

ABSTRACT

The production of biodiesel is greatly increasing due to its environmental benefits. However, the production costs are still rather high, compared to petroleum-based diesel fuel. The introduction of a solid heterogeneous catalyst in biodiesel production could reduce its price, becoming competitive with diesel also from a financial point of view whereas the production separation process becomes easier and reduce formation of water and soap. The purpose of the work to study biodiesel production by transesterification of Jatropha oil with methanol in a heterogeneous system, using six types of Zirconia catalyst (ZrO_2 , W/ZrO_2 , SO_4/ZrO_2 , $OTS-ZrO_2$, $OTS-W/ZrO_2$ and $OTS-SO_4/ZrO_2$). The resulting catalysts were characterized by Thermogravimetric Analysis (TGA), Fourier Transformed Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Analysis (EDAX) techniques. The catalytic potential of catalysts was verified by transesterification reactions of Jatropha oil with methanol in batch reactor. The $OTS-SO_4/ZrO_2$ showed the highest percentage Fame conversion and suggested as a best catalyst. In conclusion, there are two properties that make $OTS-SO_4/ZrO_2$ a very suitable catalyst in the transesterification reaction. The first property is the presence of sulfate which is better than tungsten as an active site and the second, the alkylsilation of the active site by octadecyltrichlorosilane which increase the hydrophobic character of the catalyst.

ABSTRAK

Dewasa ini, pengeluaran biodiesel menunjukkan peningkatan yang memberangsangkan oleh kerana manfaatnya yang mesra alam. Walaubagaimanapun, kos pengeluarannya masih cukup tinggi, jika dibandingkan dengan bahan bakar diesel berasaskan minyak bumi. Pengenalangan mangkin pepejal heterogen dalam penghasilan biodiesel boleh mengurangkan kos pengeluaran, menjadikan ianya kompetitif dengan harga diesel juga dari pandangan perimis kewangan disebabkan proses pemisahan produk menjadi lebih mudah dan mengurangkan pembentukan air dan sabun. Tujuan kajian ini adalah untuk mengkaji penghasilan biodiesel melalui pengtransesteran minyak Jarak dengan metanol dalam sistem heterogen, dengan menggunakan enam jenis mangkin Zirkonia (ZrO_2 , W/ZrO_2 , SO_4/ZrO_2 , $OTS-ZrO_2$, $OTS-W/ZrO_2$ dan $OTS-SO_4/ZrO_2$). Mangkin yang dihasilkan dikarakterisasi by Termogravimetri Analysis (TGA), Spektroskopi Inframerah Transformasi Fourier (FTIR), Scanning Electron Microscopy (SEM) dan Energy dispersive X-Ray Analysis (EDAX) teknik. Potensi katalitik mangkin disahkan oleh reaksi pengtransesteran minyak jarak dengan metanol dalam reaktor (batch). $OTS-SO_4/ZrO_2$ menunjukkan peratusan penukaran tertinggi Methyl Ester (FAME) dan dicadangkan sebagai mangkin terbaik. Sebagai kesimpulan, terdapat dua faktor yang menjadikan $OTS-SO_4/ZrO_2$ sebagai mangkin sangat sesuai dalam proses pengtransesteran. Faktor pertama adalah kerana kehadiran sulfat yang lebih baik daripada tungsten sebagai halaman aktif dan yang kedua adalah kerana alkylsilation dari halaman aktif dengan octadecyltrichlorosilane menyebabkan peningkatan sifat hidrofobik mangkin.

TABLE OF CONTENTS

CHAPTER	TOPICS	PAGE
	TITLE	i
	STATEMENT	ii
	DECLARATION	iii
	DEDICATION	iv
	ACKNOWLEDGEMENTS	v
	ABSTRACT	vi
	ABSTRAK	vii
	TABLE OF CONTENTS	viii
	LIST OF ABBREVIATIONS	xi
	LIST OF TABLES	xii
	LIST OF FIGURES	xiii

CHAPTER 1 INTRODUCTION

1.1	Research Background	1
1.2	Objective of Research	3
1.3	Scopes of Study	3
1.4	Problem Statement	3
1.5	Benefit and significant of Research	4

CHAPTER 2 LITERATURE REVIEW

2.1	Catalysis	5
2.1.1	Zirconia (ZrO ₂)	7
2.1.2	Tungsten (W)	8
2.1.3	Sulfate (SO ₄)	8
2.1.4	Octadecyltrichlorosilane (OTS)	9
2.1.5	Organofunctional silane Modifications	
	-A Hydrophobic Catalyst	10
2.2	Biodiesel Production – General	11
2.3	Jatropha Oil and Its Benefits	12
2.4	Characterization of Bulk and Surface Structure	13

