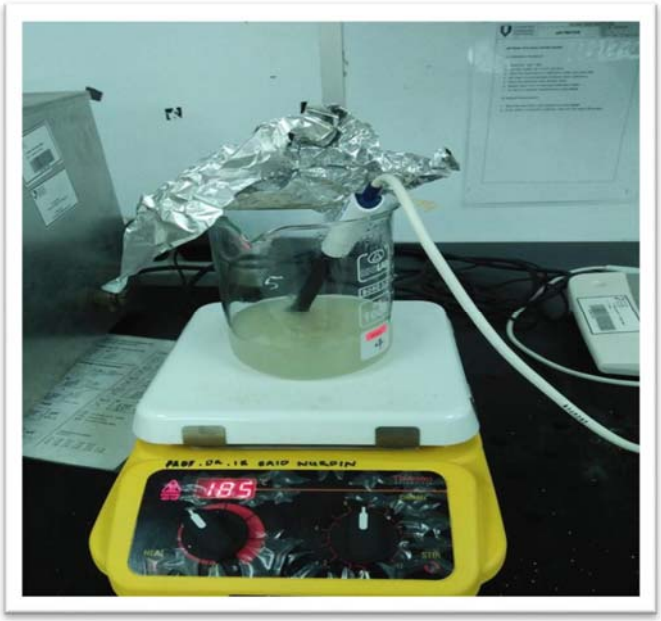

Figure 3.4: Precipitation Process

3.3.5 Drying Process

The solution was allowed to cool to room temperature for about 1 hour. The sample was filtered by using the vacuum filter. The precipitation retained on the filter was dried in the oven at 100℃ for 24 hour. The solid formed was finally ready for XRF analysis.

Figure 3.5: Drying Process

Figure 3.6: Final product of Silica

3.3.6 Yield Determination

$$\text{Silica yield (\%)} = \frac{Mproduct}{Mraw} \times 100$$

M (product) = Percentage of Silica in the final product

M (Raw) = Percentage of Silica in RAW POMFAW

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter is explaining about results and discussion. There are a few subtopics will be covered in this section. Firstly the pre-treatment process and the characterization of POMFAW. Secondly, the parameters that have been highlighted in this study which are the effects of acid concentration, the sonication power and the treatment time. Lastly, the three-dimensional (3D) response plot and the optimization of the process by the Design Expert.

4.2 Pre-treatment and characterization of POMFAW

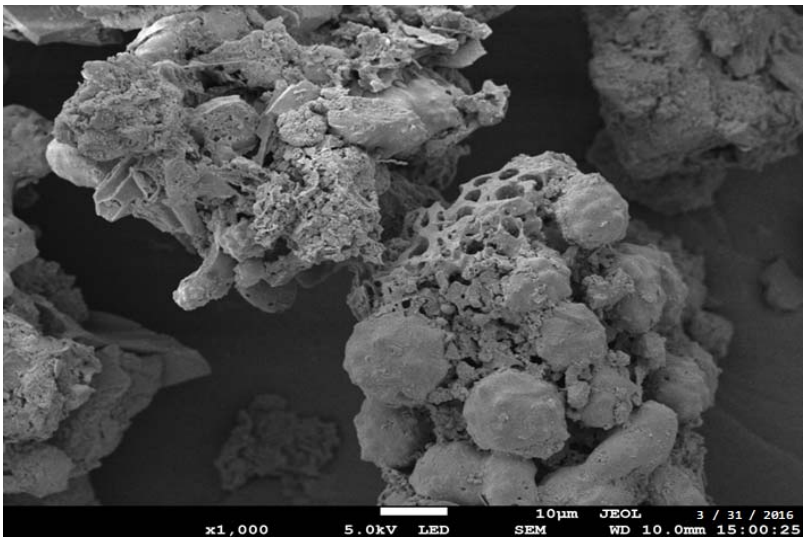

Figure 4.1: SEM Micrograph of pre-treated POMFAW

The morphology of the silica in the treated POMFAW was characterized by the SEM analysis. The image reveals the existence of spherical and porous structure of silica. According to Deshmukh et al., (2012), there are many residual pores are scattered within the ash as it was burnt in the air, thus resulting a highly porous structure of silica. The chemical composition of oxide groups present in the treated POMFAW was then analyzed by the XRF. From the analysis, the silica (SiO_2) possessed the highest percentage by weight which is 48.50%. Table 1 below indicates the percentage of composition of oxide

groups that exist in the pre-treated POMFAW. By referring to Mujah (2016), it marks that the silica has the highest percentage in POMFAW which is 63.72%. The difference in composition of elements exhibit in POMFAW obtained by another researchers were because of the numerous operating conditions of the palm oil mills as well as the sources of the raw materials.

