

EFFECT OF PALM OIL STERILISATION TECHNOLOGY ON AQUEOUS CO-PRODUCTS CHARACTERISTICS

FATAH YAH ABD. MANAF^{1*}; ROHAYA MOHD HALIM¹; ANDREW YAP KIAN CHUNG¹
and YAHAYA HAWARI¹

ABSTRACT

With the recent development and new technologies adopted at palm oil mills, the characteristic of aqueous co-products should be updated besides exploring their potential applications. This study was conducted to characterise the aqueous co-products generated from steriliser condensate and sludge separator involving four types of sterilisers (horizontal, continuous, vertical and tilting sterilisers) and empty fruit bunch (EFB) press. The biochemical oxygen demand (BOD) and chemical oxygen demand (COD) concentrations of the overall aqueous co-product were 23 400-36 000 mg L⁻¹ and 53 000-80 000 mg L⁻¹, respectively. The concentration of suspended solids (SS) and oil and grease (O&G) were 21 000-45 000 mg L⁻¹ and 2800-10 200 mg L⁻¹, respectively. Total nitrogen (TN) concentration was 940-1600 mg L⁻¹, while the ammonium nitrogen (AN) concentration was 37-174 mg L⁻¹, with pH ranging from 4.5-5.8. This study indicated that all processing types yield aqueous co-products which were within the range compared to previous studies. The powder generated from the aqueous co-product contained 10.0% oil, 11.7% protein, 5.1% moisture and 13.1% ash. Other minerals such as phosphorous (P), magnesium (Mg), calcium (Ca), zinc (Zn), manganese (Mn) and potassium (K) were also present in the aqueous co-products.

Keywords: aqueous co-products, characteristics, palm oil steriliser.

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INTRODUCTION

Palm oil production consists of several stages, namely sterilisation, stripping or threshing, digestion and extraction. A large amount of water is required to extract crude palm oil (CPO) from fresh fruit bunches (FFB). Consequently, this process generates great volume of aqueous co-products, most of which are discharged into the pond as palm oil mill effluent (POME). Approximately, 0.50-0.75 t of aqueous co-products are produced for each tonnes of FFB processed (Sahad *et al.*, 2014; Som and Wahab, 2018; Yacob *et al.*, 2005). Raw POME or known as aqueous

co-product is a non-toxic thick brownish acidic slurry with pH 3.4-5.2, containing about 95.0% water, 0.6% oil and 4.0% total solids including 2.0% SS (Hassan and Abd-Aziz, 2012; Mohammad *et al.*, 2021) that are mainly debris from palm fruits. Typically, aqueous co-product is accumulated from three different sources, namely steriliser condensate, clarification wastewater and hydro-cyclone wastewater (Wu *et al.*, 2010). Sterilisation is the first contributor to the accumulation of aqueous co-product stemming from steam condensation. Steriliser condensate originates from the condensation of water vapour either from steam injected into the steriliser or evaporation of moisture in FFB due to temperature differences. The condensate consists of oil, water, fibrous material and sand. In addition, the major source of aqueous co-product is from the clarification station. In the clarification process, the crude oil is separated into pure oil and sludge water.

¹ Malaysian Palm Oil Board,
6 Persiaran Institusi, Bandar Baru Bangi,
43000 Kajang, Selangor, Malaysia.

