

**PRETREATMENT AND SEPARATION OF CRUDE GLYCEROL FROM
GLYCEROL RESIDUE USING MEMBRANE REACTOR ON EFFECT OF TRANS
MEMBRANE PRESSURE AND CROSS FLOW VELOCITY**

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ABSTRACT

The objective of this research is to study the effect of trans membrane pressure and cross flow velocity on flux decline during crude glycerol recovery. The crude glycerol is just the waste from oleochemical process and biodiesel industry that basically wasted. The glycerol can be recovered and potentially have many uses of it by refining from transesterification process from biodiesel industry. The glycerol residue was undergoing the pre treatment process using high concentration of acid to convert to crude glycerol then followed by filtration process to remove charred substances. The neutralization process is then used to neutralize the aqueous glycerol. The 22x2 inches cylinder ceramic membrane had been used with 0.9 micron of pore size. The membrane filter had its own limitation on trans membrane pressure, time of filtrate and cross flow velocity. The result found the increasing of TMP will enhance the higher flux decline and faster to steady state. While increasing the CFV will increase the flux decline and late become steady state. The optimum CFV was 0.75 m/s with 72.22 % of flux decline. The optimum TMP was 2.5 bars with 63.88% of flux decline. The CFV give higher effluence in flux decline compared to TMP.

ABSTRAK

Objektif kajian ini adalah untuk mengkaji kesan terhadap tekanan melintas membran dan kelajuan arus lintang. ke atas kejatuhan fluk semasa rawatan gliserol mentah. Gliserol mentah adalah hasil buangan dari proses kimia oleo dan industri biodiesel. Gliserol boleh dirawat dan berpotensi dalam pelbagai guna daripada penapisan gliserol semasa transterifikasi dari industri biodiesel. Gliserol lebihan telah melalui proses pra rawatan menggunakan acid berkepekatan tinggi untuk ditukarkan kepada gliserol mentah dan diikuti dengan proses penapisan untuk membuang sisa pepejal. Kemudian, proses neutralisasi digunakan untuk meneutralkan cecair gliserol itu. Seramik membrane silinder 22x2 inchi telah digunakan dengan saiz rongga 0.9 micrometer. Penapis membrane mempunyai kelemahan tersendiri semasa menghadapi tekanan melintas membrane, masa penapisan dan kelajuan arus lintang. Keputusan telah membuktikan terhadap kenaikan tekanan melintas membran akan menggalak lebih tinggi kejatuhan di dalam fluk dan cepat untuk mencapai ke arah keadaan stabil. Kenaikan kelajuan arus lintang juga akan meningkatkan kejatuhan fluk tetapi lewat mencapai keadaan stabil. Nilai optima kelajuan arus lintang ialah 0.75 m/s mencatatkan 72.22% kejatuhan. Nilai optima bagi tekanan melintas membrane pula ialah 2.5 bar dengan 63.88% kejatuhan fluk. Oleh itu, kelajuan arus melintang memberikan lebih pengaruh daripada tekanan melintas membran.

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LIST OF SYMBOLS/ABBREVIATIONS

ANOVA	-	Analysis of variance
CCD	-	Central composite design
g	-	Gram
g/L	-	Gram per litre
hr	-	Hour
L	-	Litre
M	-	Molar
mg	-	Miligram
min	-	Minutes
ml	-	Mililitre
mM	-	Milimolar
m ²		meter square
OFAT	-	One factor at time method
RSM	-	Response surface methodology
rpm	-	Round per minute
T	-	Temperature
T		Time
V		Volume
°C	-	Degree Celsius
%	-	Percentage

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Glycerol is a renewable feedstock mostly derived from natural sources such as vegetable oils and animal fats, is produced as a byproduct in the growing biodiesel production and oleo chemical industry (Zhanyou *et. al.*, 2007). Glycerol wastes are largely waste in Malaysia and usually produce from byproduct of the oleo chemical and biodiesel industries are potentially high in used.

Many projects are now under way to convert glycerol into various marketable and value-added products such as the reactions for direct transformation of vegetable oils into methyl esters and glycerol have been known for more than a century. The reactions of interest today, mainly those producing methyl esters from rapeseed, soybean and sunflower oils, have been studied and optimized in order to manufacture the high quality diesel fuel known as biodiesel (Bournay L., 2005).