Table 3: Percentage of composition of oxide groups present in pre-treated POMFAW

	Oxide Group	Percentage of composition (%)
1.	SiO_2	48.50
2.	K_2O	10.74
3.	Fe_2O_3	6.10
4.	CaO	5.59
5.	P_2O_5	2.77
6.	SO_3	1.42
7.	Al_2O_3	1.37
8.	MgO	1.19

4.3 Effect of acid concentration

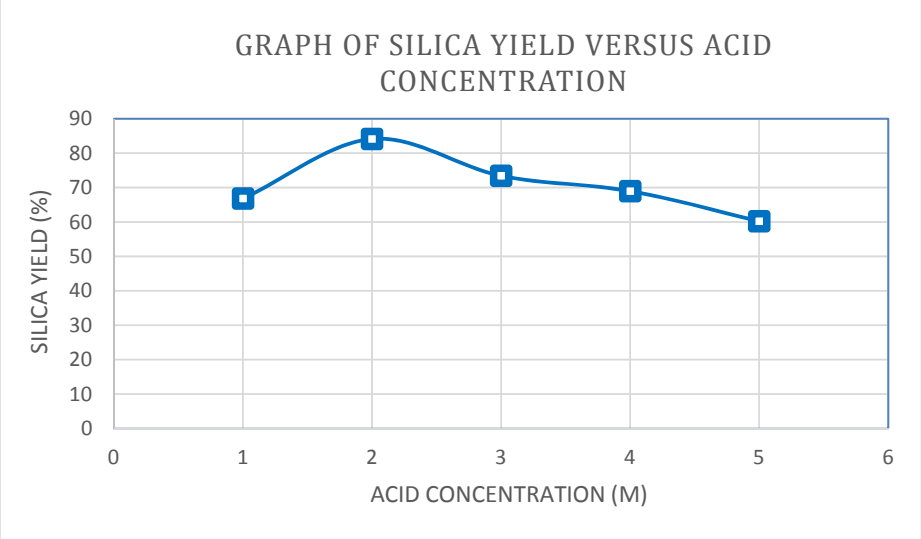

Figure 4.2: Graph of Silica yield versus acid concentration

The silica yield was examined by the XRF analysis at different concentration of sulphuric acid as the leaching agent in between the range of 1M-5M as shown in figure 4.2. At the concentration of 1M to 2M, the yield of silica increased. The maximum yield of extracted silica is up to 80% at 2M concentration of sulphuric acid. According to Xing et al., (2015), the higher acid concentration boosted the diffusion of the acid from bulk solution to the surface of the sample and increased reaction rate at the interface. However, too acidic environment has slow down the reaction rate as the amount of silica recovered tends to decrease gradually after 2M of the sulphuric acid employed.