* Corresponding author e-mail: fatahyah@mpob.gov.my

Conventionally, the palm oil mill sterilisation process is carried out in cylindrical pressure vessels at a horizontal or vertical position as a batch process (Sivasothy, 2000). For a well-controlled conventional palm oil mill, about 0.9 m³, 1.5 m³ and 0.1 m³ of steriliser condensate, clarifier sludge and hydro-cyclone wastes are generated, respectively, from each tonnes of CPO produced (Borja and Banks, 1996; Ma, 2000; Madaki and Lau, 2013). Aqueous co-products exist in the forms of high solid, oil, and grease which include biochemical oxygen demand (BOD) and chemical oxygen demand (COD) richness. These are major concerns in environmental pollutants due to their adverse effects on many forms of life (Aziz and Hanafiah, 2017; Ho *et al.*, 2021; Kamyab *et al.*, 2018; Poh *et al.*, 2020). Characteristics of aqueous co-products contain large amounts of total solids (TS) (40 500-75 000 mg L⁻¹), and oil and grease (O&G) (2000-8300 mg L⁻¹). The total nitrogen (TN) is in the range of 400-800 mg L⁻¹ and suspended solids (SS) contents are in the range of 5000-54 000 mg L⁻¹ (Basiron and Darus, 1995; Ma *et al.*, 1999). The most common value for BOD is 25 000 mg L⁻¹ as reported by Ma (2000). The BOD and COD of raw POME can reach up to 50 000 mg L⁻¹ and 100 000 mg L⁻¹, respectively.

In line with the increasing demand for palm oil, the processing system is also seen to evolve through the development of sterilisation technology. *Table 1* shows the types of sterilisers that have been used in palm oil mills in Malaysia. The most critical stage of the palm oil mill process is sterilisation. During the sterilisation process, heat penetrates into the pericarp of fruitlets and causes certain physico-chemical changes such as inactivation of lipase enzymes, thus, preventing the accumulation of free fatty acids (FFAs) within the fruits (Junaidah *et al.*, 2015; Omar *et al.*, 2018). FFAs are formed after hydrolysis of triacylglycerols in the presence of heat and water (Nanssou Koutou *et al.*, 2016). The steam sterilisation technique efficiently removes lipase producing microorganisms from oil palm fruits, but it consumes a large amount of water consequently producing huge volumes of effluent. For every 1 t of FFB sterilised, 0.32 t of steam are required,

and generates 0.27 t of aqueous co-products (Chin *et al.*, 2013). Sterilisation process also facilitates the stripping of fruits from bunches and extraction of oil and kernel. The high heat disrupts the cells in the mesocarp to release oil more readily and allows the detachment of the mesocarp from fruit nuts (Saturday *et al.*, 2017). Inefficient sterilisation in the mill contributed to oil losses and might result in secondary oxidation, leading to the discolouration of palm kernel and decreased deterioration of bleachability index (DOBI) (Wae-hayee *et al.*, 2022).

The conventional steriliser has evolved in recent years into continuous, vertical, tilting, indexer and spherical, with the purpose of improving palm oil production. The consumption of steam for the sterilisation process varies from 110 kg t⁻¹-400 kg t⁻¹ FFB, depending on the type of steriliser technology employed (Energywise, 2013), 140 kg t⁻¹ FFB for a single-peak and 224 kg t⁻¹ FFB for triple-peaks (Abdullah and Sulaiman, 2013). Recently, novel waterless sterilisation methods have been developed using microwave heating (Sukaribina and Khalid, 2009) or extraction with supercritical carbon dioxide (Omar *et al.*, 2018). However, some of these alternatives have a high specific energy consumption that prevents scale-up.

The benefits and advantages of various sterilisation systems are shown in *Table 2* (Parveez *et al.*, 2019). In previous years, millers have not fully discovered the useful purpose for aqueous co-products, which were often considered as waste and burden to the palm oil industry. Nevertheless, numerous attempts had been made in recent years to convert aqueous co-products into profitable value-added products. Fresh POME can be converted into potential feedstuff through fermentation (anaerobic, thermophilic and acidophilic). Previous studies demonstrated the possibility of recovering and concentrating the bio-resources available in POME using ultrafiltration to be more effectively used as fermentation media, fertilisers and animal feed (Suwandi, 1991; Wu, 2006).

