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# RECOVERY OF RESIDUAL OIL FROM PALM OIL MILL EFFLUENT USING POLYPROPYLENE NANOFIBER: A FIELD TRIAL

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### Abstract

Palm oil mill effluent (POME) is an inevitable by-product of palm oil industry that challenges the engineering solutions for its complexity and recalcitrance in nature. This paper reports a new approach to solve the POME pollution, through the recovery of residual oil as opposed to



the elimination approach. A field trial of this approach was successfully carried out in a local palm oil mill in Sandakan, Sabah, Malaysia. The recovery of oil was done using novel polypropylene nanofiber (NF) placed in a sludge pit before the treatment pond. NF was packed in flat sheets of wire mesh and bulk bundles, submerged in the POME stream with 5, 6, 7, 15 hours contact times. Saturated NF was removed from pit and oil was desorbed by manual roller press. It recovered 12.19 g of oil/ g NF in 33.75 hours cycle. The recovered oil contained 77% oil content and FFA of 25.05. GC-FID study of the recovered oil indicated no trace of polypropylene contamination. The NF exhibited oil recovery efficiency of 0.06% using 6.7kg of NF. The efficiency is expected to be significantly enhanced if the POME-NF contact is improved as well as the pressing technique. It can fundamentally change the landscape of POME treatment into sustainable, profitable and economical one.

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### Keywords

Nano Fiber, Oil Recovery, POME, Sterilizer Condensate, Sludge Clarification, Oil Extraction Rate

### **1. Introduction**

Palm oil is the highest yield oil crops with the lowest cost of production compares to other vegetable oils. It contributes 28% or 42 million tons annually of the total world edible oil and grease production. There are total around 800 palm oil mills in the world (Chin, Poh, Tey, Chan, & Chin, 2013) which are all located in developing countries, mainly in Malaysia (50%) and Indonesia (44%), remaining in Colombia (2%), Ecuador, Ghana, Sudan, Thailand and India.

POME is unavoidable pollutant generated by the palm milling process (Yacob, Ali Hassan, Shirai, Wakisaka, & Subash, 2006); blackish to yellowish, concentrated liquid with a distinct offensive odor(Wang et al., 2015), typically colloidal suspension of 95-96% water, 0.6-0.7% oil and 4-5% total solids (A.L. Ahmad, S. Bhatia, N. Ibrahim, & S. Sumathi, 2005; Soh Kheang Loh et al., 2013). Typically production rate for POME is reported as around 0.75 cubic meters per ton of FFB processed by mills (Liew, Kassim, Muda, Loh, & Affam, 2015; Soh Kheang Loh et al., 2013). It is rich in organic matters measure to 25,000 mg/l bio oxygen demand (BOD) & 50,000 mg/l chemical oxygen demand (COD) (Liew et al., 2015). Treating POME is still problematic across the palm oil industries due to both technologies sustainability

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and management will. There are huge demands for sustainable technology that meet the needs of industry to alleviate the perennial POME pollution problem.

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Improving the Oil Extraction Rate (OER) is the most pressing task for these palm oil production nations. It is of profitability and environment sustainability concerns for these nations' agenda to further seek OER improvement for higher productivities with existing planting area. Among many attempts, the most promising area is direct recovery of lost oil in POME. Malaysian palm oil industry's standard OER is around 20% or approximately 0.2 ton of crude palm oil (CPO) per ton of fresh fruit bunch (FFB). 1 ton of FFB produces 0.75 ton of POME and there is 0.7% by weight of residual oil in every ton of POME. It works out to be 5.25 kg of oil lost in POME for every ton of FFB processed. Alternatively there is 5.25 kg oil lost in POME for every 200kg CPO produced. With the potential total recovery of lost oil in POME, it can be translate into 2.6% improve in OER efficiency.

The emulsified oil constitutes the major part of the total pollutants in POME and large portion of this is oil in emulsified form that cannot be separated by gravity separation. The removal of oil and grease from POME is essential to guarantee its effective treatment and the recovery of oil and grease can be a by-product for reutilization (Wang et al., 2015). Untreated or inadequately treated POME can cause severe malodor, water pollution, harm to downstream population and aquatics lives, threat to biodiversity and ecosystem of the riparian zone. The discharge of POME is regulated under Environmental Quality (prescribed premises) (crude palm oil) Regulations 1977and punishable under the Environmental Quality Act 1974(EQA) (A.L. Ahmad, C.Y. Chan, S.R. AbdShukor, & M.D. Mashitah, 2008; Mohd Bakri Ishak, MMIM, & Mohd Armi Abu Samah, 2010; Soh Kheang Loh et al., 2013).