4.4 Effect of sonication power

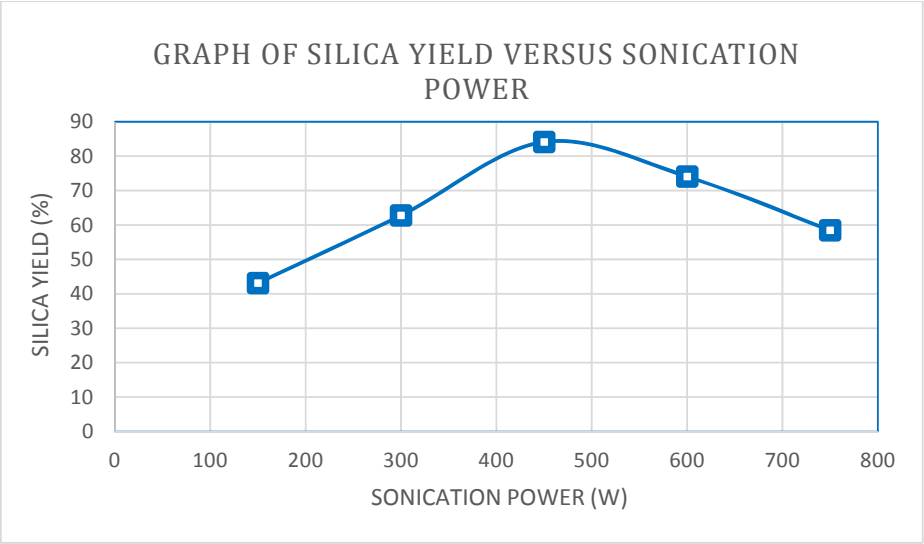

Figure 4.3: Graph of Silica yield versus sonication power

Figure 4.3 clarifies the effect of sonication power on the yield of silica obtained from the process. The maximum amount of silica recovered is achieved at the sonication power of 450 W. The maximum amount of extracted silica is achieved at the sonication power of 450W then it has declined regularly after that point. Inferring to Xing et al., (2015), as the ultrasound power increasing, the number of cavitation bubbles increasing as well. Then, the cavitation bubble collapse will be more quickly. Thus, by increasing the ultrasonic power, it will reduce the threshold limit, thus the rate of leaching will be increased.

4.5 Effect of treatment time

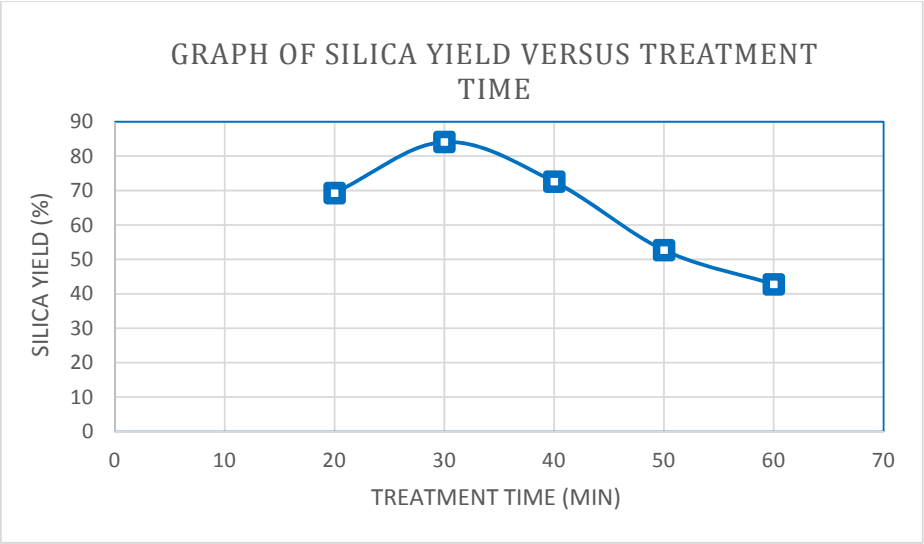

Figure 4.4: Graph of Silica yield versus treatment time

The effect of treatment time upon the yield of extracted silica is as displayed in figure 4.4 .At the first 30 minutes of exposing the sample in the ultrasonic bath, the silica yield increased from 69% up to 84.12%. However, the recovery started to drop progressively after 30 minutes of the treatment time. According to Zhang et al., (2016), the boundary layer of solid-liquid reaction interface has weaken and the particles smashed stronger to each other as the ultrasonic cavitation and the mechanical action employed to the process. Thus, it has induced the surface area of the particles which has indirectly accelerated the mass transfer diffusion in the process. Therefore, ultrasonic has triggered the leaching rate as it shorten the leaching time. It clearly portrays that the optimum treatment time to recover the maximum amount of silica is 30 minutes.

