

## REMOVAL OF RESIDUAL OIL FROM PALM OIL MILL EFFLUENT USING SOLVENT EXTRACTION METHOD

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**Abstract.** Pollution from residual oil content in the wastewater is one of the serious environmental problems. Due to the rapid development of the palm oil industry in Malaysia, a large volume of palm oil mill effluent (POME) has been produced at the same time. Removal of residual oil from POME was carried out using solvent extraction process. The effect of different solvents, extraction time, solvent/feed ratio and mixing rate on extraction efficiency were investigated throughout this work. *N*-pentane, *n*-hexane and *n*-heptane were used as solvents. The percentage of oil extracted at the optimum conditions of 10 min extraction time, 1:1 solvent: feed ratio, 200 rpm using *n*-pentane, *n*-hexane and *n*-heptane were found to be 58, 63 and 65, respectively for a single stage extraction. Multistage extraction has shown that the % of oil extracted increased significantly to more than 97% of the residual oil in the POME.

*Key words:* Extraction, mixing rate, mixing time, solvent, POME

**Abstrak.** Pencemaran akibat daripada minyak buangan yang terkandung dalam air sisa merupakan salah satu masalah persekitaran yang amat serius. Disebabkan oleh perkembangan industri kelapa sawit yang amat pesat di Malaysia, isipadu air sisa dari kilang kelapa sawit (POME) yang banyak juga dihasilkan pada masa yang sama. Pengekstrakan minyak buangan dari POME telah dijalankan menggunakan proses pengekstrakan pelarut. Dalam kajian ini, kesan masa pengekstrakan, nisbah pelarut/suapan dan kadar pencampuran pengekstrakan menggunakan beberapa jenis pelarut telah dijalankan. *N*-pentana, *n*-heksana dan *n*-heptana telah digunakan sebagai pelarut. Peratus minyak yang diekstrakkan pada keadaan-keadaan optima ujikaji menggunakan *n*-pentana, *n*-heksana dan *n*-heptana diperolehi masing-masing sebanyak 58, 63 dan 65 peratus. Pengekstrakan berbagai peringkat juga telah dijalankan. Lebih daripada 97% minyak buangan yang terkandung dalam POME telah berjaya diekstrakkan pada peringkat keempat pengekstrakan menggunakan pelarut tersebut.

*Kata kunci:* Pengekstrakan, kadar pencampuran, masa pencampuran, pelarut, POME

### 1.0 INTRODUCTION

Over the last three decades, the palm oil industry has grown to become a very important agriculture-based industry in Malaysia and a large volume of palm oil mill effluent (POME) is produced at the same time. The POME must be treated to an acceptable level before it is discharged to the environment or it will cause serious and

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adverse problems. Oil can kill quickly by coating marine and freshwater life, interfering with gas exchanges necessary for life such as oxygen gas. An accumulation of oil can cause conditions to become anaerobic. It has been found that 0.1 ppm of oil in water can upset the biological cycle in rivers.

About five to seven tonnes of water are required in palm oil milling to produce one tonne of palm oil. About half of the water used ends up as palm oil mill effluent (POME). POME is a mixture of sterilizer condensate, separator sludge and hydrocyclone wastewater. Fresh POME is a colloidal suspension of 95–96% water, 0.6–0.7% oil and 4–5% suspended solids which are mainly debris from palm fruit mesocarp [1].

With the rapid expansion of the palm oil industry coupled with increased environmental awareness, the industry has had to treat its effluent to an acceptable level before discharge. Realizing the potential pollution that can be caused by the industry, the Government has enacted the Environment Quality Regulations for the palm oil industry in 1978. Table 1 summarizes parameter limits for watercourse discharge of POME [1].