CHAPTER 3 METHODOLOGY

3.1	Chemical and Reagents	14
3.2	Catalyst Preparation for Biodiesel Production	14
3.2.1	OTS -ZrO ₂	14
3.2.2	OTS -W/ZrO ₂	16
3.2.3	OTS - SO ₄ ²⁻ /ZrO ₂	17
3.3	Characterization of Catalyst	18
3.3.1	Fourier Transformed Infrared Spectroscopy (FTIR)	18
3.3.2	Scanning Electron Microscopy (SEM) And Energy Dispersive X-Ray Analysis (EDAX)	18
3.3.3	Thermogravimetric Analysis (TGA)	19
3.4	Transesterification of Jatropha Oil with Methanol	19
3.5	Preparation of Standard for Calibration Curve	20

3.5.1	Analysis of Chemical Composition	21
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CHAPTER 4 RESULT AND DISCUSSION

4.1	Characterization of Catalyst	23
4.1.1	Fourier Transformed Infrared Spectroscopy (FTIR)	23
4.1.2	Scanning Electron Microscopy (SEM) Analysis	24
4.1.3	Energy Dispersive X-Ray Analysis (EDAX)	26
4.1.4	Thermogravimetric Analysis (TGA) Analysis	27
4.2	The Effect of Active Sites	29
4.3	The Effect of Alkylsilation	30

CHAPTER 5 CONCLUSIONS AND RECOMMENDATIONS

5.1	Conclusion	31
5.2	Recommendation of Future Work	32

REFERENCES	33
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APPENDIX	35
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LIST OF SYMBOLS/ ABBREVIATIONS

μ	-	Micron (10^{-6})
EDAX	-	Energy Dispersive X-Ray technique
FAME	-	Methyl Ester
FTIR	-	Fourier Transformed Infrared Spectroscopy
g	-	Grams
GC	-	Gas Chromatography technique
mL	-	Mililiter
mm	-	Milimeter
OTS	-	Octadecyltrichlorosilane
OTS-SO ₄ /ZrO ₂	-	Alkylsilation to Sulfated Zirconia
OTS-W/ZrO ₂	-	Alkylsilation to Tungstated Zirconia
OTS-ZrO ₂	-	Alkylsilation to Zirconia
SEM	-	Scanning Electron Microscope technique
SO ₄	-	Sulfate
SO ₄ /ZrO ₂	-	Sulfated Zirconia
TGA	-	Thermogravimetric Analysis
W	-	Tungsten
W/ZrO ₂	-	Tungstated Zirconia
Zr(OH) ₄	-	Zirconium hydroxide
ZrO ₂	-	Zirconia

LIST OF TABLE

TABLE	TITLE	PAGE
2.2	Fatty Acid Composition of Jatropha Curcas	13
3.1	Gas Chromatography temperature program Set Up for Product Analysis	21

LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Cycle of the development of heterogenous Catalyst	5
2.2	Chemical structure of Octadecyltrichlorosilane	9
2.3	Mechanism of complete reaction of OTS on the support surface to form well oedered layer	11
2.4	Mechanism of incomplete reaction of OTS on the support surface	11
3.1	A Schematic representation for the preparation of OTS-ZrO ₂	15
3.2	A Schematic representation for the preparation of OTS-W/ZrO ₂	16
3.3	A Schematic representation for the preparation of OTS-SO ₄ /ZrO ₂	17
3.4	A Schematic Apparatus Setup for Transesterification Process	20
4.1	FTIR spectra of (a) OTS-ZrO ₂ (b) OTS-SO ₄ /ZrO ₂	23
4.2	Scanning electron micrograph of the catalyst (a) ZrO ₂ , (b) OTS-ZrO ₂ , (c) OTS-SO ₄ /ZrO ₂	24
4.3	EDAX Analysis Data for OTS-SO ₄ /ZrO ₂	26
4.4	Thermogravimetric Analysis for W/ZrO ₂ sample	28
4.5	Thermogravimetric Analysis for OTS-W/ZrO ₂ sample	28
4.6	Percentage(%) of FAME conversion in Transesterification of Jatropha Oil with Methanol	29

**BIODIESEL PRODUCTION FROM JATROPHA OIL BY USING
HETEROGENEOUS HYDROPHOBIC MODIFIED ZIRCONIA**

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**A thesis submitted in fulfillment of the
requirements for the award of degree of
Bachelor of Engineering (Chemical)**

**Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang**

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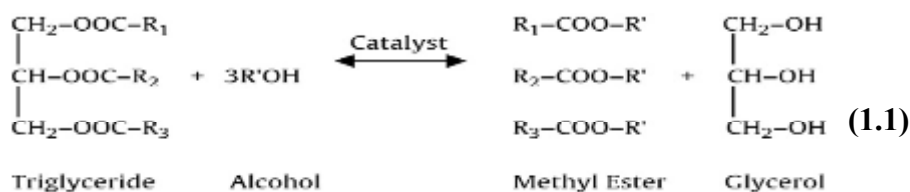
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CHAPTER 1

INTRODUCTION

1.1 Research Background

Due to the increase in the price of petroleum crude and products and environmental concerns about air pollution caused by the combustion of fossil fuels, the search for alternative fuels has gained importance. Biodiesel (fatty acid methyl esters) derived from the transesterification of vegetable oils or animal fats with methanol are potential substitutes for petroleum-based diesel fuels. Compared with conventional diesel, biodiesel has the advantages of being biodegradable, renewable, non-toxic, and have low pollutant emissions (especially SO_x) (Amish *et al.*, 2008). If the biodiesel valorized efficiently at energy purpose, so would be benefit for the environment and the local population, job creation and provision of modern energy carriers to rural communities. At present, biodiesel is usually produced by reacting methanol and vegetable oil in a batch stirred tank reactor using a liquid alkaline catalyst. The catalyst cannot be recovered from the reactor and is instead neutralized and disposed of as a waste stream. Biodiesel has been produced by transesterification of triglyceride (vegetable oils) to methyl esters with methanol using sodium or potassium hydroxide dissolved in methanol as catalyst, as represented by Equation 1.1 (Lopez *et al.*, 2009).



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Although transesterification using a conventional alkali catalyzed process gives high conversion levels of triglycerides to their corresponding methyl esters in short times, the reaction has several drawbacks: it is energy intensive; recovery of glycerin is difficult; the catalyst has to be removed from the product; alkaline wastewater requires treatment and free fatty acids and water interfere with the reaction (Martino *et al.*, 2007). Therefore, for the development of an environmentally benign process and the reduction of the production cost, a new process using heterogeneous catalyst should be introduced (Martino *et al.*, 2007).

A heterogeneously-catalyzed process offers a number of advantages over current technology, namely the simplification and economization of the catalyst removal step, the removal potential by forming emulsions and the simplification of downstream separation processes. The transesterification reaction can be catalyzed by both acids and bases (Martino *et al.*, 2007).

In general, metal oxides have been used extensively either as such or as supports in conjunction with other active components for many industrial oxidation, reduction and acid–base-catalyzed reactions. The surface of zirconium oxide is known to possess all these catalytic activities. Zirconium oxide, or zirconia, when modified with anions, such as sulfate ions, forms a highly acidic or superacidic catalyst that exhibits superior catalytic activity to catalyze many reactions. It has been widely used to catalyze reactions such as hydrocarbon isomerization, methanol conversion to hydrocarbons, alkylation, acylation, esterification, etherification, condensation, nitration, cyclization, etc. Thus, modification of Zirconia (ZrO_2) in terms of active sites and hydrophobic characteristic form an important class of catalysts, as is evident from the voluminous research that has appeared in the last decade. Moreover, these catalysts show a promising future in that they can provide environmentally clean processes for the chemical industry of the next millennium (Kaewta *et al.*, 2008).

1.2 Objective of Research

The objective of this research is to study the production of biodiesel from jatropha oil by using heterogenous hydrophobic modified zirconia.

1.3 Scope of Study

The scopes of study are listed as follows:

- i. To produce several types of modified Zirconia catalyst, ZrO_2 , W/ZrO_2 , SO_4/ZrO_2 , $OTS-ZrO_2$, $OTS-W/ZrO_2$ and $OTS-SO_4/ZrO_2$ for biodiesel production from Jatropha oil.
- ii. To investigate the characteristic of designed catalysts by using several method, TGA, FTIR, FESEM and EDAX.
- iii. To test the basics catalyst and modified catalyst produced in biodiesel production from Jatropha oil.