The conventional approach to comply with the EQA requirements is by installing the biological wastewater treatment ponds to treat the POME. However, oftentimes this technique is ineffective and requires big land area. Oil content in the POME is often singled out as the main contributor to the pollution.

The requirement to improve OER has impacted the approach to treat POME. Instead of removing the oil there are numerous works to recover the oil from POME. This approach is more sustainable as it benefits to stakeholders, environmentally friendly, and more importantly it brings financial return to the millers. Several techniques have reported on the recovery of oil such as adsorption of residual oil from palm oil mill effluent using rubber powder (A.L. Ahmad



et al., 2005); solvent extraction, chemical-biological, sedimentation, flocculation, natural fibrous sorbent (Wahi, Chuah, Choong, Ngaini, & Nourouzi, 2013), coalescing, and centrifuge, flotation and coagulation methods (T. Laohaprapanon, P. Prasertsan, & A. H. Kittikun, 2005). However, these methods have poor efficiency to separate emulsified oil-water waste with surfactants (Chen, Liang, Tang, Shen, & Hu, 2017) and normally very expensive.

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Recently a NF made of polypropylene has been discovered to be highly capable of adsorbing oil from POME(Pintor, Vilar, Botelho, & Boaventura, 2016). NF exhibits excellent adsorption behavior to recover residual oil and grease in POME even in very low concentration of oil in water. Furthermore, NF can be reused 20 times without significant loss in its adsorption capacity. Laboratory experiments as reported in earlier part of the work showed the great potential of PP NF for commercial application.

Hence, this field trial reports the oil recovery using PP NF to evaluate its applicability in an actual POME environment. This field trial is crucial to know the real capability of PP NF to absorb oil at various POME condition such as fluctuating effluent flow rates, vary oil content, presence of suspended solids and fluctuating ambient condition. As shown in the findings, PP NF has shown to be very potential for commercial application. Furthermore, several other objectives such as the analysis of quality of oil recovered, the recovery efficiency, and possible PP contamination in the recovered oil are also covered.

### 2. Materials and Methods

### 2.1 Preparation of polypropylene nanofiber

The PP nanofiber was produced using melt-blown technique using the facility in the Faculty of Engineering, UMS. Polypropylene (PP) is the raw material for the nanofiber (David J. Lockwood, Bin Ding, & Jian Yong Yu, 2013; Rosalam Sarbatly, Duduku Krishnaiah, & Zykamilia Kamin, 2016). The PP resins were melted and blow through few very thin nozzles, the thin fiber were formed by 2 forces, air-blowing forces increase the productivity and high electric static force pull and elongate the PP into nano thick fibers; finally fiber rest in the collection net. The machine was operated at applied voltage 200 V, spinning distance 2.4 cm, and the diameter of the nozzle 18 gauge. The PP has molecular weight 42.08, melt flow index 24.5g/ 10 minutes at 230°C, and melting point 160°C. NF exhibits oleophilic and hydrophobic behaviors(Zhu &Guo, 2016). Laboratory experiment results show that oil can be recovered effectively by

adsorption onto NF surface as high as 28 g oil/ g of NF. Further study is crucial to confirm NF as a sustainable solution to the perennial environmental pollution caused by the POME.

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#### 2.2 Site Selection

The week-long field trial was conducted at Syarikat Kretam Mill SdnBhd, owns and operates a 45 ton/h palm oil mill at Mile 44 Sandakan - LahadDatu highway near Bukit Garam, Kinabatangan. It is fully owned subsidiary of KLSE public listed company of Kretam Holding Berhad.Three main sources of wastewater that make up of the POME, namely the hydro cyclone (4%), sterilizer condensate (36%) and sludge clarifier (60%) (Liew et al., 2015), the raw effluent combined and discharge to digestion ponds or other treatment methods. The recovery will be done continuously at the pit before enter into ponds, the quality and quantity of the emulsified oil and grease recovered will also be studied. Recovered oil from these effluents were theoretically same quality as raw crude palm oil (A.L. Ahmad et al., 2008), namely low in FFA and high in DOBI, contain both alpha and beta carotene, which can be sent to oil tank for mixing.

