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Extraction of Oil from Filter Cake Sludge Using Soxhlet Extraction

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Abstract: This study described the solvent extraction of residual oil from filter cake sludge. This study was conducted using soxhlet method with three different solvents, namely n-hexane, methanol and acetone. Prior to that, the sample was dried at 105°C to obtain the profile of moisture content and optimum drying time. The time consumed to dry the sample is 6 days and the moisture content is 64.12%. The oil extracts were analyzed based on oil yield, pH value, antioxidant, FTIR and GCMS analysis. The result shows that the percentage of oil extracted using methanol (yield 66.6%) is the highest compared to that of acetone and n-hexane. The n-hexane extract is somewhat acidic. The FTIR and GCMS analysis suggested that the functional groups and major components are mostly from the composition of fatty acids. The extraction was able to reduce the weight of filter cake sludge for economical disposal and further studies would be needed to establish the useful applications of the oil extracts.

Key words: Filter cake sludge, oil, solvent extraction, soxhlet extraction, Malaysia

INTRODUCTION

The palm oil plantation was rapidly increased to reduce economic dependence on rubber and tin (Casas *et al*., 2015). Refining process of palm oil is imperative to meet high quality of end products. There are two types of refining process which are chemical or alkaline refining and physical refining (Zin, 2006). In palm oil mill, the waste is normally generated from screw press in order to separate oil with impurities or undesired product, containing about 3% of solid waste and 23.4% of filter cake. While in the clarification process, about 46% of the waste generated is oily sludge.

In wastewater treatment, filter cake is produced after chemical sludge clarifier and biological tank. The filter cake usually contains high moisture content and it is common to get filter cakes with moisture content of around 80% (Yang *et al*., 2011). Water in sludge can be divided into two major categories, i.e., bulk water and bound water. The bound water is often defined as water not readily removed by mechanical technique.

Large load of filter cake sludge can be minimized if it can be converted into useable material (Zaini *et al*., 2013). Normally, the sludge generation from the industries gives bad implications to the environment. Filter cake sludge can cause significant soil pollution and bad odour that affect the public health. While in sugar factory it is considered as waste, posing problems of management and final disposal. According to George *et al*. (2010), the filter cake sludge at the sugar factory can be used as fuel. Usually, land filling and/or incineration are among the solutions to dispose the sludge in palm oil mill industries. Yet, these methods are expensive, not sustainable and may become the secondary sources of air and ground pollution.

Due to great amount of sludge produced daily and taking into consideration the environmental impacts of the current practices, different routes to sludge handling and disposal should be sought. Therefore, attempt has been made in this work to extract oil from the filter cake sludge using different solvents through soxhlet extraction. Methanol, acetone and n-hexane were used as solvent. The oil extracts were analyzed based on pH value, antioxidant, FTIR analysis and composition of constituents using GCMS. This research will discover the possibility of effluent sludge management or utilization of oil extracts as valuable feedstock rather than disposal.

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MATERIALS AND METHODS

Samples preparation: The filter cake sludge was collected from IFFCO (Malaysia). it was dried at 105°C in oven for 6 day. The weight of filter cake was recorded every day for 6 day and the moisture content was calculated. All solvents used are of analytical reagent grade.

Extraction of oil: About 33 g of dried filter cake sludge was added inside a thimble in a soxhlet extractor. About 350 mL of solvent was added into a distillation flask. Three different solvents were evaluated for oil extraction, i.e., n-hexane, methanol and acetone. The solvent was heated to reflux and flood into the chamber housing the thimble containing sludge.

At this stage, oil compounds are dissolved in the warm solvent. Upon reaching a certain level, the solvent is automatically emptied via the siphon side arm and flow back down to the distillation flask. This cycle was repeated for 4 h at atmospheric pressure.

Then, the solvent was recovered using a rotary evaporator to obtain the extracted oil. The temperature of the rotary evaporator was set at the boiling point of each solvent: n-hexane (68°C), methanol (64.7°C) and acetone (56°C) while the speed of the rotary evaporator was fixed at 20 rpm.

Properties of oil extracts: The yield of oil was expressed as mass percentage. The pH value of oil extracts was determined using a digital pH meter.