Glycerol residue is produced from soap which is obtained by saponification, triglycerides are converted by alkaline hydroxides into salts of fatty acid and glycerol. Glycerol contains 20.2% glycerol, 6.6% fatty acids and 64.3% salt (Yong, 2001). Glyceric acid (GA) is one of the promising glycerol derivatives obtained by the metal-catalytic oxidation of the primary hydroxyl groups of glycerol and has the potential to be a building block for chemical compounds used in the pharmaceutical and cosmetics industries (Hiroshi Habe, 2009).

In crude glycerol recovery there are many processes involved such as acidification, decantation, neutralization, filtration and separation in order to recover the crude glycerol. On separation process many kind of methods can be used one of them is membrane filtration. In industries, membrane is used widely and very synonym in separation process.

1.2 Problem statement

The crude glycerol is just the waste from oleo chemical process and biodiesel industry that basically wasted. The glycerol can be recovered and potentially have many used of it by refining from transesterification process from biodiesel industry. Phrase ‘waste to wealth’ is the main reason the glycerol needed to be recovered. Besides that, while we are focusing on the economic subject indirectly the environment can be safe. The concern of environmental issues from industries is one aspect that needed to be focused because of glycerol residue that dumped into landfill can affect the environmental pollution such as water pollution commonly. In separation process, the usage of membrane has its disadvantage itself. The membrane is easily fouling due to adsorption, concentration, polarization, and reversible and irreversible fouling (Choi *et. al.*, 2005).

1.3 Research Objective

The objective of this study is to study the effect of trans membrane pressure and cross flow velocity in flux during crude glycerol recovery.

1.4 Research Scope

In order to achieve the objective, the following scopes are identified:

- i. Effect of trans membrane pressure at range from 0 to 2bar for flux decline on glycerol recovered.

- ii. Effect of cross flow velocity at speed from 0.15m/s to 0.75m/s for flux decline of glycerol recovered.
- iii. Time contact of membrane fouling for TMP and CFV.
- iv. Study on the pretreatment of glycerol residue.

1.5 Rational and significant

The recovery of waste glycerol can convert to another product that more valuable. The oleochemical industry can maximize their production by recovered the crude glycerol in order to increase the profit company. Besides that, the environmental can be safe by recovered the crude glycerol that abundantly wasted.

CHAPTER 2

LITERATURE REVIEW

2.1 Sources of glycerol

Glycerol produced from many sources such as vegetable oils or animal fats, lignocellulosic biomass and palm kernel oil methyl esters (Table 2.1). The different sources introduce to different of process to produce glycerol. Moreover the composition of glycerol is differing between the sources.

2.1.1 Vegetable oils and animal fats.

Glycerol is a trivalent alcohol widely used in the pharmaceutical, food, cosmetic, and chemical industries. It is produced from soaps which are obtained by saponification of triglycerides from vegetable oils or animal fats. During their saponification, triglycerides are converted by alkaline hydroxides into salts of fatty acids (soaps) and glycerol (Zajic *et. al.*, 1988). The raw glycerol phase with different compositions potassium hydroxide pure (83 wt% purity), methanol (technical grade), sulphuric acid (96 wt %), hydrochloric acid (36 wt. %), phosphoric acid p.a. (85 wt. %), and acetic acid (Zajic *et. al.*, 1988)

2.1.2 Lignocellulosic biomass

Renewable lignocellulosic biomass, as a promising alternative for the limited crude oil, can be utilized to produce biofuels and biochemicals. To make these bio-based products more cost-competitive with fossil-derived conventional commodities, pretreatment and enzymatic hydrolysis of carbohydrates have become two key processes involved in the bioconversion of lignocellulosic biomass into inexpensive reducing sugars. Glycerol, as a high-boiling-point organic solvent, is presently the main byproduct of oleochemicals industry, as high as 10% of the total biodiesel production. (Nguyen and Saddler, 1991)

2.1.3 Palm kernel oil methyl esters

In the production of palm kernel oil methyl esters, large amounts of glycerol residue are produced from glycerol refining about 1 tons/day by a particular plant alone. With the demand for methyl esters and fatty alcohols expected to increase greatly, the amount of glycerol residue generated will also rise. As most of this residue is dumped in landfills, it would be advantageous if its valuable components can be recovered for use (Ooi *et. al* 2001). Glycerol residue contains 20.2% glycerol, 6.6% fatty acids (as soap) and 64.3% salt (Yong *et al.*, 2001)