4.6 Three-Dimensional (3D) response surface by Design Expert

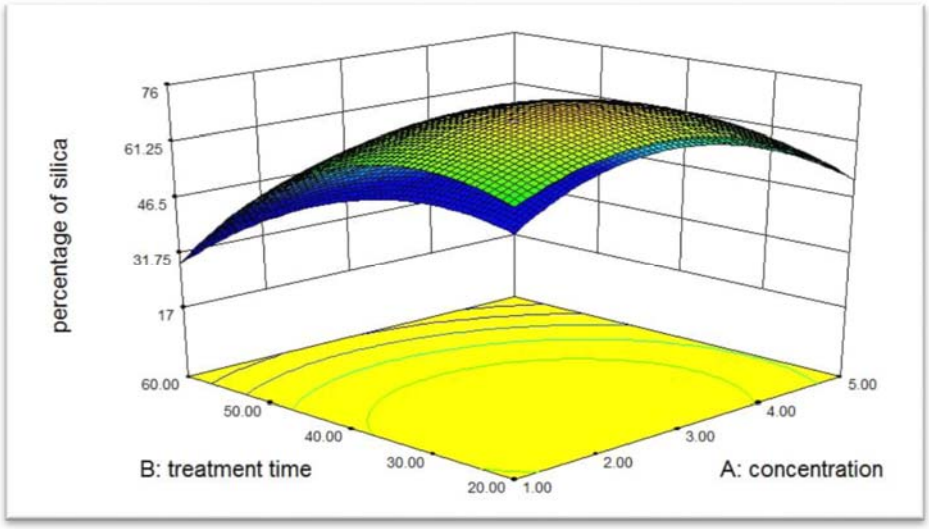

Figure 4.5: Three-Dimensional response surface (Treatment time and Concentration relationship)

By observing the 3D plot presented in Figure 4.6 which is the response surface of the relationship between treatment time and acid concentration, it portrays that the maximum yield of silica could be obtained at the treatment time between 20 minutes up to 30 minutes and at the concentration ranges between 2M to 3M.

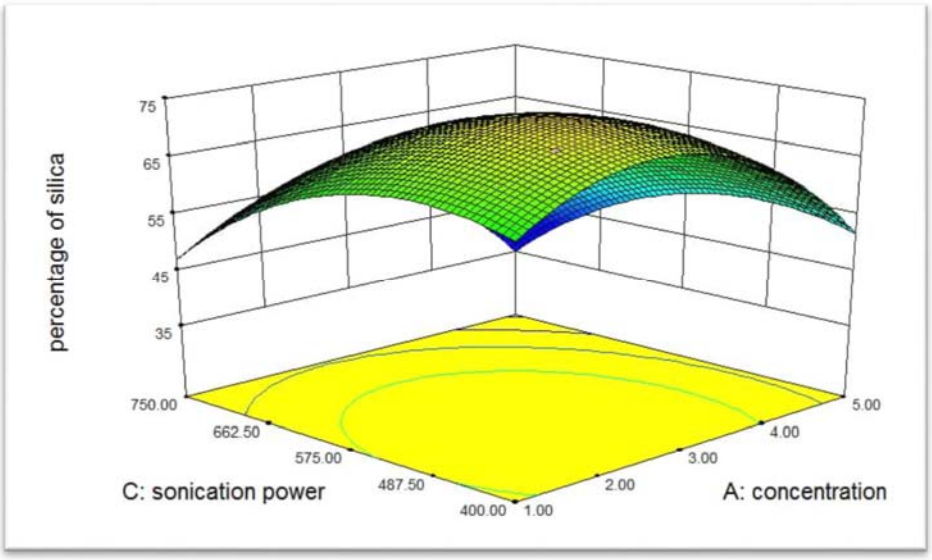

Figure 4.6: Three-Dimensional response surface (Sonication power and Concentration relationship)

Figure 4.7 above illustrates the three dimensional response of the relationship between sonication power and the concentration. It proves that the maximum amount of silica could be achieved at the sonication power between 450 W to 600 W at the concentration between 2M to 3M.