1.4 Problem Statement

In conventional industrial biodiesel processes, the methanol transesterification of vegetable oils is achieved using a homogeneous catalyst system operated in either batch or continuous mode. In most cases the catalyst is sodium hydroxide or sodium methylate. It is recovered after the transesterification reaction as sodium glycerate, sodium methylate and sodium soaps in the glycerol phase. An acidic neutralization step with, for example, aqueous hydrochloric acid is required to neutralize these salts. In that case glycerol is obtained as an aqueous solution containing sodium chloride. Depending on the process, the final glycerol purity is about 80% to 95% (Alok and Sandon., 2008). When sodium hydroxide is used as catalyst, side reactions forming sodium soaps generally occur (Ritesh *et al.*, 2005). This type of reaction is also observed when sodium methylate is employed and traces

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of water are present. The sodium soaps are soluble in the glycerol phase and must be isolated after neutralization by decantation as fatty acids. The loss of esters converted to fatty acids can reach as high as 1% of the biodiesel production.

Thus, this paper will present alternative solution to overcome these problems. The better way to reduce the amount of side product and to make the recovery of catalyst and glycerol easier is by transitioning to the heterogenous catalyst system. This paper also provide the improvement catalyst system by addition of hydrophobic characteristic in the heterogenous catalyst system to enhance the attraction of the substrates and finally, increase the catalytic activity and production of biodiesel

1.5 Benefit and significant of research

This paper gave several significant elements. Firstly, is environmental friendly since the heterogenous catalyst will reduce the side product in biodiesel production and thus, approaching the green technology. Secondly, is one of the ways to contribute in the society. As a future chemical engineer there is our responsibility to make our life better by generate alternative solution to the problem that occur in biodiesel industry. Lastly, is producing more biodiesel (large scale) and commercialize it with reasonable price since heterogenous, hydrophobic catalyst system will increase the biodiesel production and reduce the costing in the separation process.

CHAPTER 2

LITERATURE REVIEW

2.1 Catalysis

A catalyst is a substance that accelerates a reaction but undergoes no change at the end of the process. It works by lowering the activation energy of the reaction by providing an alternative path, thus avoiding the slow, rate –determining step of the uncatalyzed process. There are three types of catalyst namely, homogenous catalyst, heterogenous catalyzed and enzymes. A homogenous catalyst is a catalyst that exists in the same phase as the reaction mixture while heterogenous catalyst exist in a different phase from the reaction mixture. Enzyme on the other hand can be generalized as a homogenous biological catalyst. An enzymes catalyst usually composed of a special protein or nucleic acids that contain an active site (Misonn, 2008).

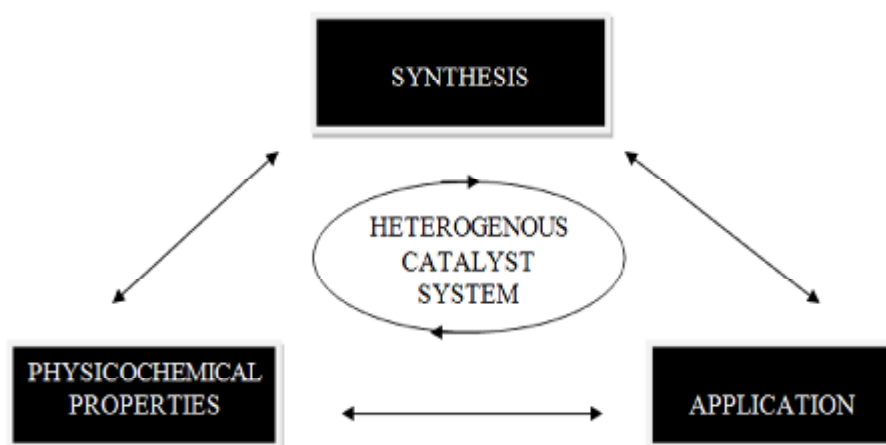


Figure 2.1: Cycle of the development of heterogenous catalyst (Misonn, 2008)

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Figure 2.1 shows the cycle of heterogenous catalyst system. The development of heterogenous catalyst may be regarded as an iterative optimization process, basically consisting of three steps, namely synthesis, physicochemical properties and application. The speed at which a progress occurs depends on the quality of the concepts or working hypothesis used to guide this process. Historically, research in catalysis is experimentally oriented, and attempts to make a general organization of information on catalysis would be more of correlation theory, although the concepts involved in such correlation rest on theoretical grounds. Nowadays, combination of physicochemical inspections of the catalyst characteristics with catalyst modeling and mechanistic simulations is the more effective approach towards understanding how the catalyst is prepared (synthesis), actually 'looks' (characterization) and performs (testing). The synthesis of catalyst is divided into three steps: preparation, reaction and isolation. The preparatory step of the catalyst is the most difficult step because many experimental parameters are involved (Misono, 2008). Heterogenous catalyst is among the most studied catalyst because it offer several advantages such as ease recovery and can be recycled. This type of catalyst contain actives site which correspond to the sides on the catalyst surface in which chemisorptions and reaction tend to occur. Generally, reactants must interact with the active sites and molecules involved in the catalytic process must undergo:

- i. Diffusion to the surface of the catalyst particle,
- ii. Diffusion through the pore network to the active sites,
- iii. Chemisorption process
- iv. Formation of activated complex
- iv. Conversion to the product while retaining its chemisorbed state,
- v. Desorption process from the active sites
- vi. Diffusion through the pore structure and,
- vii. Diffusion into the bulk fluid

Steps (ii) and (vii) are controlled by the molecular size and pore structure of the catalyst. Step (iii) until step (vi) depend on the chemical compatibility of reactant and product molecules with the active sites. Steps (i) and (viii) are the mass transfer step which depend on the reactant/product concentration, catalyst amount and

particle size. One of these steps will control the overall reaction rate and the slowest step from all the steps will be used for the reaction rate analysis (Misnon, 2008).

Catalyst has undergone major development from the fundamental knowledge perspective and also its application. Currently, the world catalyst market is about USD 9 billion with the total contribution of about 20 % of world GNP. In 2005, the worldwide market for catalyst exceeded US 25 billion and it is expected to increase further. The critical catalyst needs are in the environment, polymer, chemical and refining industries (Centi *et al.*, 2002).

2.1.1 Zirconia (ZrO_2)

Zirconium is found in earth crust and usually in the form of baddeleyite (zirconium oxide) and zircon ($ZrSiO_4$) (Salleh, 2002). Zirconia exhibits several stable crystalline structures which depend on temperature. ZrO_2 is monoclinic and stable at temperature below 1172 °C, tetragonal, at the temperature range of 1172-2347 °C, cubic, at above 2347 °C and rhombic at high pressure (Kuznetsov *et al.*, 2002). In the monoclinic phase, each zirconia atom has seven coordination number and Zr-O stretch range from 2.05 Å to 2.28 Å while in the tetragonal phase; each zirconium atom is attached to eight oxygen atoms in the same Zr-O distance and each oxygen atom has a coordination with four zirconium atoms in tetrahedron form (Salleh, 2002).

Zirconia has both acidic and basic properties in the surface which leads to high selectivity in catalysis and makes the catalyst more stable. Besides, it also shows an oxidation and reduction capability on its surface (Buang, 1999). Some of the physical properties that make it useful support under harsh condition are its high melting point (2370 °C), low thermal conductivity, high corrosion resistance (Zhu *et al.*, 2004), remarkable mechanical strength and unique properties of the surface (Kuznetsov *et al.*, 2002).

2.1.2 Tungsten (W)

Tungsten also known as wolfram, is a chemical element with the chemical symbol W and atomic number 74. A steel-gray metal, tungsten is found in several ores, including wolframite and scheelite. It is remarkable for its robust physical properties, especially the fact that it has the highest melting point of all the non-alloyed metals and the second highest of all the elements after carbon. Tungsten is often brittle (Martino, 2007) and hard to work in its raw state; however, if pure, it can be cut with a hacksaw (Ritesh *et al.*, 2005). The pure form is used mainly in electrical applications, but its many compounds and alloys are used in many applications, most notably in incandescent light bulb filaments, X-ray tubes (as both the filament and target), and superalloys. Tungsten is also the only metal from the third transition series that is known to occur in biomolecules, and is the heaviest element known to be used by living organisms.

2.1.3 Sulfate (SO₄)

The sulfate ion can act as a ligand attaching either by one oxygen (monodentate) or by two oxygens as either a chelate or a bridge (Amish *et al.*, 2008). An example is the neutral metal complex $\text{PtSO}_4(\text{P}(\text{C}_6\text{H}_5)_3)_2$ where the sulfate ion is acting as a bidentate ligand. The metal-oxygen bonds in sulfate complexes can have significant covalent character.

The sulfate ion is a polyatomic anion with the empirical formula SO_4^{2-} and a molecular mass of 96.06 daltons; it consists of a central sulfur atom surrounded by four equivalent oxygen atoms in a tetrahedral arrangement (Kaewta *et al.*, 2008).

2.1.4 Octadecyltrichlorosilane (OTS)

Surfactant is a chemical capable of being located or adsorbed at interfaces, or form colloidal aggregates at low molar concentration. It contain two major parts, polar head and non polar-tail. Octadecyltrichlorosilane is in the family of organofunctional silane or organosilane, one from surfactant class and widely used for surface modification.OTS has the advantage over other organic compounds that the silanes have potential for bonding via several mechanisms (Nur *et al.*, 2005). The chemical structure of OTS is shown in Figure 2.2.