### 2.3 Experimental Setup for the Oil Recovery Work

### 2.3.1 Design of continuous adsorption contact

6.7 kg of NF is prepared into 2 forms, flat sheets sandwich in between 25mm size wire mesh of 45 x 90 cm and flat sheets turn into bulk bundle of rolls. The flat sheets are placed into the pit area; the sheets are floated on the surface of the pit. The rolls are slip into the drain area and in contact with POME that flow through the pit towards the first cooling pond.

### **2.3.2 Determination of effluent flow rate**

The volumetric flow rate of POME was estimated using the historical data of POME flow versus the fresh fruit bunch (FFB) processed. For every ton of FFB produces 0.7 ton of combined POME. This estimation is well in-line with what was reported in literatures.

### 2.4 Design of oil recovery experiments

The palm oil mill effluents (POME) originated from both sterilizer condensate (36%) and sludge clarification (60%) were combined in a mixing pit and flow through a reinforced concrete pit and drain before flow to the first cooling pond. NF was packed in flat sheets of wire mesh and bulk bundles, submerged in the POME stream of pit with 5, 6, 7, 15 hours contact times. Saturated NF was removed from pit and oil was desorbed by manual roller press.

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### 2.5 Recovery of oil from NF

Recover oil from unsaturated NF was proven a very difficult task, for pressed oil quickly re-adsorb into those unsaturated NF surface. By visual inspection of the NF, due to variation in effluent flow rate, the saturated NF was removed from tanks and desorb by using manual roller pressing. Various methods of presses have been deployed during the trial period to desorb oil from NF, among others, press manually by using a long 5cm diameter round rod, heavy round rod of 30cm diameter of approximately 50 kg and by hands on a steel plate perforated with 5mm holes, over a collecting drum underneath. NF of various degrees of saturation, various temperature and direction of pressings were studied. During the actual trial, NF was retrieved and pressed from tanks with temperature range from 60 to  $70^{\circ}$ C.

The pressed NF of various samples was brought back to university laboratory for both solvent extraction and Soxhlet analysis. Known weight of pressed NF was place into the conical flask, known volume of hexane was introduced and magnetic stir continuously for 30 minutes and the extracted oil was weighed. The samples also analyzed by Soxhlet for verification purpose.

### 2.6 Sample analysis

### 2.6.1 Sample preparation (Separation of layers)

The pressed liquid from the NF comprises of water, oil and sludge. Oil was separated by using manual skimming methods when layers formed. For the quantity of oil layer, two methods were adopted; first the skimmed oil layer was weighed and recorded. Second method, the pressed liquid was well mixed on the drum; 250 ml was sampled and placed into a separation flask. It was left for separation for 30 minutes and each layer was weighed. The composition of the sample was used to represent the composition of the total pressed liquid, hence the weight of the recovered oil. All the recovered oil in pressed fluid was manually skimmed into storage drum for further tests.

### 2.6.2 Determination of Oil Content

The recovered oils qualities were analyzed at the mill's laboratory. Among other oil content measurement techniques, such as using nanoemulsion (Costa, Farias, Queirós, & Mansur, 2013), the standard Soxhlet method was used to analyze the oil content of the top layer, supposedly the oil layer. Same method was used to analyze the oil content of water layer. Sample of 20g were poured on to cotton in beaker & measured, dry in microwave for 1 minute, rest for 5





minutes and dry in microwave for another 1 minute. Dried cotton consist of oil was placed into thimble and placed into the Soxhlet. 200ml of n-hexane was used for extraction for 4 hours. Solvent was distilled and removed; the oil sample remained in the still pot was dry in oven of 80°C for 30 minutes, and then cooled in desiccators before it was measured. Minus the known weight of the empty still pot, the weight of the oil content can be determine out of the original sample weight.

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The effluent at the inlet in both condensate and sludge streams were sampled every 30 minutes intervals. 500 ml of the effluent were collected into sampling bottle. The oil content of the effluent samples was analyzed using the standard Soxhlet method. The oil contents were calculated using the theoretical proportion of the effluents from condensate and sludge clarification.

The used and pressed NF samples from condensate and sludge were brought back to university laboratory for oil content analysis. 10 g of pressed NF was sampled; they were respective put into a conical flask with solvent hexane to extract oil from pressed NF, with 10 minutes 150 rpm of magnetic stirring. Solvent were evaporated in 102°C for 30 minutes and weight of remaining oil were recorded. Oil contents were also tested using standard Soxhlet method. This is to determine the amount of oil adsorbed on the NF surface and not recoverable by pressing. This may be referring to as oil holding capacity.