With the development and new technologies adopted at palm oil mills, the characteristics of aqueous co-product need to be updated besides exploring their potential applications. Unfortunately, there is yet any study on the characteristics of aqueous co-products produced by different processing systems. Information on the physical/chemical characteristics of these aqueous co-products has not been updated since 1990. Therefore, this study aimed to characterise and analyse the aqueous co-products produced from the different types of processing systems (steriliser and EFB press). Results from the study will provide beneficial information to the palm oil industry to preserve the environment while exploring the economic potential of the aqueous co-products.

TABLE 1. TYPES OF STERILISERS USED IN MALAYSIAN PALM OIL MILL

Type of steriliser	Percentage adopted (%)
Conventional	63.5
Continuous	9.7
Vertical	13.1
Tilting	6.0
Indexer	5.5
Spherical	2.1

Source: Siti Mashani Ahmad (2020).

TABLE 2. THE BENEFITS AND ADVANTAGES OF VARIOUS STERILISATION SYSTEMS

Type of steriliser	Advantages
Horizontal (conventional)	Less investment cost is needed due to simple design. Lower oil losses in condensate compared to other types of sterilisers.
Vertical	High throughput due to no cages. Enable 'one way traffic' FFB handling. System which moves away completely from the conventional cages. Good sterilisation efficiency. Low maintenance cost and minimum downtime due to less moving parts. Low steam consumption and closed batch process. Facilitate automation.
Continuous	Reduce labour dependency significantly. Smaller footprint (conveyer concept) and less machinery. Oil extraction rate (OER) of mills improves significantly. Low pressure vessel for better safety. Efficient use of energy. Facilitate automation.
Tilting	High throughput due to no cages. Lower operation and maintenance costs compared to horizontal steriliser. High sterilisation efficiency due to closed and compacted chamber. Facilitate automation.

MATERIALS AND METHODS

Materials

Aqueous co-products which include a mixture of steriliser condensate, separator sludge wastewater and hydro cyclone wastewater was collected from two sampling points, steriliser condensate and clarification station (sludge separator) (Figure 1). Sterilisation systems consisting of horizontal, vertical, continuous and tilting sterilisers, and EFB press were examined for

their effects on aqueous co-products. Ten palm oil mills were involved in this study and categorised as follows:

- i) Mills A1 and A2 - horizontal steriliser
- ii) Mills B1 and B2 - vertical steriliser
- iii) Mills C1 and C2 - continuous steriliser
- iv) Mills D1 and D2 - tilting steriliser
- v) Mills E1 and E2 - EFB press

Samples were collected hourly over two days using polyethylene containers and divided into