Table 2.1: The different sources of glycerol residue

Sources	Authors	Title journals
Vegetable oils or animal fats	Martin Hajek, 2010)	Treatment of glycerol phase formed by biodiesel production
Lignocellulosic biomass	Nguyen and Saddler, 1991	Enhanced enzymatic hydrolysis of wheat straw by aqueous glycerol pretreatment
Palm kernel oil methyl esters	Ooi <i>et. al</i> , 2001	Crude glycerol recovery from glycerol residue waste from a palm kernel oil methyl ester plant

Shochu yeast	Kajiwara et. al, 2000	Enhanced Glycerol Production in Shochu Yeast by Heat-Shock Treatment is Due to Prolonged Transcription of GPD1
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2.2 Biodiesel

The focus of many biodiesel producers is currently to get production facilities up and running as quickly as possible to take advantage of current demand. Biodiesel producers around the world are in a variety of stages of development: in early design stages, final equipment specification stages, or currently producing biodiesel. In United States, the biodiesel production capacity has increased drastically from less than 100 million gallons/year in 2005 to an expected 580 million gallons/year in 2007 (National Biodiesel Board).

During the biodiesel production process, oil/fats are mixed with methyl alcohol and alkaline catalyst to produce esters of free fatty acids, with glycerol as a primary by-product (National Biodiesel Board). Biodiesel fuels have some advantages over petroleum based diesel fuels. Biodiesel fuels are biodegradable, non-toxic and produce less particles, smoke and carbon monoxide. Besides that, with growing environmental awareness about the negative implications brought by excessive usage of fossil fuels, the race for finding alternative energy as their substitutions is getting heated up. The Figure 2.1 had shown that both petroleum and natural gas energy are most contributed in Malaysia's primary energy production.

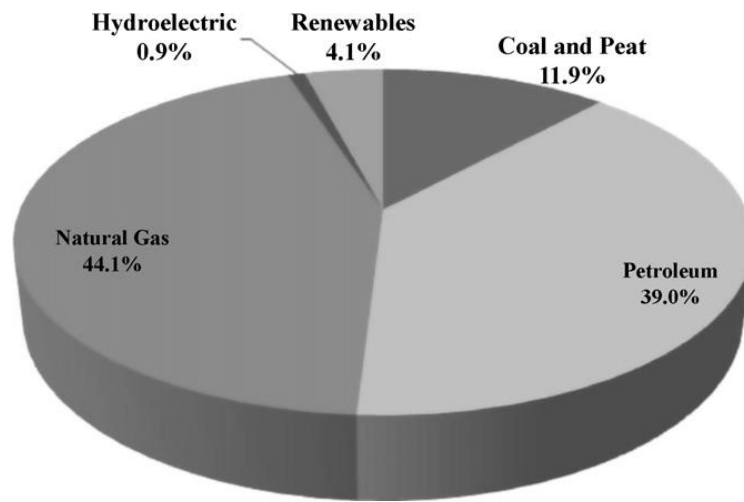


Figure 2.1: Malaysia primary energy production in 2006 by source (Lim *et. al.*, 2010).

Even though the market share of liquid fuels like petroleum will decline eventually due to high oil price in the future, natural gas and coal will continue to remain as an important source of energy supply due to the former being more efficient and less carbon intensive than other fossil fuels while the latter being one of the cheapest and most abundant source of energy available (Energy Information Administration 2009). The Figure 2.2 had shown step overall of biodiesel production and glycerol as its by-product.