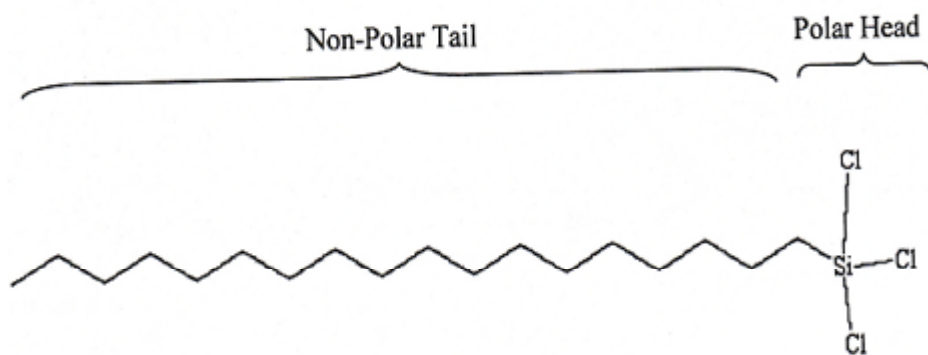


Figure 2.2: Chemical structure of OTS (Nur *et al.*, 2005)

The long alkyl chain is the part of non-polar tail meanwhile the trichlorosilane group makes up the polar head as depicted in Figure 2.2. The polar head component is located on the surface of zirconia support surface and the outer layer of modified zirconia will be hydrophobic due to the presence of non-polar tail (Nur *et al.*, 2005). Octadecyltrimethylsilane has a capability as catalyst in biodiesel production such as:

- The catalysts are hydrophobic (no H₂O adsorption at reaction temperatures)
- Eliminate water from deactivate active sites (W, SO₄²⁻)
- Anti-water, probably, due to their surface hydrophobicity
- Promote the attraction of the substrate (oil, methanol) to active site

2.15 Organofunctional silane Modification – A Hydrophobic Catalyst

The critical problem faced when ziolite or metal oxide is used in the oxidation in the liquid phase is hydrophobicity. This phenomenon hampers the diffusion of organic substrate onto the surface of catalyst. As a simple solution, one might transform the hydrophilic surface of the catalyst to hydrophobic by using a surfactant (Nur *et al.*, 2006). A surfactant is a chemical capable of being adsorbed in the interfaces or form colloidal aggregates in solution at low molar concentration. It contain two major parts, a polar head and a non polar tail.

Alkylsilane such as octadecyltrichlorosilane (OTS) are in the family of organofunctional silane or organosilane, widely used for surface modification an functionalization. OTS is preferred over other organic compounds since the silanes can form bonds via several mechanism (Nur *et al.*, 2006). Organosilane deposition has additional benefits over other preparation methods because of its fast preparation, stable finish, and applicability to a wide range of substrates.

The assembly or coating occur by hydrolysis reaction of free surface hydroxyl group on the support and the organosilanes. In case of complete formation of assembled monolayer using OTS, all chlorine species hydrolyze to form bonds with other hydroxyl groups as demonstrated in figure 2.3. OTS reacts with the substrate surface through only one hydroxyl group, while two other group produce uniform surface coverage through cross polymerization.

Figure 2.4 represents incomplete mechanism of OTS. In case of incomplete reaction, there are some unreacted chlorosilane groups remaining on the surface which might be hydrolyze later, especially by trace amount of water in the reaction vessel.

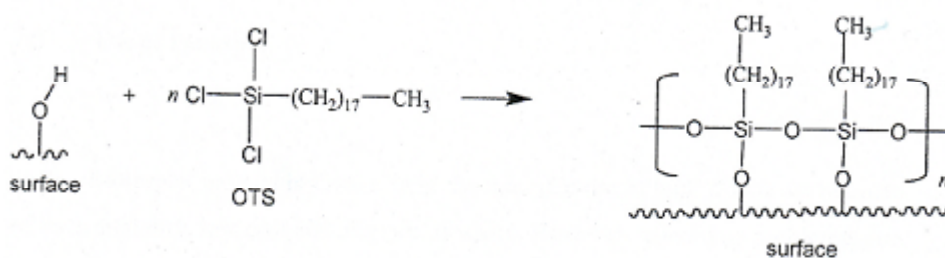


Figure 2.3: Mechanism of complete reaction of OTS on the support surface to form well ordered layer (Mison, 2008)