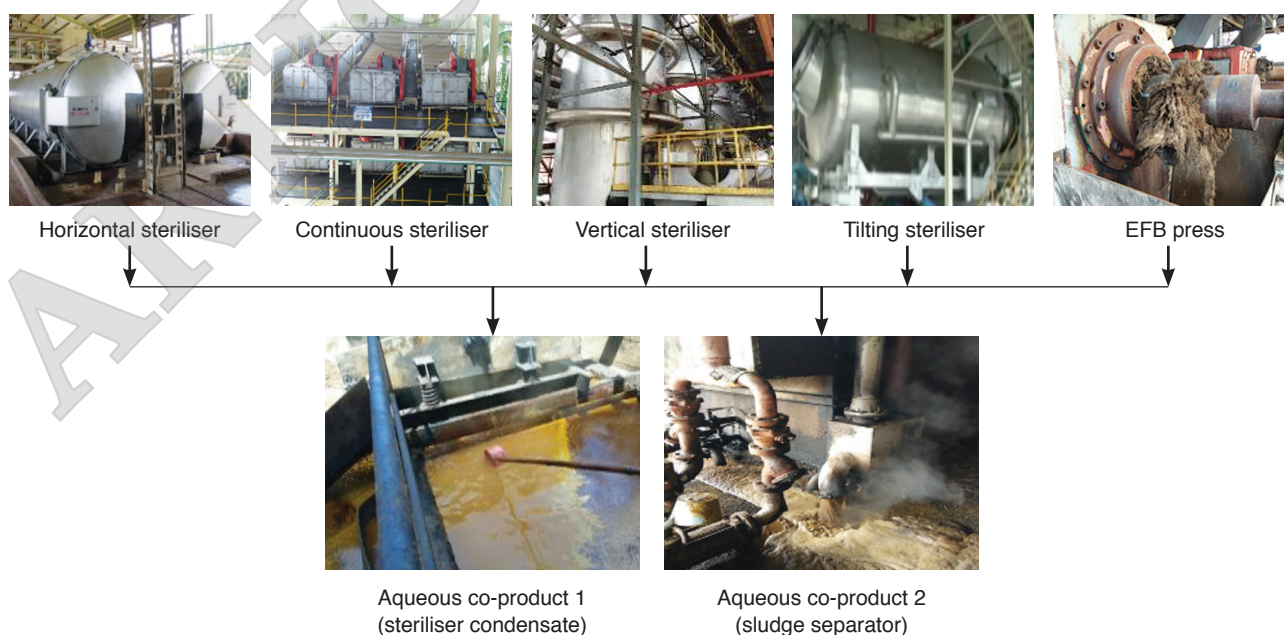


Figure 1. Samples of aqueous co-products from different processing systems.

two batches. Analysis of variance (ANOVA) was employed to study the significant differences between technology employed at the mills and aqueous co-products generated.

Determination of Aqueous Co-product Characteristics

The collected samples were analysed for BOD, COD, SS, TS, O&G, pH and AN according to Standard Methods for the Examination of Water and Waste Water, 21st Edition, American Public Health Association (APHA, 2005).

Production of Dried Aqueous Co-products

Samples of the aqueous co-products were dried using LabPlant SD-06 (OF301) laboratory spray dryer (Figure 2) into powder form. The dried aqueous co-products were analysed for proximate (moisture, protein, oil, ash and caloric value), nutrient content (N, P, K, Mg) and heavy metal (Mn, Ni, Zn, Fe, Cd and Cr). Oils extracted from the dry powder were then analysed for FFA, peroxide value (PV), DOBI and its fatty acid composition (FAC) using gas chromatography.



Figure 2. LabPlant SD-06 (OF301) laboratory spray dryer.

RESULTS AND DISCUSSION

Characteristics of Aqueous Co-products from Different Types of Sterilisation Systems

Steriliser condensate. Table 3 shows the characterisation results of aqueous co-products from steriliser condensate generated from different types of processing systems. Mills A1 and A2 produced aqueous co-products with BOD concentrations of 23 425 mg L⁻¹ and 24 871 mg L⁻¹, respectively. The BOD concentrations for Mills B1, B2, C1, C2, D1 and D2 were 28 696 mg L⁻¹, 25 533 mg L⁻¹, 26 212 mg L⁻¹, 25 650 mg L⁻¹, 27 900 mg L⁻¹ and 26 300 mg L⁻¹, respectively. Overall, the BOD concentrations of horizontal sterilisers were slightly lower than those of new sterilisation systems. Vertical steriliser contributed to higher BOD concentrations as the FFB were immersed in the condensate during the sterilisation process. On the contrary, aqueous co-products produced by continuous steriliser had high BOD as the palm bunches were crushed prior to sterilisation, thus, leaching out oil during the process. In the case of tilting steriliser, the fruit bunches were dropped from the top of the inlet end at a substantial height to the inside of the vessel. Since most sterilisation vessels measured more than 6 m in length, the falling impact is imparted to the oil palm bunches and their fruits. However, the aqueous co-products from the tilting steriliser should have low BOD as the process was similar to the horizontal steriliser.