The antioxidant activity of oil was determined via spectrophotometerical method using free radical 2,2-diphenyl-1-picrylhydrazyl (DPPH). About 3 mg of DPPH was diluted with 100 mL methanol to obtain 3 mL of DPPH solution. Then, 25 mg oil was diluted with 10 mL of methanol in a 15 mL sample bottle. For the antioxidant analysis, About 103 μL of diluted oil was transferred into another sample bottle and 3 mL of diluted DPPH was added in. The sample was analyzed using UV-VIS spectrophotometer (Shimadzu/UV-1800) at a wavelength of 517 nm and the reading was recorded. The antioxidant was calculated as:

$$\text{Antioxidant percentage (\%)} = \left(1 - \frac{A_s}{A_c} \times 100\right) \quad (1)$$

Where:

A_s = Absorbance of the DPPH solution mix with sample

A_c = Absorbance of solution without sample

The available functional groups were determined using a IRTracer-100 (Shimadzu) FTIR analyzer. The compositions of oil extracts were examined using Gas Chromatography (Agilent Technologies-5975 with inert mass selective detector).

RESULTS AND DISCUSSION

Characteristics of filter cake sludge: From Fig. 1, it was observed that the moisture content reaches 64.12 % after 6 day of drying. Besides, the weight of filter cake becomes constant after 4 day. Before soxhlet extraction, moisture content should be reduced to increase the effectiveness of extraction. Basically, drying before extraction helps to rupture the matrix walls so that the solvent can more readily dissolve into oil. In contrast, moisture content would affect the composition of the solvent during soxhlet extraction process, thus increasing the resistance to oil extraction.

The generated filter cake from sugar production industry has a high percentage of moisture content in the range of 75-77% (Kumar *et al*., 2010). The water content in sludge from mill residues of the paper industry is about 76.1% (Wang *et al*., 2006) while that from municipal wastewater treatment contains about 98-99% water (Nunes *et al*., 2008). In addition, the upstream and downstream processes in petroleum industry also generate a great amount of oily sludge containing 30-85% water (Ramaswamy *et al*., 2007).

Effect of solvents in extraction: Normally, oily sludge contains a reasonable amount of oil. Figure 2 illustrates that the highest percentage of oil yield was extracted using methanol, as the solvent gives 66.64% by weight followed by acetone and n-hexane with yields of 33.33% and 13.33%, respectively.

Based on polarity index, the most polar solvent used in this research is methanol. Methanol is commonly used in the production of methyl ester (biodiesel) and is

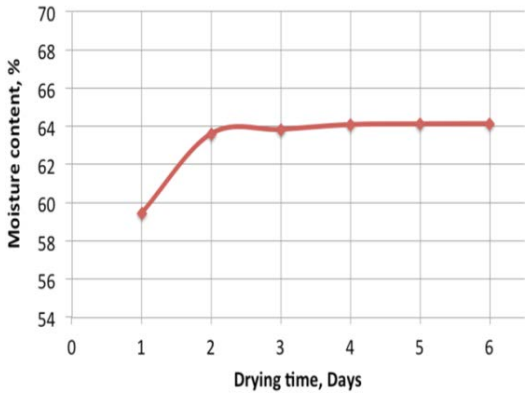

Fig. 1: Moisture contents against drying time

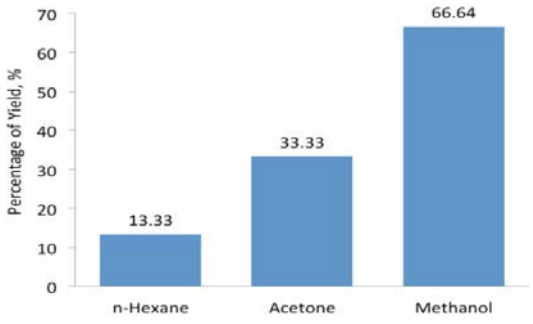

Fig. 2: Oil yields by three solvents

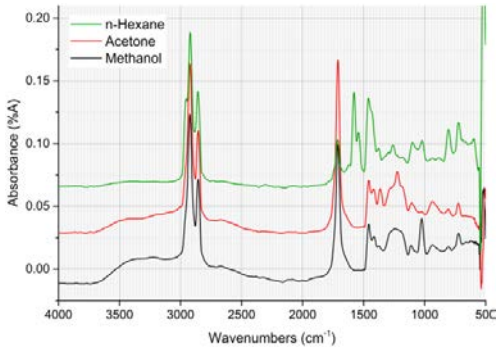

Fig. 3: FTIR spectra of oils extracted by methanol (black), acetone (red) and n-hexane (green)