### 2.6.3 Determination of Free Fatty Acid (FFA)

FFA of the oil and water layers was analyzed by using the standard titration method. 0.1 mole of NaOH was used on the known weight of sample. The amount of FFA can be deduced by the amount of NaOH used.

### 2.6.4 Determination of Moisture Content

Moisture content test of top layer was conducted by placing known weight of sample in oven of 80°C for 4 hours, the remaining weight of the sample was measure and record after the moisture was evaporated during the process.

### 2.6.5 Determination of traces of PP

The oils samples were brought back to university laboratory; determination of residual PP was conducted using GC-FID analysis, Table 1 shows the GC setting parameters, for any possible trace of NF pollutant presence in oil samples in micro level.



Column	$30m\times320\mu m\times0.25\mu m$
Inlet temperature:	250°C
Column Flow	1 ml/min
Split Ratio	25:1
Injection Volume	1 µL
Oven Program Initial Temperature	100°C
Ramp 1:	20°C/min
Hold time:	4.00 min
Oven Program Final Temperature	300°C
Hold Time 2:	10.00 min
Carrier Gas:	Helium
FID Temperature:	300°C
H <sub>2</sub> flow	45mL/min
Air flow	450mL/min

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# 3. Results and Discussion

### **3.1 Recovery Efficiency**

The main challenges in conducting experiment in the actual mill operation were the vigorous fluctuation in operation parameters and conditions that included mainly the variation in volumetric flows & concentration of oil content in effluents. Being the two major contributor of the POME (Liew et al., 2015), the quantity of sludge clarification of 60% and sterilizer condensate 36% made up the combined POME. The oil contents of the both effluents from the de-oiling tanks were analyzed using Soxhlet every 30 minutes at the inlets to the CSTR. The sterilizer condensate effluent has an average oil content of 0.92% ( $\pm$  0.20) by weight & the sludge clarification effluent has 1.77% ( $\pm$  0.51) by weight of oil content. Taking into consideration of the percentage, the raw POME oil content is calculated as 1.39% by weight.

Table 2 shows the production of palm oil mill during the actual trial period. It will be used as reference to indicate the total flows of the combined POME in the pits area.



Day	FFB tonnage, t	POME Volume, t	Oil lost in POME, kg
1	375.18	262.63	3,649.79
2	623.80	436.66	6,068.39
3	236.96	165.87	2,305.17
4	293.60	205.52	2,856.17
TOTAL	1,529.54	1,070.78	14,879.52

Table 2: FFB production,	POME volume and oil lost in POME	of the mill during the trial period

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Saturated NF was removed from pit, placed on the steel plate perforated with 5 mm holes and with a collecting drum underneath. It was laid flat and pressed by using roller with force to recover oil; specifically pressing the NF along the fibers' direction. The temperature of between 60 to  $70^{\circ}$ C gave the best desorption results. Hot water was poured on the NF during pressing dissociates oil from NF with much ease. Care was taken to avoid over-compressed and twist that may hurt the fiber's structure for reusability. The level of manual presses may vary but effort was put in to try achieving greatest possible consistency.

It was also observed that NF arrange in flat sheet lined in between 2 flat sheets of iron wire mesh achieve saturation in much short time and adsorption happen in a more effective way for large contact surface. The wire meshes also serve as a protection to over-press of the NF to avoid damages to the NF surface. This packing method points to the potential usage in the actual prototype or industrial applications.

The oil and water mixture from the press was let settle by gravity for at least 10 minutes, separation of layers occurred. Top layer supposedly mainly consist of oil was manual skimming by using container. However, industrial equipment such as centrifuge and skimmer can always be employed to separate pressed liquid from NF where oil was no longer in emulsified form but separate into multiple layers.



Day	Production hours, h	Pressed liquid, kg	Oil recovered, kg
1	7.83	0.71	0.55
2	14.75	3.92	3.02
3	5.00	0.84	0.65
4	6.17	4.76	3.67
	33.75	10.23	7.89

**Table 3:** Amount of oil & moisture recovered by pressing during the trial period

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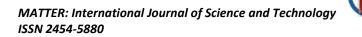
Tables 3 Illustrates the total pressed liquid recovered during the 33.75 hours cycle, where total pressed liquid was 10.23 Kg and taking into consideration of the moisture content in oil layer was 23% or oil content of 77%, the total pure oil recovered was 7.89kg.