Statistical results showed that the BOD concentrations in the condensates of all sterilisers were significantly different ($p < 0.05$) and were within the range (10 250-43 750 mg L⁻¹) as reported by Mohammad *et al.* (2021). On the other hand, the COD concentrations of steriliser condensates of all mills were high, ranging from 62 000 to 74 000 mg L⁻¹. Previous study reported that the COD concentrations of steriliser condensate could reach up to 100 000 mg L⁻¹ (Ma *et al.*, 1999).

TABLE 3. CHARACTERISTICS OF STERILISER CONDENSATE AQUEOUS CO-PRODUCT FROM DIFFERENT STERILISER SYSTEMS

Types of steriliser/mills	BOD	COD	SS	O&G	TN	AN	pH	
Horizontal	A1	23 425	64 300	25 640	3 282	955	40	5.36
	A2	24 871	71 200	22 950	2 818	978	37	5.41
Vertical	B1	28 696	53 000	23 320	5 127	962	85	5.79
	B2	25 533	59 900	21 580	5 683	944	119	5.57
Continuous	C1	26 212	62 200	24 040	5 127	1 270	75	5.59
	C2	25 650	73 700	23 610	3 319	1 034	70	5.63
Tilting	D1	27 900	69 200	28 480	4 492	1 149	45	4.90
	D2	26 300	72 175	26 490	5 769	1 439	93	4.64

Note: BOD - biochemical oxygen demand; COD - chemical oxygen demand; SS - suspended solids; O&G - oil and grease; TN - total nitrogen; AN - ammonium nitrogen. All parameters are in units of mg L⁻¹ except pH.

The O&G contents in aqueous co-products from Mills A1 and A2 were 3282 mg L⁻¹ and 2818 mg L⁻¹, respectively. In addition, the O&G contents in steriliser condensate for mills using vertical (5127 mg L⁻¹ and 5683 mg L⁻¹), continuous (5127 mg L⁻¹ and 3319 mg L⁻¹) and tilting (4492 mg L⁻¹ and 5769 mg L⁻¹) sterilisation systems were higher compared to mills using the horizontal system. High O&G content in new steriliser systems could be due to additional pre-treatment prior to sterilisation process. On the other hand, the TN concentrations of steriliser condensate for vertical, continuous and tilting sterilisers were in the range of 900-1500 mg L⁻¹. The normal value of TN is 750 mg L⁻¹ (Ahmad, 2003; Basiron and Darus, 1995; Ma and Ong, 1985). The highest TN value was found in mills using tilting system (1100-1500 mg L⁻¹). Meanwhile, AN and pH produced from all types of mills processing systems were found to be in the range of 37-120 mg L⁻¹ and 4.6-5.8, respectively.

Sludge separator. Table 4 shows the characterisation results of aqueous co-products from the sludge separator. The BOD concentrations in the aqueous co-products obtained from Mills B1 (30 217 mg L⁻¹), B2 (35 901 mg L⁻¹), C1 (30 065 mg L⁻¹), C2 (33 750 mg L⁻¹), D1 (33 800 mg L⁻¹) and D2 (34 050 mg L⁻¹) were slightly higher than conventional mills, namely Mills A1 (26 475 mg L⁻¹) and A2 (30 028 mg L⁻¹). The O&G contents were also slightly higher than that of the conventional mill. High COD concentrations ranging from 68 800-77 800 mg L⁻¹ was recorded for sludge separator aqueous co-product from the clarification station of all mills.

SS is the amount of solids that can be filtered in aqueous. The SS content of aqueous co-products reported by Ma *et al.* (1999) was 18 000 mg L⁻¹, while the SS range reported by Basiron and Darus (1995) was 8000-54 000 mg L⁻¹. Residual O&G are generally trapped in the SS. Removal of SS will remove the O&G as well as some portions of insoluble matter (Ropandi *et al.*, 2010). Statistical

analysis of SS data showed that there was no significant difference ($p>0.05$) in sludge separator aqueous co-products from all mills. In comparison to steriliser condensate, the SS values of the sludge separator were higher. TN concentrations of aqueous co-products from sludge separator for all mills showed no significant difference ($p>0.05$) as well. The TN values ranged from 1000 mg L⁻¹ to 1600 mg L⁻¹. The pH values of sludge separator aqueous co-products for all mills ranged from 4.5 to 5.8.

