Pertanika J. Sci. & Technol. 25 (S): 157 - 168 (2017)

SCIENCE & TECHNOLOGY

Journal homepage: http://www.pertanika.upm.edu.my/

Physico-chemical Properties of Residual Oil Extracted from Oil Palm Decanter Cake

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ABSTRACT

In this study, residual oil from oil palm decanter cake (OPDC) was recovered using n-hexane through Soxhlet extraction process. The residual oil recovered was analysed for its physical and chemical properties. The oil content, moisture, Free Fatty Acid (FFA), Peroxide Value (PV), Iodine Value (IV), Deterioration of Bleachability Index (DOBI) and carotene were measured as 15.43±0.45% (dry), 70.96±0.14%, 6.42±0.11%, 4.37±0.04 meq/kg, 53.28±0.02 g/100 g, 1.93±0.03 and 833.20±39 ppm respectively. The results showed that its fatty acid composition, carotene and IV were comparable with the Malaysian Palm Oil Board (MPOB) standard for crude palm oil (CPO), whilst the properties of the other value had slightly exceeded due to oxidation during the extraction process.

Keywords: Carotene, crude palm oil (CPO), oil palm decanter cake (OPDC), physico-chemical properties, Soxhlet extraction

ARTICLE INFO

Article history:
Received: 05 January 2017
Accepted: 17 January 2017

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INTRODUCTION

Oil palm was first introduced to South East Asia in the 19th century. Since then, Indonesia and Malaysia have become the world largest palm oil producers accounting for 85% of the world palm oil production (Koushki, Nahidi, & Cheraghali, 2015). In a study by Mohd Fauzi and Sarmidi (2010), about 80% of the palm oil production is for human consumption whilst the rest was used in animal feed preparation and other industries. Due to

ISSN: 0128-7680

rapid expansion of palm oil production to meet growing demand, large quantities of biomass wastes such as empty fruit bunch (EFB), mesocarp fibre (MF), palm kernel shell (PKS), palm kernel cake (PKC), oil palm decanter cake (OPDC) and palm oil mill effluent (POME) were unfortunately also generated in the mill (Abdullah & Sulaiman, 2013; Embrandiri, Rupani, Quaik, Ibrahim, & Singh, 2012).

The abundance of waste generated has become a major concern due to their environmental effects with attendant impact on sustainability of the palm oil industry. According to Maniam et al. (2013), in Malaysia alone, over 83 million tonnes of dry solid biomass was produced annually by the palm oil industry and the figure is expected to increase up to 85-110 million tonnes by 2020. The POME is the most significant pollutant making up 50% of the waste generated at the mill while OPDC is neglected and left for natural degradation in certain mills (Abdul Razak, Ibrahim, Phang, & Hassan, 2012). Generally, the OPDC consumes a large space at the mill. However, problems will arise when the OPDC becomes dry and could potentially become a fire hazard at the mill (Dewayanto, Isha, & Nordin, 2014). Hence, any strategy in overcoming the environmental problems due to oil palm biomass waste and ways to fully utilise the potential of these biomass wastes into high-value product is welcomed by the government and the palm oil industry.

Currently, CPO is produced in the mills by mechanically extracting it from the digested mesocarp of the palm fruits and using screw presses for the oil extraction process. However, the setback of this mechanical method is that some oil still remains in the mesocarp (Subramaniam, Menon, Sin, & Choo, 2013). It was also reported in other studies presence of residual oils in other oil palm biomass besides mesocarp such as in EFB, POME, OPDC and others. Due to the oil losses in these biomass wastes, it has also brought negative impact to the total oil extraction rate (OER) in palm oil industry (Sahad, Md. Som, Baharuddin, Mokhtar, Busu, & Sulaiman, 2014). Additionally, the presence of residual oil in the biomass waste has indirectly hindered further development in utilising biomass waste. Norul Izani, Paridah, Astimar, Mohd Nor and Anwar (2012), found that the residual oil in EFB need to be removed first before further treatment can be made to EFB in making fibre board and it was reaffirmed by Adam et al. (2014) where the oil in OPDC was removed first before the OPDC-natural polymer composite (NPC) can be produced. In addition, POME alone is made up of 95-96% water, 4-5% total solids, 2-4% of suspended solids and 0.6-0.7% of oil (Wan Sharifudin et al., 2015).