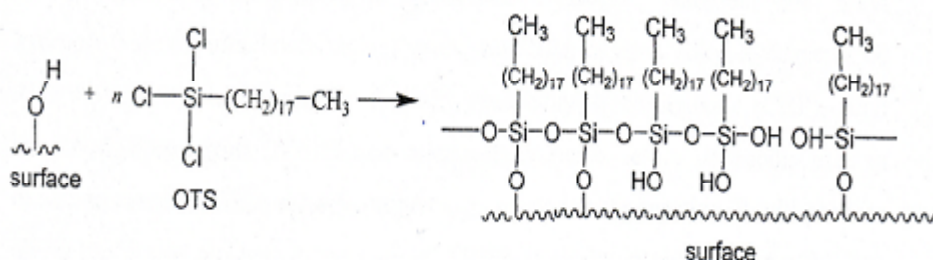
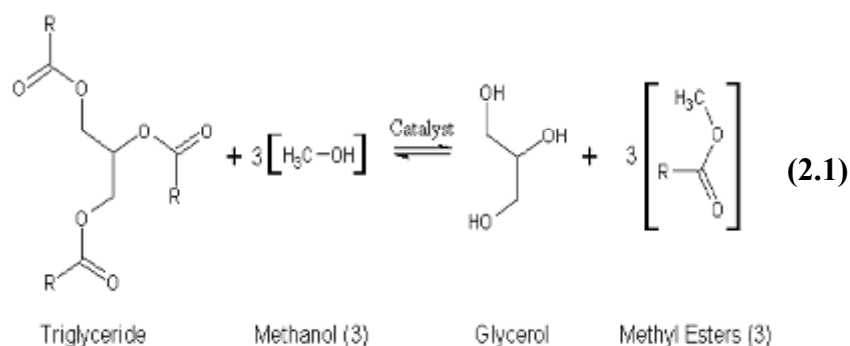


Figure 2.4: Mechanism of incomplete reaction of OTS on the support surface (Mison, 2008)

2.2 Biodiesel Production – General

At present, biodiesel is usually produced by reacting methanol and vegetable oil in a batch stirred tank reactor using a liquid alkaline catalyst. The catalyst cannot be recovered from the reactor and is instead neutralized and disposed of as a waste stream. Biodiesel has been produced by transesterification of triglyceride (vegetable oils) to methyl esters with methanol using sodium or potassium hydroxide dissolved in methanol as catalyst, as represented by the following equation 2.1 (Ritesh, 2005) :



Once the reaction is complete, two major products exist: glycerin and biodiesel. Each has a substantial amount of the excess methanol that was used in the reaction. The reacted mixture is sometimes neutralized at this step if needed. The glycerin phase is much more dense than biodiesel phase and the two can be gravity separated with glycerin simply drawn off the bottom of the settling vessel. In some cases, a centrifuge is used to separate the two materials faster (Amish *et al.*, 2008).

2.3 Jatropha Oil and Its Benefits

Jatropha oil is vegetable oil produced from the seeds of the *Jatropha curcas*, a plant that can grow in marginal lands and common lands. *Jatropha curcas* grows almost anywhere, even on gravelly, sandy and saline soils. It can also thrive on the poorest stony soil and grow in the crevices of rocks. When jatropha seeds are crushed, the resulting jatropha oil can be processed to produce a high-quality biodiesel that can be used in a standard diesel car, while the residue (press cake) can also be processed and used as biomass feedstock to power electricity plants or used as fertilizer (it contains nitrogen, phosphorous and potassium). The plant may yield more than four times as much fuel per hectare as soybean, and more than ten times that of maize (corn). A hectare of jatropha has been claimed to produce 1,892 litres of fuel. However, as it has not yet been domesticated or improved by plant breeders, yields are variable (Khurmi, 2006). The fatty acid composition of jatropha curcas oil is shown in Table 2.2.

Table 2.2: Fatty acid composition of *Jatropha curcas*, (Khurmi, 2006).

COMPOSITION	PERCENTAGE (%)
Palmitic acid (C16:0)	11.3
Stearic acid (C18:0)	17.0
Oleic acid (C18:1)	12.8
Linoleic acid (C18:2)	47.3
Arachidic acid (C20:1)	4.7
Arachidoleic acid (C20:1)	1.8
Behenic acid (C22:0)	0.6
Lignoceric acid C24:0	0.44

2.4 Characterization of Bulk and Surface Structure

Before the catalyst embarking onto the catalytic activity test, the surface modified of zirconia had to be characterized. The properties should comply well with their surface structure. Those particulate may be characterized via several characterization methods. The catalyst characterization is detailed in Section 3.2.