The BOD concentrations in aqueous co-products contributed by all types of sterilisers both for condensate and sludge separators were in the range reported by a previous study (Mohamad *et al.*, 2021). Mills using horizontal steriliser had the lowest O&G content in steriliser condensate and sludge separator. The ANOVA showed a significant difference ($p<0.05$) for O&G content in mills with different steriliser technologies. Normally, palm oil mills using new steriliser systems experienced higher oil loss in steriliser condensate compared to conventional steriliser. In the conventional milling process, FFB are loaded into cages and pushed into sterilisers for sterilisation. Therefore, FFB did not experience stress and bruising before the sterilisation process. Thus, the oil loss in conventional steriliser condensate was the lowest among other types of sterilisers.

Nevertheless, the quality of oil in the condensate, such as FFA and DOBI, from new steriliser technology is still good (Parveez *et al.*, 2019). Excess O&G is extracted from the oil recovery pit using an oil skimmer.

The results showed that there were significant differences in the COD concentrations of aqueous co-products from sludge separators for all types of sterilisers. It was also found that there was a significant difference ($p<0.05$) in the COD, both in aqueous co-products from sterilisation condensate and sludge separator for all types of sterilisers.

TABLE 4. CHARACTERISTICS OF SLUDGE SEPARATOR AQUEOUS CO-PRODUCT FROM DIFFERENT STERILISER SYSTEMS

Types of steriliser/mills	BOD	COD	SS	O&G	TN	AN	pH
Horizontal	A1	26 475	77 800	44 980	4 901	1 542	4.66
	A2	30 028	77 600	23 600	3 899	1 074	4.70
Vertical	B1	30 217	75 400	29 920	6 468	1 230	5.56
	B2	35 901	69 800	29 860	6 493	1 030	5.53
Continuous	C1	30 065	74 400	43 140	10 177	1 256	5.78
	C2	33 750	68 800	32 610	8 776	1 437	5.82
Tilting	D1	33 800	71 900	43 200	5 648	1 537	4.59
	D2	34 050	77 250	40 880	7 992	1 591	4.56

Note: BOD - biochemical oxygen demand; COD - chemical oxygen demand; SS - suspended solids; O&G - oil and grease; TN - total nitrogen; AN - ammonium nitrogen. All parameters are in units of mg L⁻¹ except pH.

Characteristics of Aqueous Co-products from Mills Using EFB Press

Table 5 shows the characteristics of aqueous co-products in steriliser condensate and sludge separator from mills using EFB press. The BOD, COD, O&G, SS, TN and AN concentrations were in the range of 26 000-31 203 mg L⁻¹, 67 750-79 800 mg L⁻¹, 3065-6607 mg L⁻¹, 24 270-40 160 mg L⁻¹, 440-1147 mg L⁻¹ and 85-155 mg L⁻¹, respectively. Both Mill E1 and E2 use conventional horizontal sterilisers. Results showed that aqueous co-products from mills using EFB press had higher concentrations in terms of the characters studied than that of the conventional mill.

Characteristics of Dry Aqueous Co-product Powder

Table 6 summarises the compositions of dry aqueous co-products powder. The dry powder contained 4.5% moisture and oil content in the range of 11.0%-13.0%. There are more than 59 million tonnes of aqueous co-products produced annually in Malaysia which can be converted into 2.5 million tonnes of powder. Assuming that the dry powder has 10.0% oil content and the oil can be extracted, nearly 0.3 million tonnes of oil can be recovered based on 5.0% solid in aqueous co-products.