likely to extract most of the polar (apart from that of non-polar) compounds from the material matrix. Gopal and co-workers (Dashiny, 2009) stated that the oil yields using acetone and methyl ethyl ketone are higher than that of n-hexane and petroleum ether. However, n-hexane was reported to give the highest yield of 10.6% for oil extraction of olive sludge cake from sugar industry (Visioli and Galli, 1998).

Characteristics of oil extracts: Table 1 shows the pH value of oils extracted by three solvents. The pH measurement is very important for analytical and chemical characteristics. Also, the pH value can also be used to identify the quality, storage life and control and monitoring of micro biotical production (Evison, 1989). While in the soap and detergent production, the pH of oil that meets the standard requirement is neutral at scale 6 and 7 (Mustapa, 2015). From Table 1, the oils extracted using acetone and methanol exhibit a somewhat neutral pH of 6.37 and 6.54, respectively. In addition, acidic oil was obtained using n-hexane that gives a pH of 5.27.

Antioxidant analysis is important if the extracted oil is meant for food and soap production. It should be noted that antioxidant is very sensitive to ultra-violet and temperature. In this work, the effect of ultra-violet can be neglected because the experiment was conducted at room temperature and without excessive exposure to ultra-violet. In Table 1, the result shows that methanol extract displays the highest antioxidant percentage of 44.47%.

The least antioxidant percentage was recorded by acetone extract with 32.30%. The main factor that impacts the antioxidant percentage is temperature because soxhlet extraction is based on the boiling point of solvent used (Mustapa, 2015).

Figure 3 displays the FTIR spectra of oil extracts and the peaks assignment are tabulated in Table 1. From Fig. 3, the major functional groups in all oil extracts are C-H (alkanes) and C = O (carboxylic acid). The result

Table 1: Characteristics of oil extracts

			Functional group (cm^{-1})	
Oil extract	pH	Antioxidant (%)	C-H stretch	C = O
n-Hexane	5.21	42.24	2858-2954	1712
Acetone	6.37	32.3	2857-2923	1710
Methanol	6.54	44.47	2856-2923	1710

indicates the presence of fatty acids in the oil extracts. In addition, n-hexane extracts demonstrates several sharp peaks centred at 1600 cm^{-1} indicating the presence of unsaturated (alkene) chains of fatty acid. This explains why oil extracted by n-hexane displayed a lower pH than the other two extracts.

The oil from sewage sludge was reported to display C-N-H bend with peak at 1514 cm^{-1} and peak at 1378 cm^{-1} that corresponds to phenol group (C-O-H) bend and C = O (carboxylic acid) at 1710 cm^{-1} (Samanya, 2011).

The components presence in the oil extracts were analyzed using GCMS. The dominant compounds are shown in Table 2.

Different solvents used in oil extraction show a variety in the composition of volatiles. The number of peak at different retention times for extracted oil by n-hexane is 89. The methanol extract yielded 28 peaks, whereas acetone extract yielded 64 peaks. The major constituents of oil extracts that correspond to the peaks and retention times are tabulated in Table 2. In general, the extracts possess a higher concentration of silicic acid. The presence of silicic acid (ester) in oil is likely to be originated from the use of silicate-type flocculant in the biological tank. Methanol oil extract shows a greater concentration of esters (35.8%). It indicates that the extraction involves esterification reaction and the extract could be further evaluated for biodiesel production. In addition, the methanol oil extract also contains more polar compounds and less fatty acids compared to the other two counterparts.