Adam et al. (2014) explained that OPDC was produced from a decanter machine installed after the clarification process of CPO in order to improve the separation of oil and solid. The physical characteristic of the OPDC obtained was a brown-blackish paste made up of oil palm fibre debris. Other than that, OPDC contains mainly water (76% wet basis), residual oil (12% dry basis), nutrients, cellulose, lignin and ash (Maniam et al., 2013). According to Dewayanto, Isha and Nordin (2014), the production rate of OPDC is approximately 4-5 wt% of fresh fruit bunch (FFB) processed which is equivalent to 3.6 million tonnes in 2012. Previously, OPDC is commonly used as fertiliser and animal feed due to the presence of carbon (C), nitrogen (N), phosphorus (P), potassium (K) and magnesium (Mg) (Dewayanto, Husin, Liew, & Nordin, 2010). To date, OPDC has been utilised as feedstock for the production of cellulose and polyose

(Abdul Razak et al., 2012), bio-surfactant (Noparat, Maneerat, & Saimmai, 2014), bio-diesel (Maniamet al., 2013), bio-butanol (Loyarkat, Cheirsilp, & Umsakul, 2013), bio-oil (Dewayanto, Isha, & Nordin, 2014) and as a main component in producing OPDC-NPC (Adam et al., 2014). Previously, Sahad et al. (2014) examined the characteristics and physico-chemical properties of OPDC for better understanding in residual oil recovery. A further study on the residual oil extracted from OPDC should be carry out to understand its material, physical and chemical properties. Thus, this study was carried out to investigate and confirm the physico-chemical properties of residual oil extracted from OPDC.

MATERIALS AND METHOD

Sample Preparation

The OPDC sample was obtained from a three-phase decanter located at FELDA Trolak Palm Oil Mill, Perak,. Based on MPOB Test Method (2004), the OPDC sample was stored at -20°C to maintain its freshness and inhibit degradation. Prior to analysis, the sample was defrosted, oven dried at 103°C (UNB-400 Memmert, USA) and ground to fine powder of <250 µm.The percentage of moisture content was determined using the formula below (1).

$$\text{Moisture content (\%)} = \frac{\text{(Initial weight of sample – Weight after drying) (g)}}{\text{Initial weight of sample (g)}} \times 100\% \quad (1)$$

Extraction of Oil from OPDC using Soxhlet Extraction

Ten grams of dried and ground OPDC was extracted using 300 ml of n-hexane (Merck, Germany) for eight hours in a Soxhlet extractor. The extracted oil was concentrated by removing n-hexane using vacuum rotary evaporator and left in an oven for a while to ensure complete removal of n-hexane. The percentage of oil content was calculated using equation (2).

$$\text{Oil content (\% dry basis)} = \frac{\text{Weight of extracted oil (g)}}{\text{Initial weight of dry sample (g)}} \times 100\% \quad (2)$$

Chemical Properties Analysis of Residual Oil Extracted from OPDC

The residual oil extracted was analysed for FFA, DOBI, PV, IV and carotene content using MPOB Test Method (2004). Each sample was obtained in triplicate. Fatty acid composition was obtained using method AOAC 996.06, 17th Edition.

Fourier Transform Infrared Spectrometer (FTIR) Analysis

The Perkin Elmer Spectrometer, USA was used in this study. A thin film of OPDC was placed between KBr plates and four scans of spectrum in 500-4000 cm^{-1} range were accumulated. A blank KBr plate was used as background for measurement. By using FTIR, the presence of functional groups in OPDC was determined.