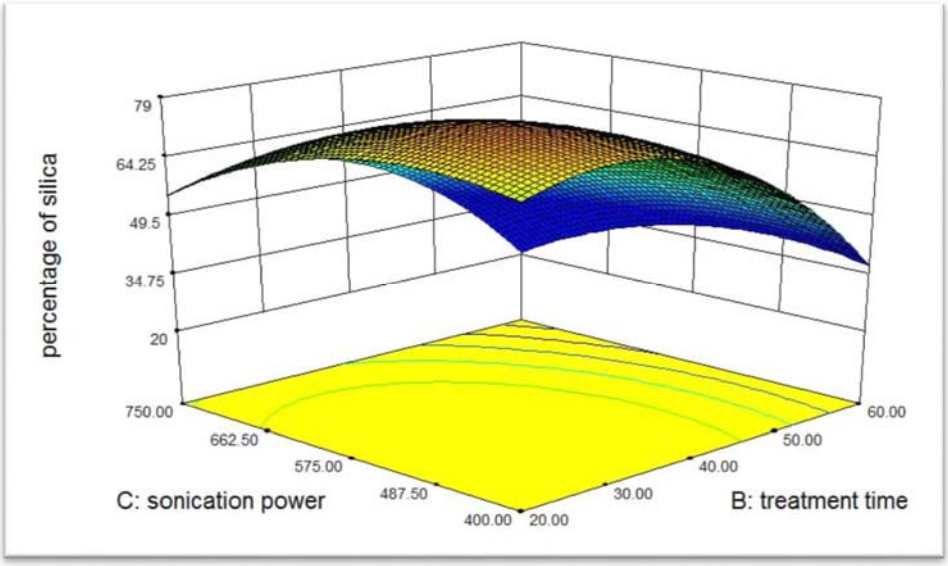

Figure 4.7: Three-Dimensional response surface (Sonication power and Treatment time relationship)

The three-dimensional response plot of the relationship between sonication power and treatment time is demonstrated in the figure 4.8 above. It validates that, at the sonication power ranges between 450 W to 600 W and the treatment time between 20 minutes to 30 minutes, the maximum yields of silica could be gained.

4.7 Optimization by Design Expert

According to Design Expert, there are a few optimum conditions are predicted to maximize the silica yield. The predicted optimum conditions from the design expert result with their confirmed values respectively such as listed in table below. The percentage of silica from the design expert result is compared with the experimental value. The calculation of error is calculated based on the formula presented below. The acceptable percentage error is below 10%.

$$\%\ error = \frac{M(exp) - M\ (DE)}{M(exp)} x\ 100$$

M (exp) = Percentage of Silica from the experimental value

M (DE) = Percentage of Silica from the Design Expert

Table 4: Optimum Condition Results from Design Expert

Concentration (M)	Treatment time (min)	Sonication power (W)	Percentage of Silica	Error (%)
2.68	26.12	453.60	77.9609	7.32
3.00	30	450	77.8506	7.45
2.00	30	450	77.4585	7.92

The table above shows that, the maximum yield of silica could be achieved is 77.9609% and has the least value of error which is 7.32% if compared with the experimental result. The optimum condition to produce silica at the highest yield based on the design expert result is at the acid concentration is 2.68M and exposed with the sonication power at 453.60 W for 26.12 minutes.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In conclusion, POMFAW can be successfully employed as the free source of silica. Experimentally, it is proven that the maximum amount of silica which is 84.12% can be extracted by using the 2M concentration of sulphuric acid at 30 minutes of treatment time. The optimum sonication power for this study is 450 W. However, inferring to the optimization results by the Design Expert, there are slight differences between the optimum conditions predicted with the experimental results. The suggested optimum conditions by the Design Expert is by using 2.68M acid , assisted by 453.60 sonication power and treated up to 26.12 minutes. This predicted condition provides 77.9609% of the Silica yield and possess the very small error if being compared with the experimental condition which is 7.32%. According to the previous study, there are a lot of sources used to recover silica. However, there is none of the research done yet to recover silica from POMFAW assisted by ultrasonic acid leaching. Thus, this research is considered worth to be studied.

5.2 Recommendation

There are a few recommendation have to be taken into consideration in order to improve this research. The parameters of this study should be widen so it will provides clearer view about this research. Secondly, the use of ultrasonic bath in this research should be replaced with ultrasonic to improve the leaching process. This is because, in an ultrasonic bath, the cavitation occurs non-conformable and uncontrollably distributed through the tank. The sonication effect is low intensity and unevenly spread. Thus the repeatability and scalability of the process is very poor to be compared with the ultrasonic probe-type devices due to its highly focus and evenly distribution of ultrasonic power input which might affects the efficiency of the leaching process.