Figure 3 depicts the composition of the aqueous co-product powder and the oil extracted from them. The quality of the oil extracted from the dry powder

TABLE 5. CHARACTERISTICS OF AQUEOUS CO-PRODUCTS FROM MILL USING EFB PRESS

Analysis	Steriliser condensate		Sludge separator	
	Mill E1	Mill E2	Mill E1	Mill E2
BOD	28 751	26 009	31 203	29 355
COD	67 750	68 000	78 700	79 800
SS	24 270	27 480	34 290	40 160
O&G	3 065	3 115	6 607	5 948
TN	1 061	440	1 147	958
AN	85	123	85	155
pH	5.51	5.63	5.33	5.57

Note: BOD - biochemical oxygen demand; COD - chemical oxygen demand; SS - suspended solids; O&G - oil and grease; TN - total nitrogen; AN - ammonium nitrogen. All parameters are in units of mg L⁻¹ except pH.

TABLE 6. COMPOSITION OF DRY AQUEOUS CO-PRODUCT POWDER

Element	Composition (% w/w)
Moisture	5.10
Ash	13.10
Protein	11.70
Oil	13.00
Nitrogen (N)	1.650
Phosphorus (P ₂ O ₅)	0.520
Potassium oxide (K ₂ O)	3.150
Magnesium oxide (MgO)	1.070
Calcium oxide (CaO)	1.230
Manganese (Mn)	0.010
Copper (Cu)	0.010
Zinc (Zn)	0.010
Molybdenum (Mo)	0.002
Selenium (Se)	0.003
Calorific value (kg kg ⁻¹)	17 700

was not as good as CPO, with high FFA (15.56%) and PV (59.00 mEq kg⁻¹) content and very low DOBI (0.13) and carotene content (30.87 ppm). The oil extracted from the aqueous co-product can be used as an energy source in animal feed.

Figure 4 shows the FAC in the oil extracted from the dry aqueous co-product powder. Results showed that the FAC were similar to that of CPO. Palmitic acid (C:16) and stearic acid (C:18:1) in the oil extracted from the aqueous co-product powder were 44.4% and 40.4%, respectively. Meanwhile, the total protein content of the aqueous co-product powder was in the range of 10.0%-12.0%. Protein can be isolated from the powder to be used as animal feed. Further studies can be performed to seek a more cost-effective method of isolating protein from the aqueous co-product powder. In addition, the calorific value of the powdered aqueous co-product was about 17 742 kg kg⁻¹, indicating an interesting potential as animal feed. According to Wu *et al.* (2009), fresh POME as dietary substitute for aquaculture organisms has become increasingly important.

Apart from organic composition, the aqueous co-product powder was also rich in minerals, containing an appreciable amount of N (1.85%), P (0.62%), K (3.55%) and Mg (1.17%), which are vital elements for plant growth. The potential use of aqueous co-products as inexpensive organic fertilisers may offer an alternative to the excessive application of chemical fertilisers (Wu *et al.*, 2009). Due to its non-toxic nature and fertilising properties, aqueous co-product powder can be used as fertiliser or animal feed to provide sufficient mineral requirements.

Aqueous co-products of condensate steriliser and clarification station wastewater contained highly degradable organic matter which can be converted into value-added products and chemicals. The aqueous co-products are non-toxic and considered a good source of organic nutrients that can be utilised as renewable energy sources. Previous studies have pointed out the possibility of recovering and concentrating the available bio-resources in raw POME by ultrafiltration to reuse concentrated bio-resources more effectively as fermentation media, fertilisers and animal feeds (Suwandi, 1991; Wu, 2006). According to Habib (1997) and Hwang *et al.* (1978), the possibility of reusing raw POME as fermentation media is largely due to the high concentration of carbohydrate, protein, nitrogen compounds, lipids and minerals contained therein. Wu *et al.* (2009) found that raw POME can be used as a dietary substitute for pigs, poultry and small ruminants, as well as aquaculture organisms. Minerals present in raw POME, such as Fe, Zn, P, Mg, Ca and K, are also suitable for microalgae growth. This is supported by previous studies which reported that the nutrients contained in aqueous co-products (fresh POME) are N, P, K, Mg and Ca (Habib, 1997; Muhrizal *et al.*, 2006).