Microscopic Observations on OPDC

A Dino-Lite Digital Microscope PREMIER AM/AD7013 Series (Taiwan), at a magnification 200x was used to observe the attachment of oil onto OPDC. Fresh OPDC was mixed with 2 mL of distilled water and placed on a microscope slide. A few drops of Sudan (III) dye solution (Merck, Germany) were added to increase contrast of the preparation.

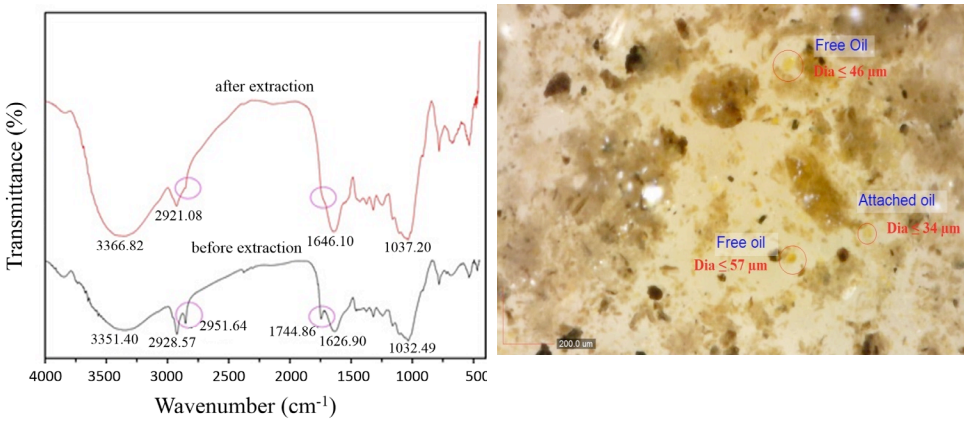
RESULTS AND DISCUSSION

FTIR Analysis and Microscopic View

Based on standard method outline by MPOB, Soxhlet extraction in palm oil was carried out for eight hours. Extraction can be terminated when the yellowish colour of the sample has faded in the extraction chamber of the extractor. In this study, it was observed that after 8 hours of extraction, the yellowish colour of the sample faded indicating that the residual oil in OPDC was successfully recovered. Thus, in order to support this observation, FTIR analysis was carried out to prove that the oil was indeed recovered.

Figure 1(a) showed the FTIR spectra of OPDC before and after extraction. Based on the FTIR result before extraction, several functional groups could be identified. For lignocellulosic material, the O-H group usually corresponds to band at 3600-3200 cm^{-1} and in this study, the peak appeared at 3351.64 cm^{-1} (Sim, Mohamed, Mohd Irwan, Sarman, & Samsudin, 2012; Zakaria, Roslan, Amran, Chia, & Bakaruddin, 2014). Absorption bands at (2928.57 cm^{-1}, 2851.64 cm^{-1}) and (1744.86 cm^{-1}, 1626.90 cm^{-1}) corresponds to the stretching region of CH_2 and CH_3, C=O stretching vibration of carboxyl group (Sahad et al., 2014). According to Laurens and Wolfrum (2011), the hydroxyl and phosphate group are represented at band 1200-500 cm^{-1} and showed the characteristics for lipids and fingerprints for phospholipids. In this study, the peak appeared at 1032.49 cm^{-1} and thus, indicating the presence of residual oil in OPDC.

In addition, the presence of oil in OPDC was further supported by the microscopic view of OPDC in Figure 1(b). From Figure 1(b), several free oils and some oil attached to OPDC fibres can be observed. Based on the result, most of the oil droplets was less than 50µm in size. According to Abbas, Jameel, Muyubi, Karim and Alam (2011), oil droplets in oil-water mixture, with sizes ranging from 20 to 150 µm, are classified as a dispersed oil mixture. According to Chow and Ho (2002), the small oil droplets may have originated from two sources; from the ripe or unripe oil cells which may be covered by phospholipids membrane and another is from the turbulent pumping at various stages of milling process. The surfactants of the cellular fragments tend to be adsorbed at the interface of these oil droplets and thus stabilise them.