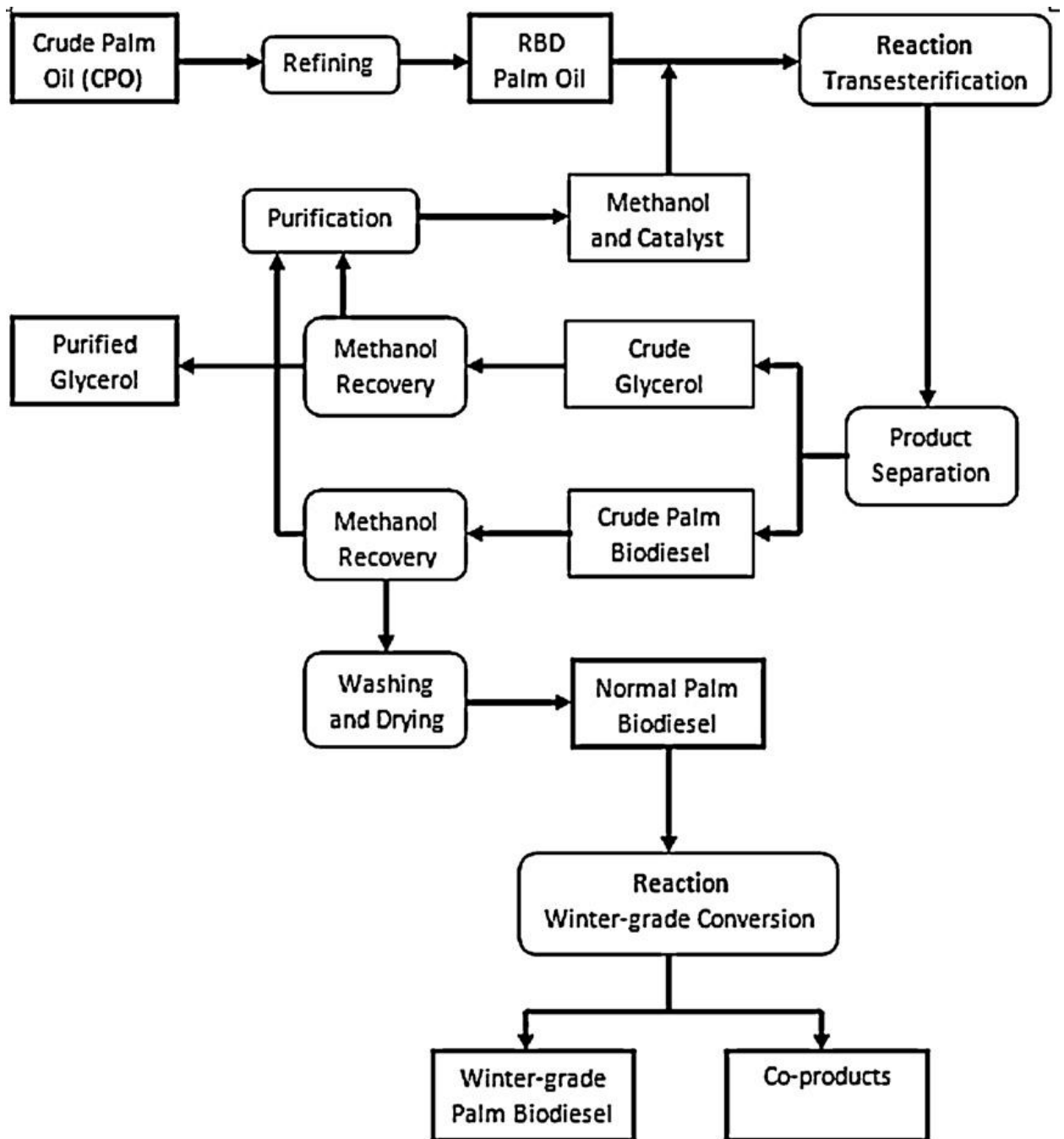


Figure 2.2: Schematic diagram for palm biodiesel processing by MPOB.

Biodiesel is produced by chemically reacting between fat and oil with alcohol, in present of catalyst. The product of the reaction is a mixture of methyl esters, which is known as biodiesel. The process is known as transesterification.

2.3 Crude Glycerol from Biodiesel Industry

The waste glycerol is the by-product from biodiesel manufacturing from vegetable oil and animal fat has a good potential as alternative diesel fuel (Zhanyou et. al., 2007). The biodiesel has attracted increasing attention in recent years such as in United States has been experiencing in high energy prices and exposing the desire to use the greener fuels to become energy independent. Biodiesel can also be known as fatty acid methyl ester (FAME), is produced from transesterification of vegetable oils or animal fats with the addition of methanol as shown in Figure 2.3.