A point worth mentioned at this juncture that, there were factors affected the oil recovery in field trial setting; much of oil cannot be skim manually. Oil samples found stuck and lost in press-rods, gloves, drums, beakers, bottles and all apparatus involved. Oil spilt during pressing was unavoidable. All these factors could pose sources of errors in results, more than often, the oil recovery quantities were under stated.

Some weight of oil and moisture remained in pressed NF after compression, they can be deduced by using total weight of pressed NF deducts the 6.7kg of fresh NF used. The weight oil and moisture remained in pressed NF was recorded as 73.82 kg. These samples were further analyzed by using solvent extraction in order to determine the actual amount of oil remained in NF. Table 4 shows the yield of the extraction from both solvent extraction and Soxhlet extraction, where both gives very similar extraction rate of around 32-34%.

	Solvent extraction	Soxhlet extraction
Weight of pressed NF, g	61.11	35.76
Weight of washed/extracted NF, g	18.34	9.33
Oil extracted, g	19.47	12.06
Yield, % wt.	31.86	33.73
Oil holding capacity, kg oil/ kg NF	1.06	1.29

Table 4: Oil extracted by solvent washing and Soxhlet extraction, oil holding capacity of NF





The laboratory reported 4 g oil/ g of NF of oil holding capacity. Table 4 shows the oil extraction from pressed NF using both solvent and Soxhlet extraction methods in order to determine the oil holding capacity of NF. Theoretically, Soxhlet extraction should provide a more accurate extraction results for it was subjected to heat bath. The oil holding capacity for polypropylene NF is around 1.06 to 1.29 g oil/ g NF. Any oil quantity beyond the oil holding capacity, surplus oil adsorbed on to the NF will be able to recover by pressing. The lower the oil holding capacity can be interpreted as easier oil can be desorbed.

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The NF exhibited promising recovery of 12.19 kg oil and moisture/kg NF. Table 5 shows that it recovered 4.89 g pure oil / g of NF over a period of 33.75 h cycle.

	Oil and moisture recovered	Pure oil recovered
Recovered, kg	7.88	7.88
Extract from pressed NF, kg	73.82	24.90
Total, kg	81.70	32.78
Weight of NF, kg	6.70	6.70

**Table 5:** Total oil and pure oil recovered by NF

**Table 6:** Oil recovery efficiency

Run	Total oil lost, kg	Oil recovered, kg	<b>Recovery efficiency, %</b>
1	3,649.79	0.55	0.01
2	6,068.39	3.02	0.05
3	2,305.17	0.65	0.03
4	2,856.17	3.67	0.13
		Average	0.06
Recov	ery, kg oil / kg NF	12.19	4.89

The efficiencies of oil recovery by NF were calculated with necessary information on total oil lost through the system over the 33.75 h of production. The efficiencies were dependent of weight of NF and contact time. Table 6indicate 0.06% of recovery efficiency using 6.7kg of NF, this provides a possible prediction of quantity and contact time necessary for designated recovery efficiency based on this linear relationship of adsorption over time.

### 3.2 Quality of oils recovered

The well mixed samples was left to separate in a separating flask. The quality of oil and water layer was analyzed. Oil content was analyzed by using standard Soxhlet method and FFA by NaOH titration. The oil layer consists of 77% by weight of pure oil and the FFA was 25.

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The quality of oil recovered was in line with earlier reports by other researchers that the recovered sludge oil is poor in quality and will not be included in the production of oil. Instead, these oils are drummed and sold as technical oils for non-edible applications (Liew et al., 2015). However some reported that oil quality recovered was theoretically same quality as raw crude palm oil (A.L. Ahmad et al., 2008).

#### **3.3 Reusability of PP NF**

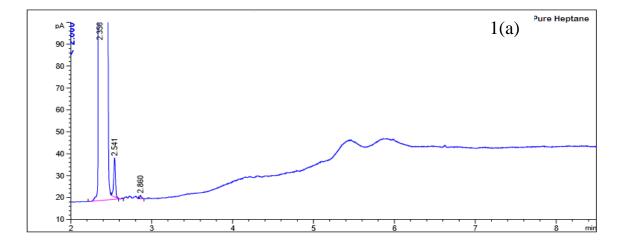
Performance of NF remained effective after 4 rounds of reuse. The GC-FID analysis may also possibly indicated that no disintegration of the PP NF after 4 rounds of reuse. However, such small number of reuse cycle may not be conclusive yet; therefore it would be area for future study pertaining to reusability, in terms of its adsorption efficiency as well as integrity of the material structure in further reuse cycles.