(a) (b)

Figure 1. (a) FTIR spectra of OPDC before and after extraction; and (b) Microscopic view of oil attachment in fresh OPDC

Nonetheless, after the extraction of oil, several disappearances of the peaks in the FTIR spectra were observed. The prominent peaks that disappeared were at band 2951.64 cm^{-1} and 1744.86 cm^{-1}. The evident peak of 2951.64 cm^{-1} represent the C-H asymmetric and symmetric stretching vibration of long alkyl chain whereas the peak at 1744.86 cm^{-1} represents carbonyl of carboxyl group (Laurens & Wolfrum, 2011). These peaks were responsible in indicating the characteristics of lipids. Thus, this proves that the residual oil in OPDC was fully recovered using n-hexane in Soxhlet extraction process.

Physico-Chemical Properties of Residual Oil Extracted from OPDC

Physical analysis of residual oil extracted from OPDC. The extracted oil from OPDC is semi-solid at room temperature and is yellowish to brownish in colour. Table 1 shows the physico-chemical properties of OPDC compared with previous studies, and the MPOB standard for CPO. From this study, the oil recovered from OPDC was 15.43±0.45% (dry basis) whilst the moisture content was about 70.96±0.14%. A substantial amount of residual oil recovered from this study is consistent with a previous study by Sahad (2015). Although the oil content was quite low considering the annual FFB processed in the mill, the abundance of OPDC at the mill will gradually ensure the residual oil recovered become significant. The moisture content as shown in this study is similar with that reported by Dewayanto, Husin, Liew and Nordin (2010), Maniam et al. (2013) and Sahad (2015).

Table 1
Physico-chemical properties analysis of residual oil extracted from OPDC and comparison with previous study and MPOB standard for CPO

Properties	This study	Previous study (Sahad, 2015)	MPOB standard for CPO (*Source:* MS 814:2007)
Physical analysis			
Oil yield (%) (dry)	15.43±0.45	12.55±3.15	-
Moisture content (%)	70.96±0.14	78.2±1.27	<0.25
Chemical analysis			
FFA (%)	6.42±0.11	6-15%	<5.0
DOBI	1.93±0.03	1-2	>2.3
PV (meq/kg)	4.37±0.04	-	<2.0
IV (g/100g)	53.28±0.02	-	50.4 to 53.7
Carotene content (ppm)	833.20±39.00	614.67±64.21	474-689

Mean ± standard deviation

Chemical analysis of residual oil extracted from OPDC. The FFA value is an important parameter in determining the CPO quality during production, storage and marketing because it indicates the level of deterioration in oil (Li et al., 2012; Tan, Ghazali, Kuntom, Tan, & Ariffin, 2009). In this study, the FFA value was 6.42±0.11% which has slightly exceeded the standard limit for CPO (<5%) based on MPOB. Nonetheless, the FFA value obtained in this study is consistent with that of Sahad (2015). FFA value will be affected when there is any delay in processing starting from FFB harvesting, storage tank and bruised fruits that will release lipase enzyme which triggered FFA formation and become hydrolysed during the sterilisation process (Sahad, 2015; Vincent, Shamsudin, & Baharuddin, 2014). According to Sahad (2015), the deterioration in FFA value in the residual oil extracted may be due to degradation of OPDC, improper sampling and prolonged thermal treatment during the extraction process.