CHAPTER 3

METHODOLOGY

3.1 Chemical and Reagents

Zirconium hydroxide ($Zr(OH)_4$), Tungstated zirconia (W/ZrO_2), and sulfated zirconia (SO_4/ZrO_2) used as raw material are supplied by Mel Chemical. Surface modification was carried out by using n- octadecyltrichlorosilane (OTS) as a surfactant, and toluene (99.5 v/v) as a solvent. Both of the chemicals were purchased from Sigma-Aldrich Chemical. For the transesterification process, jatropha oil were supplied by Faculty of Science and Industry Lab and methanol (99.5% purity) were obtained from Sigma-Aldrich Chemical.

3.2 Catalyst Preparation for Biodiesel Production

3.2.1 OTS - ZrO_2

$Zr(OH)_4$ was calcined at condition at 550 °C for 2 hour in carbolite furnace to produce ZrO_2 . The temperature was set with 2°C temperature increment per minute. The second step was by alkysilation of OTS, whereas 10ml of toluene containing 250µmole of OTS were mixed with 3 gram of ZrO_2 powder in container and shaken for 20 minute. The processes continued with centrifugation at 7000rpm for 20 minute. After that, the catalyst produced was heat at 100 ° C at overnight. A

schematic representation of the step involved for the preparation of OTS-ZrO₂ is illustrated in Figure 3.1.

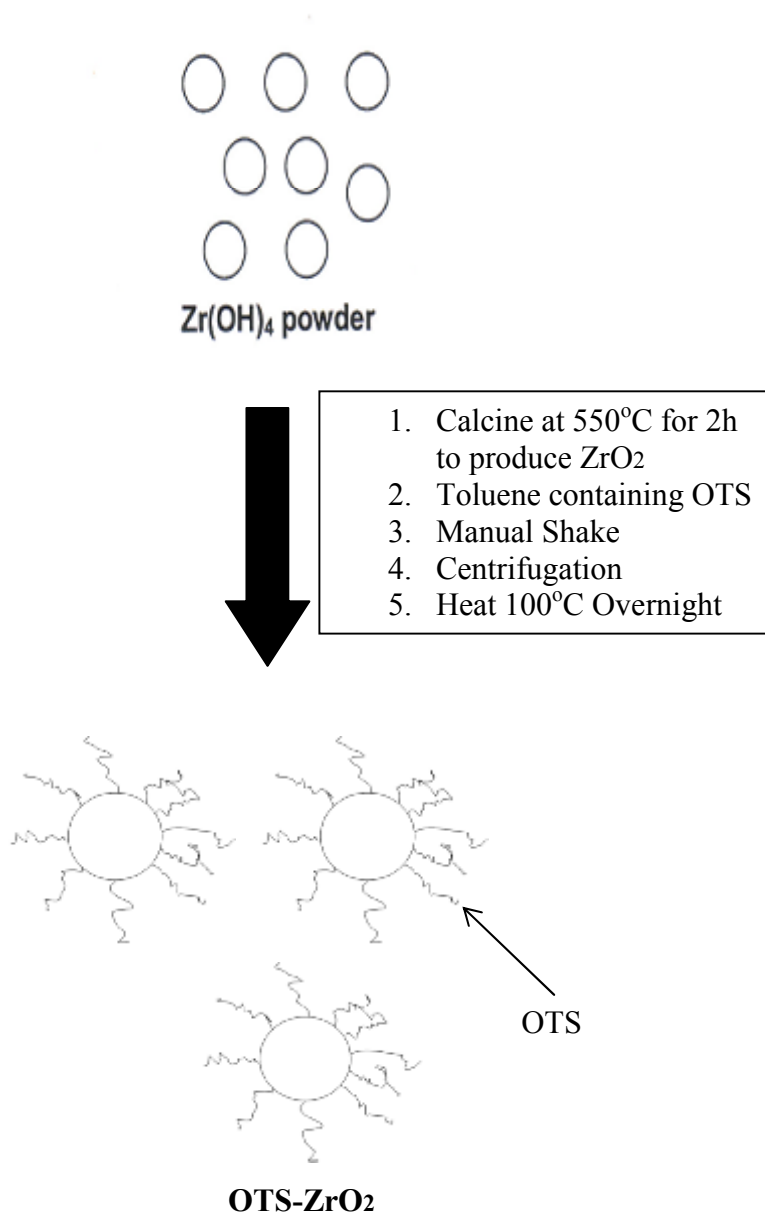


Figure 3.1: A Schematic representation for the preparation of OTS-ZrO₂