**Table 1** Parameter limits for water course discharge of POME [1]

*Biochemical Oxygen Demand (BOD) (mg/L)	50
Suspended Solids (mg/L)	400
Oil and Grease (mg/L)	50
Ammoniacal Nitrogen (mg/L)	150
Total Nitrogen (mg/L)	200
pH	5-9

\* BOD – sample incubated for 3 days at 30°C

Treatment of palm oil mill effluent was subjected to many studies [2-6]. Biodegradation of palm oil mill effluent (POME) was also carried out under anaerobic conditions to environmentally acceptable products [6]. Studies were carried out in three experimental set-up such as; (i) a single stage anaerobic ponding system, (ii) a single stage anaerobic tank digester with a certain degree of mixing, (iii) recycled spent POME sludge in a single stage anaerobic tank. Operational variables such as optimum pH, the COD or BOD removal efficiencies and the overall usefulness of the digester modes to local communities and farmers are discussed.

The Membrane Anaerobic System (MAS) was used to treat industrial wastewater from a palm oil mill [7]. A clear final effluent was produced but membrane flux rate deterioration was observed due to membrane fouling. Membrane fouling could be minimized with faster crossflow feed velocities and regular membrane flushing. The

membrane fouling and polarization at the membrane surface played a significant role in the formation of the strongly attached cake layer limiting membrane permeability.

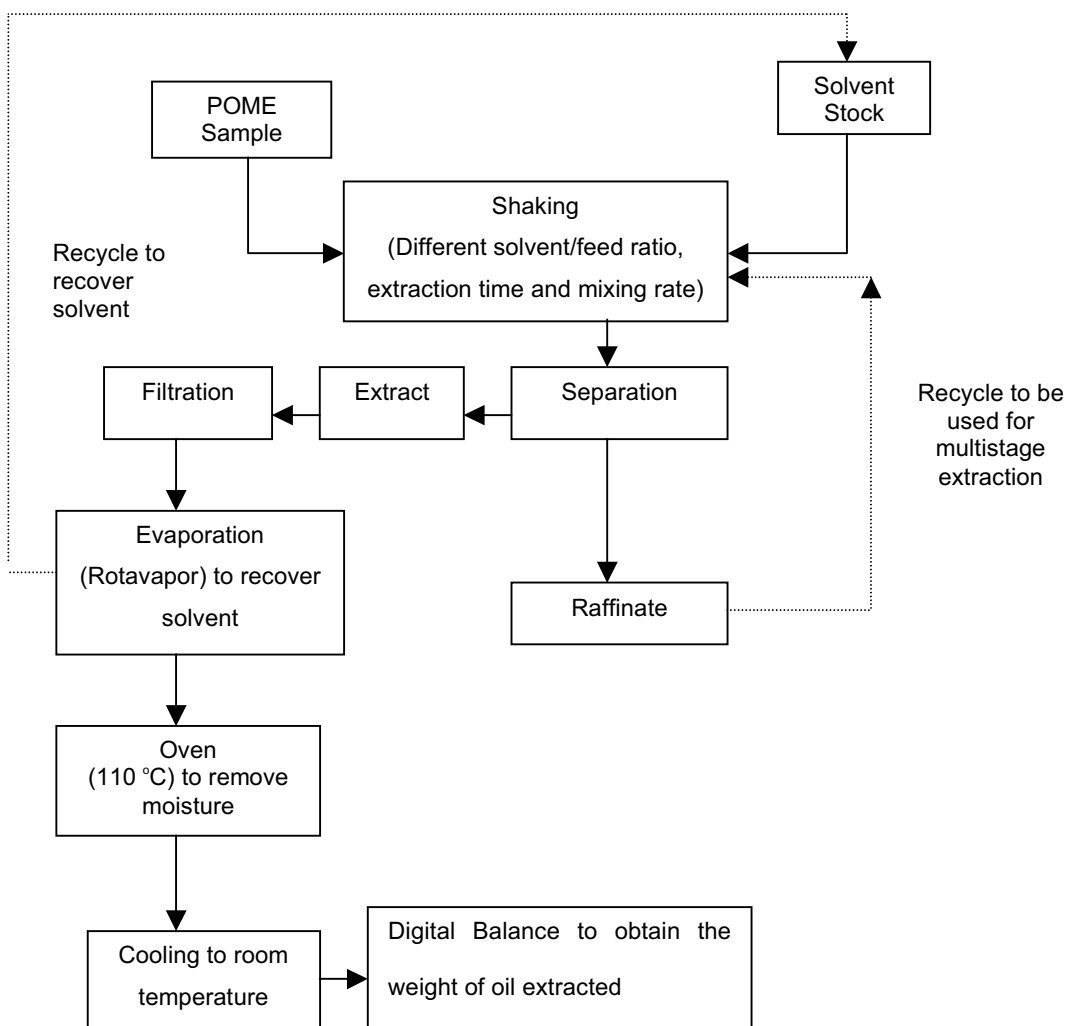
Chin et al [8] investigated the palm oil mill effluent treatment efficiency using a pond system consisting of 8 ponds in series treating 600 m<sup>3</sup>/day of POME. The pond system has been in operation since the mid 1980's. Effluent COD was 1,725 mg/L, BOD 610 mg/L, ammonia-N 115 mg/L, nitrate nitrogen 5 mg/L and phosphate 60 mg/L. The effluent was not able to meet the discharge standard of 50 mg/L BOD.