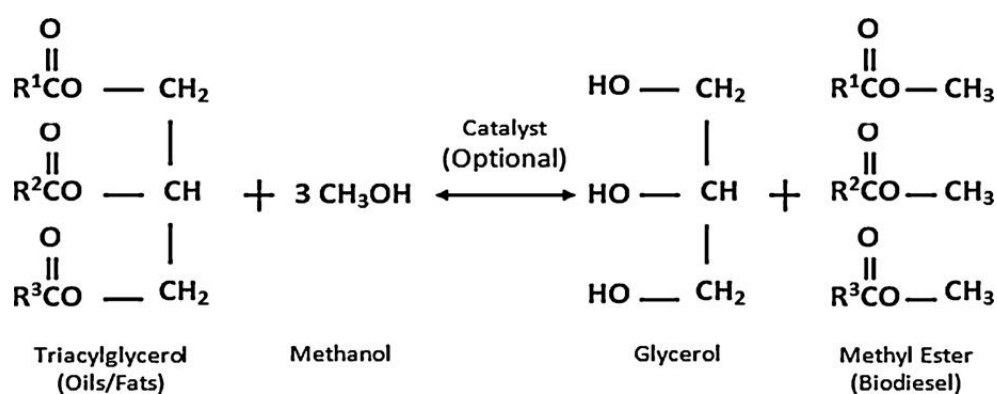


Figure 2.3: General equation for transesterification of triacylglycerol.

Currently, total approved installed capacity of biodiesel production in Malaysia equals to almost 92% of the world biodiesel production output in 2008 (Lim *et. al.*, 2010). Production of biodiesel is also a proven technology with established commercialization activities. The huge potential of biodiesel coupled with the abundance of palm oil which is one of the most cost-effective feedstock for biodiesel is responsible for the pledging of Malaysia to become the leading producer of high quality biodiesel in the region.

2.4 Properties of glycerol

Glycerol, a renewable feedstock mostly derived from natural sources such as vegetable oils and animal fats, is produced as a byproduct in the growing biodiesel production and oleo chemical industry. Many projects are now under way to convert glycerol into various marketable and value-added products. Glycerol also called glycerin or glycerine. It is a colorless, odorless, viscous liquid that is widely used in pharmaceutical formulations.

Glycerol has three hydrophilic hydroxyl groups that are responsible for its solubility in water and its hygroscopic nature. The glycerol substructure is a central component of many lipids and it is sweet-tasting and low of toxicity. Glycerol, as a high-boiling-point organic solvent, is presently the main byproduct of oleo chemicals industry, as high as 10% of the total biodiesel production. Glycerol has a specific gravity of 1.261, melting point of 18.2°C and the boiling point is 290°C under normal atmospheric pressure, accompanied by decomposition. Glycerol may form crystals which melt at low temperature, 17.9°C.

Table 2.2: Physicochemical Properties of Glycerol at 20°C

No	Item	Description
1	Chemical Formula	$C_3H_4(OH)_3$
2	Molecular mass	92.09382 g/mol
3	Density	1.261 g/cm
4	Viscosity	1.5 Pa.s
5	Melting Point	18.2°C
6	Boiling Point	290°C
7	Food Energy	4.32 kcal/g
8	Flash Point	160°C
9	Surface Tension	64 mN/m

10	Temperature coefficient	-0.0598 mN/mK
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2.5 Application of Glycerol

Glycerol structure is 1,2,3-propanetriol, is a simple alcohol with many uses in the cosmetic, paint, automotive, food, tobacco, pharmaceutical, pulp and paper, leather and textile industries. Glycerol has also been considered as a feedstock for new industrial fermentations in the future. For example, glycerol can be fermented to 1, 3 propanediol (Biebl *et al.*, 1998), which is used for the chemical synthesis of poly (trimethylene terephthalate).

Another use of glycerol is can apply to draw water from its surroundings and the heat produced by the absorption makes it feel warm. The glycerol is added to adhesives and glues to keep them from drying rapidly. It is also acts as a solvent, sweetener, and preservative in food processing, because it is nontoxic. Glycerol is also used in alkyd resin manufacture to impart flexibility.

The glycerol has many uses in personal care such as emollient, humectants, solvent and lubricant. The enormous varieties of products such as toothpastes are estimated to make up almost one-third of the personal care in market product from glycerol.

The glycerol usage is also widely used in medical and pharmaceutical like as means of improving smoothness, providing lubrication and as humectants which is a hygroscopic substance for preparation moist. Glycerol helps to maintain texture and add humetancy that controls water activity and prolongs shelf life in a host of applications. It is also widely used as a laxative and based on the same induced hyper osmotic effect, in cough syrups (elixirs) and expectorants.

Table 2.3: Current applications of glycerol (Wang *et. al.*, 2001).