The DOBI analysis is a good indicator for oxidative status and to determine the oil quality. In this study, DOBI value obtained was 1.93±0.04, which meant the quality of the oil was poor. The DOBI of commercial CPO in palm oil mill is graded as: poor (1.78-2.30), fair (2.31-2.92), good (2.93-3.24) and excellent (above 3.24) (Abdul Hadi, Ng, Choo, & Ma, 2012). Similar to factors that can affect FFA value, the same also applies to DOBI. Mat Jusoh, Abd Rashid and Omar (2013) found the DOBI value decreased significantly due to lengthened thermal treatment as sterilisation process takes some time. The other reasons for low DOBI are high percentage of unripe fruit bunches, contamination of CPO with condensation and oxidation of oils (Abdul Hadi et al., 2012). Thus, in this study, since the OPDC was also exposed to prolonged thermal treatment during oven drying and oil extraction process, it had indirectly caused lipid oxidation in the residual oil extracted.

The PV is an indicator that shows the degree of oxidation in oils (Ekwenye, 2006). There are several factors affecting PV such as temperature, storage time, light and contact with air (Kaleem et al., 2015; Mobin Siddique, Ahmad, Ibrahim, Hena, Rafatullah, & Mohd Omar, 2010). The oxidation process are also explained by Nwabueze and Okocha (2008), whereby when the double bonds in the unsaturated fatty acids are attacked, peroxides will be released

leading to decomposition; thus, secondary products are produced which caused rancidity. Rancidity is also referred to as the spoilage of fats and oils during storage, and usually accompanied with foul odour (Ekwenye, 2006). The PV limit for CPO set by MPOB is below 2.0 meq/kg whereas the PV in this study has exceeded the standard limit with 4.37±0.04 meq/kg. This meant that lipid oxidation has occurred and similar to studies conducted on CPO, the PV of residual oil extracted from OPDC was also affected with prolonged exposure to heat, light, air and long storage time. An earlier study by Sahad (2015) did not examine the PV of residual oil recovered. Information on PV is important because it can measure the extent of primary oxidation of oils (rancidification) that has occurred and this is pivotal because potentially toxic compounds can be produced (Kaleem et al., 2015).

The IV is an indicator to measure the degree of unsaturation or double bonds of fats and oils and the ease of oxidation (Ahmad Tarmizi, Siew, & Kuntom, 2008). Besides that, it can also be used to define the quality and functionality of the fractions (Kumar & Krishna, 2014). In a study by Mobin Siddique et al. (2010), the position of the double bonds or the amount of olefinic carbon are not indicated by IV, however, it is still able to provide overall status of unsaturation in oils so that the position of double bonds prone to oxidisation can be identified. Based on Malaysian Standard MS814:2007, the IV range for CPO is about 50.4 to 53.7. Palm oil is unique as it has an almost equal amount of saturated and unsaturated fatty acids. The IV of the residual oil extracted in this study is 53.28±0.02 g/100 g which is in accordance with the IV for CPO. Nonetheless, similar with PV, IV is also prone to oxidation if exposed to air and light (Mobin Siddique et al., 2010). Nonetheless, similar to PV, IV has not been tackled by Sahad (2015). Thus, this study provides information on residual oil recovered from OPDC.

The distinctive orange-red colour of crude palm oil is due to its high content of carotene (700-800 ppm) with 90% of the total carotenoids made of α- and β-carotene and they also act as precursors of vitamin A with β-carotene having the highest provitamin A activity (Mohd Fauzi & Sarmidi, 2010; Sundram, Sambanthamurthi, & Tan, 2003). Additionally, carotenoids also act as biological antioxidant by having themselves oxidised first before the oxidative attack on triacyclglycerols (TAG) and thus, maintaining the stability and quality of palm oil (Mba, Dumont, & Ngadi, 2015). According to Sundram, Sambanthamurthi and Tan (2003), carotenes are sensitive to oxygen and light and the oxidation of carotenes is accelerated by hydroperoxides generated from lipid oxidation. In this study, a significant amount of carotene, 833.20±39.00 ppm was obtained in the extracted oil. Although several parameters, FFA, DOBI and PV showed lipid oxidation, fortunately the carotene content in the residual oil extracted from OPDC was not greatly affected.