A laboratory-scale anaerobic filter (AF) and a fluidized-bed reactor (FBR) were compared for their ability to treat palm oil mill effluent (POME) [9]. The reactors were operated at mesophilic temperature (35°C). Reactor performance was assessed by measuring chemical oxygen demand (COD), volatile fatty acids (VFA), gas composition and gas production. COD removals higher than 90% were reached in both reactors at 6h residence time, equivalent to a loading of 10 g COD/L day. At higher loadings the FBR gave a better performance.

This paper is concerned with a study of removal of residual oil from POME using solvent extraction method. The effect of different solvents, extraction time, solvent/feed ratio, and mixing rate on the extraction efficiency were investigated through this work. Multistage extraction was also carried out in this study.

## 2.0 EXPERIMENTAL PROCEDURES

The sample of POME was obtained from Felcra Nasaruddin Sdn. Bhd., Perak. The three solvents used were *n*-hexane (> 99%, Merck), *n*-pentane (> 99%, Merck) and *n*-heptane (> 99%, Merck). Figure 1 shows a schematic diagram for the sequence of experimental steps. To study the effect of solvent/feed ratio on % of extraction, each of the three conical flasks was filled with 100 ml of raw POME after stirring manually. A 25 ml of each of *n*-pentane, *n*-hexane and *n*-heptane solvent was added into the conical flasks, respectively and shook by using a shaker at 200 rpm for 10 minutes. The two phases were then separated in a separator funnel. The extract was filtered by a filter paper and filled into a conical flask for evaporation. The solvent was recovered by using a rotavapor. The temperature was set slightly higher than boiling point of the solvent. After solvent recovery, the conical flask containing extracted oil was placed into an oven at temperature ~110°C for about 30 minutes for further drying of any traces of solvent or moisture residue. After drying, the conical flask was cooled to room temperature and weighted the extracted oil by using a four digits electronic balance. The procedures above were repeated with 50, 75 and 100 ml of each solvent. To determine the initial oil content in the raw POME for this experiment, raffinate was re-extracted by following the steps above with 100 ml of *n*-heptane and 30 minutes extraction time until the oil was completely extracted. Then the amount of oil extracted was found using the digital balance.