Use field	Use in %			
	USA (160000 tons/year)	Europe (190000 tons/year)	Japan (50000 tons/year)	China (80000 tons/year)
Drugs	39.5	23.1	34.0	5.2
Tobacco	15.8	2.5	5.3	7.3
Glycerintriacetate	No Data	14.4	No Data	No Data
Food	14.5	5.6	No Data	No Data
Polyether alcohol	10.5	13.1	11.6	5.2
Paints	9.2	13.1	19.5	49.0
Cellophane	2.0	4.4	3.8	1.5
Dynamite	0.6	3.1	1.9	3.1
Toothpaste	No Data	No Data	No Data	16.0
Cosmetics	No Data	No Data	No Data	6.3
Miscellaneous	7.9	20.6	23.9	7.2

2.6 Glycerol waste composition

During the biodiesel production process the impure crude glycerol produced that has a little of economic value. During the pretreatment of glycerol, the methanol is used to drive the chemical transesterification and not all the methanol recovered. Therefore the glycerol present in glycerol because of the excess of methanol. In the production of palm kernel oil methyl esters, large amounts of glycerol residue are produced from glycerol refining about 1 ton per day (Ooi, 2001) by a particular plant alone. With the demand for methyl esters and fatty alcohols expected to increase greatly, the amount of glycerol residue generated will also rise.

As most of this residue is dumped in landfills, it would be advantageous if its valuable components can be recovered for use. The free fatty acids present in the initial feedstock can react with the base to form soaps that are soluble in the glycerol layer. The crude glycerol also contains a variety of minerals such as calcium, magnesium, phosphorous and sulfur (Thompson *et. al.*, 2006).

Glycerol makes up anywhere from 65% to 85% (w/w) of the glycerol streams (Gonzalez *et al.*, 2005). The remaining weight in the crude glycerol streams is mainly methanol and soaps (Thompson *et al.*, 2006). The different of purity values can attributed in different techniques and methods in order to purify the glycerol that usually used by the biodiesel producers.

2.7 Pre treatment process of glycerol residue

The pre treatment is the first step in order to convert the glycerol residue into crude glycerol. It was found that when crude glycerol was directly mixed with artificial seawater from the oleo chemical refinery then the soaps will precipitated from the liquid which later proved detrimental to cell growth (Zhanyou Chi, 2007). In order to eliminate the cell growth the pre treatment is used .The pre treatment is needed such as in oleo chemical industries, the glycerol residue treated before transfer it to the waste storage. The glycerol residue is needed to undergo pre treatment process as the first step in the purification of glycerol.

In the pre treatment process, the crude glycerol is dilute in distilled water in order to reduce the viscosity of glycerol (Zhanyou Chi, 2007). Then the pretreatment is undergoing with acidification to create the dual layer which are crude glycerol and fatty acid layer. The high concentration of sulphuric acid is used to acidify the mixture and skimmed off the fatty acid to the top layer. Then the process is allowed to settle for 30 to 45 minutes (Hazimah *et. al.*, 2002).

The fatty acid layer which is at the top layer contained a tar-like solid or paste (Hazimah *et. al.*, 2002). The fatty acid and other solid particles then are removed by filtration process to further remover any solid materials. The NaOH is used in order to neutralize the mixture to pH 7 in 50% concentrations NaOH (Hazimah *et. al.*, 2002). The NaOH neutralized the mixture will against the H₂SO₄ because of the salt crystallizing out was removed by decanting. The H₂SO₄ and NaOH were chosen as the reagents for pretreatment because of the sodium sulphate (Na₂SO₄) that form was less soluble in the aqueous solution of neutralized crude glycerol with NaCl and would easy for recovery by subsequent

evaporation and decanting (Ooi *et. al.*, 2001). The other pretreatments that had been done had shown in Table 2.4. There are many types of process and methods that separating the crude glycerol. One of them is separating by using membrane.

Table 2.4: The different of pretreatment process

Descriptions	References
Glycerol was diluted in methanol. Acidified with concentrated phosphoric acid	Martin Hajek <i>et. al.</i> , 2010
Glycerol was diluted in distilled water, acidified with sulphuric acid and neutralize with NaOH.	Ooi <i>et. al.</i> , 2001
Glycerol was mixed with distilled water at a ratio of 1:4, pH adjusted to around 6.5 with hydrochloric acid	Chi <i>et. al</i> 2007

The membrane reduce the chemical oxygen demand (COD), biochemical oxygen demand (BOD), oil and grease (O& G) and Suspended solids (SS) efficiently shown in Figure 2.4 about 85% to 99.9% remove. Almost overall SS were eliminated using the membrane meaning that the membrane is the best in removing the SS.