Fatty Acid Composition

Similar to all oils, the major constituents of palm oil are triacyclglycerols (TAG) with over 95% of palm oil consisting of mixtures of TAGs, which is glycerol molecules, each esterified with three fatty acids (Sundram et al., 2003). The fatty acids are any class of aliphatic acids such as palmitic acid (C16:0), stearic acid (C18:0) and oleic acid (C18:1) in animal and vegetable fats and oil (Sundram et al., 2003). According to Koushki, Nahidi and Cheraghali (2015), the

major fatty acids in palm oil are palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2) and myristic acid (C14:0).

The fatty acid composition of the residual oil extracted from OPDC was compared with that of an earlier study by Sahad (2015) and typical fatty acid composition in CPO (Sundram, Sambanthamurthi, & Tan, 2003) in order to confirm whether the compositions were the same (see Figure 2). In this study, the major components of fatty acids extracted are (C16:0) with 45.04%, (C18:1) with 37.49%, (C18:0) with 4.46%, (C18:2n6c) with 9.03% and (C14:0) with 1.09% respectively. Based on the result, more than 50% of the fatty acid composition is made up of saturated fatty acids (palmitic acid, stearic acid and myristic acid). Similar trend was also observed in both previous studies and thus confirming that the composition is the same although the (C16:0) in Sahad, (2015) was slightly higher than this study and CPO whereas its (C18:2n6c) was slightly lower. Palm oil is known for its uniqueness for having an almost equal composition of saturated fatty acids and unsaturated fatty acids making it naturally semi-solid at room temperature (Mba, Dumont, & Ngadi, 2015).

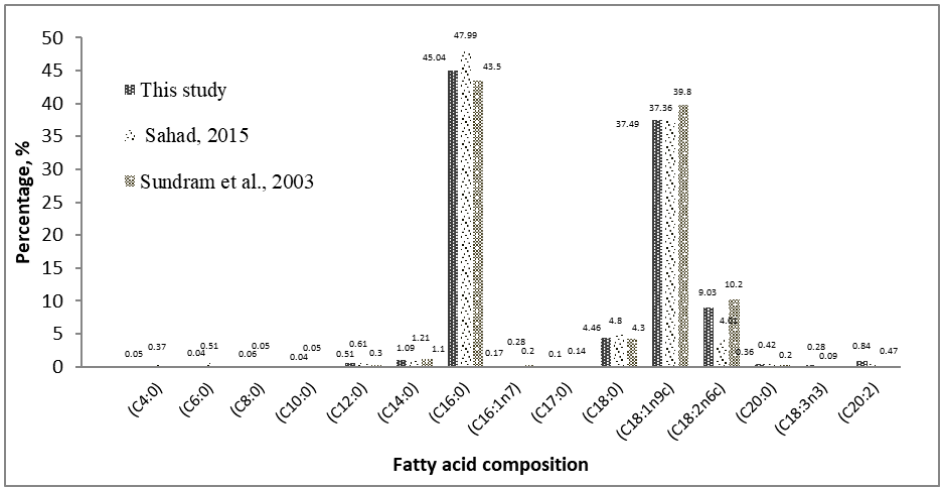


Figure 2. Fatty acid composition (%) of residual oil extracted from OPDC and comparison with previous studies

CONCLUSION

In this study, residual oil from OPDC was successfully recovered and its physico-chemical properties were determined. The residual oil extracted showed satisfactory oil content of 15.43±0.45% (dry basis) with IV of 53.28±0.02 (g/100 g) and a significant amount of carotene at 833.20±39.00 ppm. Since its IV and carotene content is comparable to standard CPO, this also means that there are possibilities for further study in improvising the properties of the residual oil so that it can be utilised in other areas or turned into value-added products.

ACKNOWLEDGEMENTS

The authors express their gratitude the Ministry of Higher Education (MOHE) LRGS Research Grant (600/RMI/LRGS 5/3 1/2012) for financially supporting this study. Thanks also to Universiti Teknologi MARA for providing us the opportunity to undertake this study.

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