**Figure 1** Schematic diagram for the sequence of experimental steps

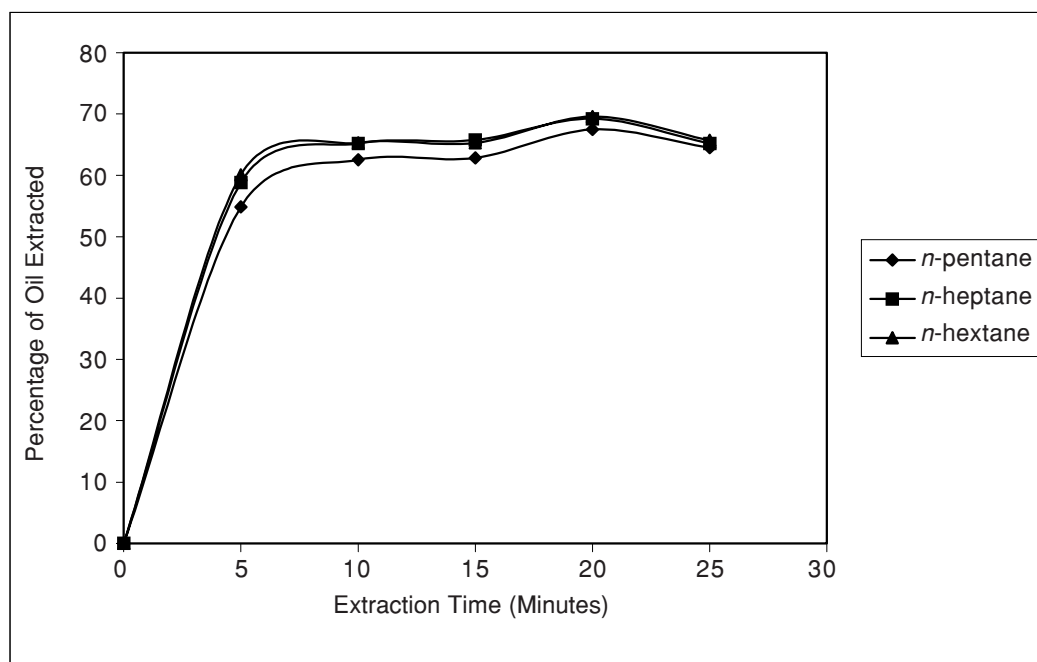
To study the effect of mixing rates, the procedure was same as above but 100 ml each of the solvents were used. These experiments were conducted at mixing rates of 100, 150 and 200 rpm.

In multistage extraction, the raffinate from the first extraction batch was reextracted four times by following the steps above and the oil extracted in each stage was recorded.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Effect of extraction time on percent of extraction

Figure 2 shows that the % extraction was slightly increased with the increasing of extraction time and almost constant at extraction time between 10–25 minutes. At this