#### 3.4 Detection of contamination in the recovered oil

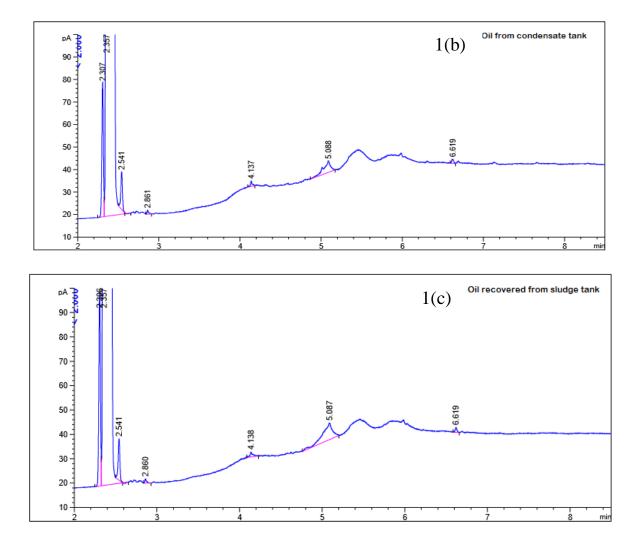
GC-FID analysis was done on recovered oil sample, Figure 1 shows the images of GC-FID analysis for pure heptane and PP diluted in heptane. From the results obtained, it shows that no PP was detected in the solvent. This could infer that PP was not dissolve in heptane even after when vortex vigorously using the GC-FID analysis. There could be possibility that PP is not present in the oil extracted using PP nano-fibers. Figure 1(a) shows the peaks for pure heptane. Three peaks were observed (2.356 s, 2.541 s, 2.860 s). Mixture of PP and heptane was analyzed using GC-FID and Figure 1(b) shows oil recovered by NF from condensate tank, Figure 1(c) oil shows recovered by NF from sludge, shows no additional peaks or contamination for oil recovered samples. Peak at time 5.088 s might refer to one of the fatty acids in the oil. More peaks of fatty acids were expected. However, only one peak was observed. This may due to very low concentration of samples were injected into the GC. By comparing the figures, there is no indication of presences of PP. It can be concluded that, PP is absent in both recovered oil samples, the contamination of PP in recovered oil at the micro level is highly unlikely.







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**Figure 1:** Gas Chromatographic Flame Ionized Detection (GC-FID) analysis of 1(a) pure heptane; 1(b) oil sample from condensate; 1(c) oil sample from sludge. Both sample 1(b) & 1(c) does not show peaks identifiable as PP, it may indicate the absence of PP in the recovered oil samples

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### 4. Conclusion

The field trial of oil recovery using NF was conducted at Kretam Palm Oil Mill, Sandakan, Sabah. The results showed that NF recovered promising 12.19 g of oil/ g of nano fibers recovery in a 33.75 hours operation, which is of remarkable and promising adsorption capacity for residual oil recovery from POME. The oil recovery efficiency is 0.06 % based on 6.7kg of NF used, this provide indicative amount of NF for desired adsorption quantity. NF exhibited good reusability and recyclable behavior, making it a sustainable material for the recover residual oil from palm oil mil. In terms of quality of recovered oil, the recovered top layer has 77% oil by weight and FFA of 25. The GC analysis pointed the absent of PP which showed that contamination of recovered oil by PP was highly unlikely and of good quality oil. It was observed that higher temperature was favorable for desorption of oil from NF and proper contact also enhance adsorption efficiency. The oil holding capacity in the press NF is 1.02 to 1.29 g oil/ g NF; where low oil holding capacity can be interpreted as oil can be easily be desorbed if proper mechanism put in place. Due to complex nature of POME, fluctuation in the operation parameters of pH, temperature, flow rate and large amount of suspended solid, it makes laboratory simulation of POME impossible. Therefore it can only be verified in field trial with actual mill setting. And there are unavoidable fluctuations in operating parameters and oil contains in the POME, which affect the accuracy of readings during field trials. The performance of the NF is expected to improve tremendously in future by optimum hydrodynamics setting of the contact of NF sheets with POME and the mechanization of the oil recovery technique. NF has strong potential to recover residual oil and grease from POME in attempt to further improve OER for palm oil industries.

### 5. Acknowledgement

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