Table 2: Major components presence in the oil extracts

Component	Composition (mole %)		
	n-hexane	Methanol	Acetone
Silicic acid, diethyl bis (trimethylsilyl) ester	15.2	25.0	19.6
1-Nitro-9,10-dioxo-9,10-dihydro-anthracene-2-carboxylic acid diethylamide	1.3	7.6	7.1
Cyclotrisiloxane, hexamethyl-	2.4	1.6	2.6
Esters	5.4	10.8	12.1
n-Hexadecanoic acid	16.6	0.9	-
Dodecanoic acid	4.1	-	9.4
n-Decanoic acid	1.8	-	5.2
Tetradecanoic acid	4.6	-	4.9
Octanoic acid	0.6	-	1.2
2-Hydroxycarbazole	3.2	-	-
Octadecanoic acid	0.5	-	-
2-Pentene	0.1	-	-
4-Phenyl-3,4-dihydroisoquinoline	-	5.3	1.0
Pyridine, 1,2,3,6-tetrahydro-1-methyl-4-[4-chlorophenyl]-	1.2	6.8	-
7-Chloro-4-methoxy-3-methylquinoline	1.3	3.6	3.5
Purine-2,6-dione, 8-(3-ethoxypropylamino)-1,3-dimethyl-3,9-dihydro-	-	6.0	2.5
Phenol, 2,6-bis(1,1-dimethylethyl)-	1.2	-	-
Disulfide, di-tert-dodecyl	1.6	-	-
Trifluoroacetoxy hexadecane	2.0	-	-
9,10-Anthracenedione, 2-ethyl-	2.9	-	-
(-)-1-Methylbutyl decanoate	3.1	-	-
i-Propyl tetradecanoate	1.7	-	-
Indole-2-one, 2,3-dihydro-N-hydroxy-4-methoxy-3,3-dimethyl-	2.7	-	-
2-Ethylacridine	2.4	-	-
4-Ethylacridine	0.7	-	-
p-Dicyclohexylbenzene	1.4	-	-
N-(4-Bromophenyl)-2-furancarbothioamide	-	3.5	-
1H-Indole, 5-methyl-2-phenyl-	-	6.2	-
2-(Acridin-9-ylamino)-3-methyl-butyric acid	-	3.0	-
1,2-Bis(trimethylsilyl)benzene	-	3.3	-
3-Amino-6-nitro-4-phenyl-1H-quinolin-2-one	-	2.8	-
1-Pyrene-carboxaldehyde	-	-	0.8
4-Hydroxyphenyl pyrrolidinyl thione	-	-	1.4
2-Nitro-4-(trifluoromethyl)phenol	-	-	1.0
2-(Acetoxymethyl)-3-(methoxycarbonyl)biphenylene	-	-	1.1
Phenanthridinium, 5,6-dimethyl-, iodide	-	-	5.8
Tridecanoic acid	-	-	3.6
3-Isopropoxy-1,1,1,5,5,5-hexamethyl-3-(trimethylsiloxy)trisiloxane	-	3.4	-
Anthracene, 9-ethyl-9,10-dihydro-9,10-dimethyl-	-	6.9	-

The n-hexane oil extract contains a higher fraction of saturated fatty acids (28.2%) compared to the acetone oil extract (24.3%). N-hexadecanoic (palmitic or $C_{16}H_{32}O_2$) acid and octadecanoic (stearic, $C_{18}H_{36}O_2$) acid are abundant in the n-hexane oil extract. On the contrary, the compositions of dodecanoic (lauric, $C_{12}H_{24}O_2$) acid, tridecanoic acid ($C_{14}H_{28}O_2$) and n-decanoic acid ($C_{10}H_{20}O_2$) are higher in acetone oil extract than that of n-hexane oil extract.

For comparison, oil extracted using filter cake from sugar factory contains hexadecanoic (palmitic or $C_{16}H_{32}O_2$) acid and octadecanoic (stearic, $C_{18}H_{36}O_2$) acid as the most predominant fatty acids with the compositions of 11.5 and 24.6 mg kg^{-1}, respectively (Casas *et al*., 2015).

CONCLUSION

The filter cake sludge contains considerable fractions of water and oil. The extraction of oil was productively completed in reducing the weight of filter cake sludge for convenient and economical disposal. The oil yield from methanol extraction is higher compared to that of acetone and n-hexane. Yet, the methanol extracts is likely to contain some polar compounds and methyl ester. N-hexane gives an acidic extract. In addition, all extracts exhibit common features of fatty acids. Nevertheless, further investigations would be needed to establish possible utilizations of the oil extracts.

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