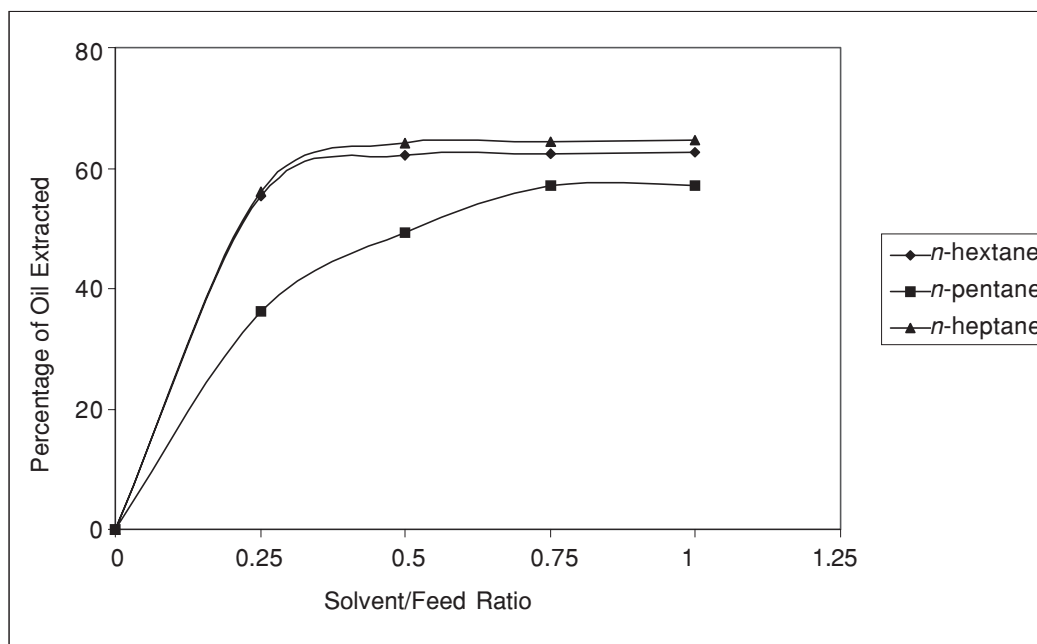


**Figure 2** Percentage of oil extracted verses extraction time at solvent/feed ratio of 1.0, and mixing rate of 200 rpm

range however, it was observed that the extract would become more yellowish. This observation showed that the more oil could be transferred from the aqueous phase to solvent phase and thus increasing the % of extraction. The % of extractions of about 60–70 was achieved by using these solvents at extraction time of above 10 minutes.

### 3.2 Effect of solvent/feed ratio on percent of extraction

These experiments were conducted at solvent/feed ratio of 0.25, 0.5, 0.75 and 1.0. The extraction time and mixing rate were 10 minutes and 200 rpm, respectively. It is clear from Figure 3 that the % of extraction was increased with the increasing of solvent/feed ratio. It is obvious that for both solvents, *n*-hexane and *n*-pentane the % of extraction increases slightly when the solvent/feed ratio increases from 0.25 to 0.5 and reaches a constant value at ratio greater than 0.5. While for *n*-pentane the % of extraction increases gradually when the solvent/feed ratio increases from 0.25 to 0.75 and reaches a constant value after 0.75. This is probably due to the fact that the boiling point of *n*-pentane is lower than that of *n*-heptane and *n*-hexane (Table 2) and this may lead to loss of solvent during the extraction process. The optimum solvent/feed ratio obtained was found to be 1.0 for *n*-pentane and 0.5 for both *n*-heptane and *n*-hexane. The maximum % of extractions of 58, 63 and 65 % was achieved by using *n*-pentane, *n*-hexane and *n*-heptane, respectively at solvent/feed ratio of 0.75–1.0.



**Figure 3** Percentage of oil extracted verses solvent/feed ratio at extraction time of 10 minutes, and mixing rate of 200 rpm

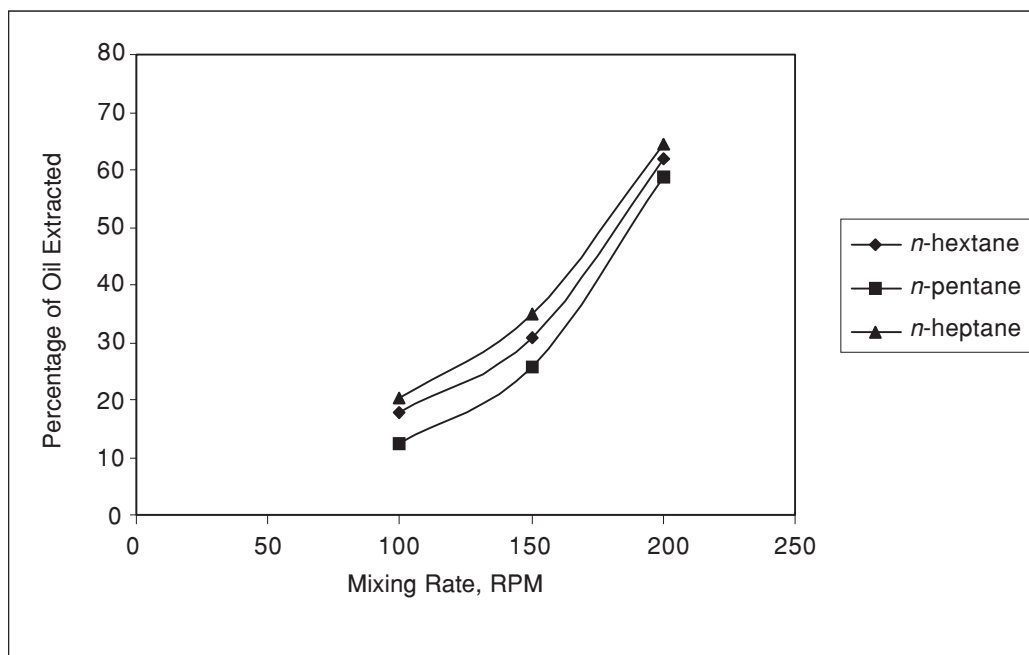
**Table 2** Solubility in water and boiling point of some selected solvents [10]