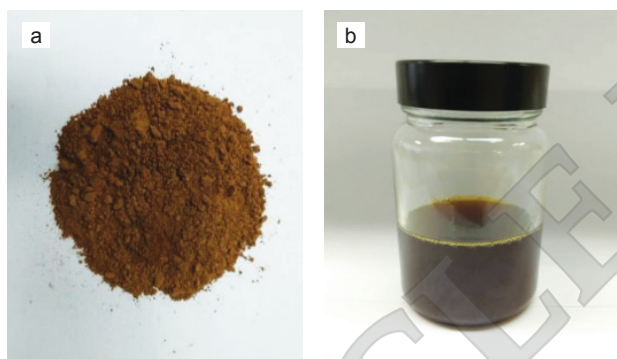
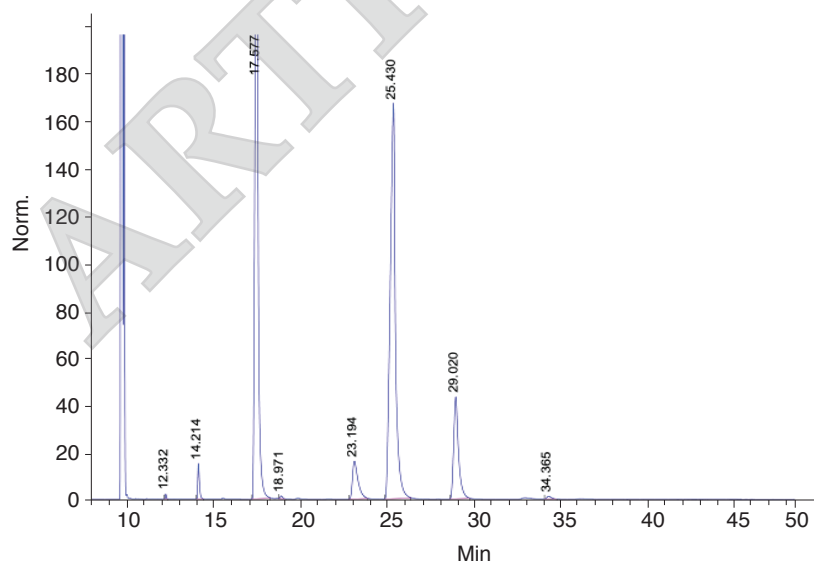


Figure 3. (a) Aqueous co-product powder, (b) oil extracted from aqueous co-product powder.



#	Time	Area%	FA
1	12.332	0.1	C 12:0
2	14.214	1.1	C 14:0
3	17.577	44.4	C 16:0
4	18.971	0.1	C 16:1
5	23.194	4.1	C 18:0
6	25.43	40.4	C 18:1
7	29.02	9.5	C 18:2
8	34.365	0.3	C 18:3

Figure 4. Fatty acid (FA) compositions of oil extracted from dry aqueous co-product powder.

CONCLUSION

The characteristics of aqueous co-product may vary considerably between batches, days and mills, depending on the processing techniques and the age or type of fruits as well as the discharge limit of the mill, climate and conditions of palm oil processing. Analysis results of aqueous co-products indicated that mills using horizontal (conventional) steriliser recorded lower BOD and O&G compared to mills using new technologies. The new system designs induced high oil losses via steriliser condensate and sludge separator. Analysis of variance showed that mills with different steriliser technologies had significant differences ($p < 0.05$) in producing aqueous co-products depending on various factors, such as harvesting season and efficiency of palm oil extraction process and technology. Aqueous co-products should be considered as a valuable resource, and their recovery for other applications is a preferred alternative to conserve the environment than to current practice of treatment and disposal.

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