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APPENDIX

Table 5: Data of effect of acid concentration

Concentration (M)	Percentage of Silica (%)	Yield (%)
1	32.4	66.80412
2	40.8	84.12371
3	35.6	73.40206
4	33.45	68.96907
5	29.2	60.20619

Table 6: Data of effect of sonication power

Sonication Power(W)	Percentage of Silica (%)	Yield (%)
150	20.91	43.1134
300	30.45	62.78351
450	40.8	84.12371
600	35.92	74.06186
750	28.35	58.45361

Table 7: Data of effect of treatment time

Treatment time (min)	Percentage of Silica (%)	Yield (%)
20	33.6	69.27835
30	40.8	84.12371
40	35.2	72.57732
50	25.56	52.70103
60	20.74	42.76289

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CERTIFICATE OF ANALYSIS (COA)

To / Attn	Norazuwan bin Rusli		
Address	FKKSA, UMP		
Tel No	011-29801410	Fax No	
Sample Lab No	2016/601	No of sample	5

Sample marking : 2016/601 (1)
Sample description : Raw POFA
Date of sample received : 08-12-2016
Date reported : 09-12-2016

RESULTS:

No	Parameter	Results	Unit	Test Method
1.	Silicon Dioxide (SiO2)	48.50	%	Quantexpress (Full Analysis) by XRF S8 Tiger
2.	Potassium Oxide (K2O)	10.74	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Iron (III) Oxide (Fe2O3)	6.10	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Calcium Oxide (CaO)	5.59	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Phosphorus Pentoxide (P2O5)	2.77	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	Chlorine (Cl)	1.52	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	Sulphur Trioxide (SO3)	1.42	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Aluminium Oxide (Al2O3)	1.37	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Magnesium Oxide (MgO)	1.19	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Titanium Dioxide (TiO2)	0.23	%	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Zirconium Dioxide (ZrO2)	0.15	%	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Manganese Oxide (MnO)	0.08	%	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Rubidium oxide (Rb2O)	0.06	%	Quantexpress (Full Analysis) by XRF S8 Tiger
14.	Strontium Oxide (SrO)	0.05	%	Quantexpress (Full Analysis) by XRF S8 Tiger

15.	Copper Oxide (CuO)	0.03	%	Quantexpress (Full Analysis) by XRF S8 Tiger
16.	Zinc Oxide (ZnO)	0.02	%	Quantexpress (Full Analysis) by XRF S8 Tiger
17.	Chromium (III) Oxide (Cr2O3)	0.02	%	Quantexpress (Full Analysis) by XRF S8 Tiger
18.	Nickel Oxide (NiO)	73	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
19.	Bromine (Br)	35	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

Sample marking : 2016/601 (2)
Sample description : Sample 1
Date of sample received : 08-12-2016
Date reported : 09-12-2016

RESULTS:

No	Parameter	Results	Unit	Test Method
1.	Silicon Dioxide (SiO2)	40.80	%	Quantexpress (Full Analysis) by XRF S8 Tiger
2.	Iron (III) Oxide (Fe2O3)	5.97	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Sodium Oxide (Na2O)	3.20	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Phosphorus Pentoxide (P2O5)	3.18	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Potassium Oxide (K2O)	1.81	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	Sulphur Trioxide (SO3)	0.69	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	Aluminium Oxide (Al2O3)	0.60	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Calcium Oxide (CaO)	0.59	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Titanium Dioxide (TiO2)	0.39	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Magnesium Oxide (MgO)	0.22	%	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Chlorine (Cl)	0.12	%	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Zirconium Dioxide (ZrO2)	0.02	%	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Manganese Oxide (MnO)	0.01	%	Quantexpress (Full Analysis) by XRF S8 Tiger
14.	Rubidium oxide (Rb2O)	0.01	%	Quantexpress (Full Analysis) by XRF S8 Tiger
15.	Strontium Oxide (SrO)	87	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
16.	Arsenic Trioxide (As2O3)	57	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

17.	Copper Oxide (CuO)	42	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
18.	Nickel Oxide (NiO)	37	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

Sample marking : 2016/601 (3)
Sample description : Sample 2
Date of sample received : 08-12-2016
Date reported : 09-12-2016

RESULTS:

No	Parameter	Results	Unit	Test Method
1.	**Silicon Dioxide (SiO2)**	**33.45**	%	Quantexpress (Full Analysis) by XRF S8 Tiger
2.	Iron (III) Oxide (Fe2O3)	7.02	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Sodium Oxide (Na2O)	3.73	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Phosphorus Pentoxide (P2O5)	2.53	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Potassium Oxide (K2O)	1.53	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	Calcium Oxide (CaO)	0.59	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	**Sulphur Trioxide (SO3)**	0.58	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Aluminium Oxide (Al2O3)	0.43	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Titanium Dioxide (TiO2)	0.28	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Magnesium Oxide (MgO)	0.15	%	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Chlorine (Cl)	0.08	%	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Molybdenum Trioxide (MoO3)	0.02	%	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Manganese Oxide (MnO)	0.01	%	Quantexpress (Full Analysis) by XRF S8 Tiger
14.	Rubidium oxide (Rb2O)	80	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
15.	Copper Oxide (CuO)	74	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
16.	Strontium Oxide (SrO)	71	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
17.	Arsenic Tricxide (As2O3)	49	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
18.	Zirconium Dioxide (ZrO2)	38	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

Sample marking : 2016/601 (4)
Sample description : Sample 3
Date of sample received : 08-12-2016
Date reported : 09-12-2016

RESULTS:

No	Parameter	Results	Unit	Test Method
1.	**Silicon Dioxide (SiO3)**	28.35	%	Quantexpress (Full Analysis) by XRF S8 Tiger
2.	Iron (III) Oxide (Fe2O3)	6.62	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Sodium Oxide (Na2O)	4.81	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Phosphorus Pentoxide (P2O5)	2.54	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Potassium Oxide (K2O)	1.04	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	**Sulphur Trioxide (SO3)**	0.52	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	Calcium Oxide (CaO)	0.43	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Aluminium Oxide (Al2O3)	0.36	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Titanium Dioxide (TiO2)	0.27	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Magnesium Oxide (MgO)	0.12	%	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Chlorine (Cl)	0.08	%	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Zirconium Dioxide (ZrO2)	0.01	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Strontium Oxide (SrO)	78	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
14.	Rubidium oxide (Rb2O)	58	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
15.	Arsenic Trioxide (As2O3)	45	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
16.	Copper Oxide (CuO)	41	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

Sample marking : 2016/601 (5)
Sample description : Sample 4
Date of sample received : 08-12-2016
Date reported : 09-12-2016

RESULTS:

No	Parameter	Results	Unit	Test Method
1.	**Silicon Dioxide (SiO2)**	20.74	%	Quantexpress (Full Analysis) by XRF S8 Tiger

2.	Iron (III) Oxide (Fe2O3)	4.59	%	Quantexpress (Full Analysis) by XRF S8 Tiger
3.	Sodium Oxide (Na2O)	4.40	%	Quantexpress (Full Analysis) by XRF S8 Tiger
4.	Phosphorus Pentoxide (P2O5)	2.71	%	Quantexpress (Full Analysis) by XRF S8 Tiger
5.	Potassium Oxide (K2O)	0.88	%	Quantexpress (Full Analysis) by XRF S8 Tiger
6.	Titanium Dioxide (TiO2)	0.40	%	Quantexpress (Full Analysis) by XRF S8 Tiger
7.	Sulphur Trioxide (SO3)	0.33	%	Quantexpress (Full Analysis) by XRF S8 Tiger
8.	Calcium Oxide (CaO)	0.33	%	Quantexpress (Full Analysis) by XRF S8 Tiger
9.	Aluminium Oxide (Al2O3)	0.32	%	Quantexpress (Full Analysis) by XRF S8 Tiger
10.	Magnesium Oxide (MgO)	0.11	%	Quantexpress (Full Analysis) by XRF S8 Tiger
11.	Chlorine (Cl)	0.05	%	Quantexpress (Full Analysis) by XRF S8 Tiger
12.	Zirconium Dioxide (ZrO2)	0.02	%	Quantexpress (Full Analysis) by XRF S8 Tiger
13.	Manganese Oxide (MnO)	70	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
14.	Rubidium oxide (Rb2O)	56	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger
15.	Copper Oxide (CuO)	41	ppm	Quantexpress (Full Analysis) by XRF S8 Tiger

The above analysis is based on the sample submitted by the customer.

NORHASLINA ABU SAMAH
SCIENCE OFFICER