Solvent	Solubility in 100 Parts of Water	Boiling Point (°C)
n-pentane	0.036 <sup>15</sup>	36.3
n-heptane	0.005 <sup>15</sup>	98.4
n-hexane	0.014 <sup>15</sup>	69.0
Cyclo-hexane	Insoluble	80.1
Chloroform	0.82 <sup>20</sup>	61.2
Dichromethane	0.07 <sup>22</sup>	40.1
Benzene	13	80.1
Acetone	∞	56.5

Superscripts = temperature

### 3.3 Effect of mixing rate on percent of extraction

To study the effect of extraction rate on the percent of extraction, these experiment were conducted at a solvent/feed ratio and extraction time of 1.0 and 10 minutes,

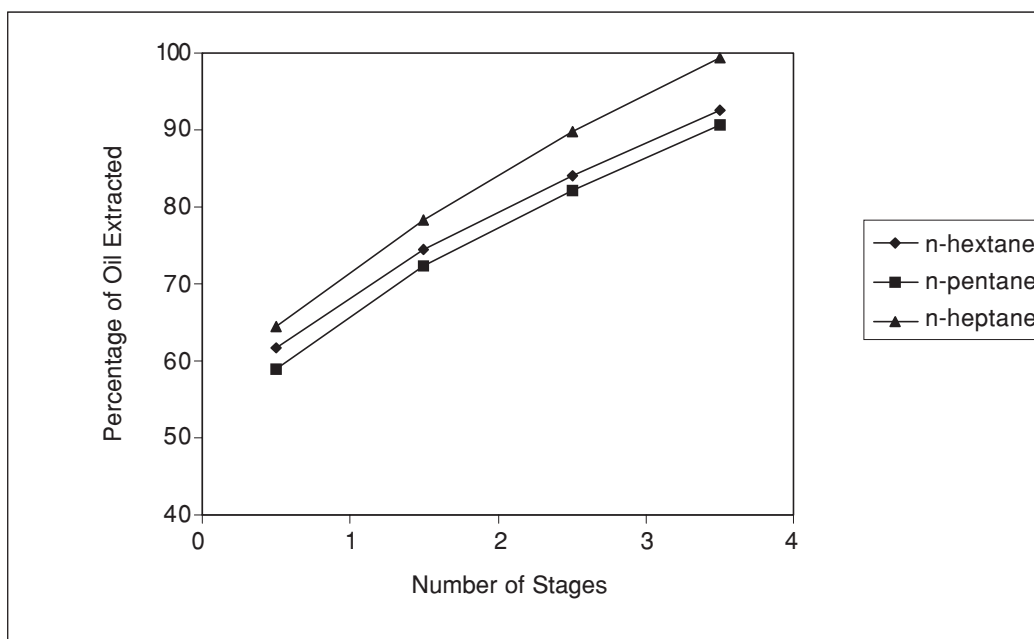


**Figure 4** Percentage of oil extracted vs. mixing rate at extraction time of 10 minutes, and solvent/feed ratio of 1.0

respectively. Figure 4 shows that the % of extraction is increased with increasing mixing rate. This suggests that the extractions are occurred in the diffusional regime where the extraction rate is dependent on the stirring rate. However due to operation limitation of the shaker, further runs at mixing rate higher than 200 rpm were not possible and therefore the behavior of the curve trend was not studied beyond this value. The % of extractions of 58, 63 and 65, was achieved by using *n*-pentane, *n*-hexane and *n*-heptane, respectively at mixing rate of 200 rpm.

### 3.4 Multistage Extraction

The first batch of extraction was conducted at solvent/feed ratio, extraction time, and mixing rate of 1.0, 10 minutes, and 200 rpm, respectively. The raffinate from this batch was used for further extraction and conducted at the same conditions of the first batch. Figure 5 shows the results of these experiments. It can be concluded that the % of extraction is increased with increasing the number of stages from 1.0 to 4.0. Almost 99% of oil was extracted at the 4<sup>th</sup> stage using *n*-heptane while 91 and 93% were obtained using *n*-pentane and *n*-hexane, respectively.



**Figure 5** Percentage of oil extracted verses number of stages for solvent/feed 1.0, extraction time 10 minutes and mixing rate of 200 rpm

#### 4.0 CONCLUSIONS

1. It is found that *n*-heptane is the most effective solvent following by *n*-hexane and *n*-pentane in the extracting of palm oil form POME.
2. The % of extracted oil from POME increases with the increase of mixing time, solvent/feed ratio, and mixing rate for all solvents.
3. The % of extracted oil increase with the increase of the number of stages. Almost 99% of oil was extracted at the 4<sup>th</sup> stage using *n*-heptane while 91 and 93% were obtained using *n*-pentane and *n*-hexane, respectively.

#### ACKNOWLEDGEMENT

The authors are gratefully acknowledging Universiti Sains Malaysia, which has supported this research through a short-term IRPA grant.

#### REFERENCES

- [1] Ngan, M.A. 1999. Environmental management for the palm oil industry. 1-9.
- [2] Setiadi, T and D.A. Husaini. 1996. *Water Sci. and Tech.* 34, Issue 11: 59-66.
- [3] Ahmad, A.L., S. Bhatia, N. Ibrahim and J.M. Tan. 2000. Membrane technology in palm oil mill effluent treatment. *Chemical and process engineering conference 2000*, Singapore, 11-13 December.



- [4] Hoon, N.G., B.H. Hameed, A.L. Ahmad, 2001. Palm oil mill effluent treatment using extraction process: Preliminary results. *Proceedings regional conference for young chemist 2001*: 276-279, 3-4<sup>th</sup> April.
- [5] Esther O. U. 1997. Anaerobic digestion of palm oil mill effluent and its utilization as fertilizer for environmental protection. *Renewable Energy*. 10. Issue 2-3: 291-294.
- [6] Fakhru'l-Razi, A. and M. J. M. M. Noor. 1999. Treatment of palm oil mill effluent (POME) with the membrane anaerobic system (MAS). *Water Science and Technology*. 39. Issues 10-11: 159-163.
- [7] Lai, L. S, A. Fakhru'l-Razi, A. Idris and M.A. Hassan. 1999. The performance and kinetic study of membrane anaerobic system in treating POME. Palm oil mill effluent. *Artificial Cells, Blood Substitutes, And Immobilization Biotechnology*. 27. Issue 5-6: 469-474.
- [8] Chin, K.K, S. W. Lee and H. H. Mohammad. 1996. A study of palm oil mill effluent treatment using a pond system. *Water Science and Technology*. 34, Issue ii: 119-123.
- [9] Borja, R. and C.J. Banks. 1995. Comparison of an Anaerobic filter and an anaerobic fluidized bed reactor treating palm oil mill effluent. *Process Biochemistry*. 30, Issue 6:511-521.
- [10] Robert, H.P. and W.G. Don 1997. *Perry's Chemical Engineers Handbook*. Mc Graw-Hill, Singapore: 2-28