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Unlocking the chemistry and properties of oil-containing sludge for potential utilization

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Abstract. At present, possible utilization of oil-containing sludge so as to achieve the sustainable environment has become a subject of considerable interest. The oleochemical factory generates nearly two metric tons of sludge per day from the effluent processing plant. Its disposal is costly and strenuous towards the environment, therefore different alternatives to handling and utilization were studied. The sludge and its derivatives were characterized for elemental composition, proximate analysis, ash and oil constituents, surface analysis, leaching tests and calorific value. Results show that the oil-containing sludge is rich in carbon, calcium, and iron. It has a high calorific value of 33.8 MJ/kg, that is partly attributed to the oil content of 68 %. These preliminary characteristics data offer insight into several promising applications of converting “trash” into “treasure” towards sustainable environment.

***Keywords*:** oil-containing sludge; scheduled waste; sustainable environment; characterization; utilization.

1. Introduction

Oleochemical factory produces refine glycerine, distilled fatty acids, soap noodles and soap bar. Amid the profit-generating activities, it inevitably bears nearly 2 metric tons per day oil-containing sludge from the effluent processing plant. Generally, the sludge is classified under schedule waste under the Environmental Quality Act 1974 [1]. The current practice is to send the sludge to the nearby landfills for disposal, which is undeniably costly. As the world is now moving forward to embrace sustainable environment to achieve circular economy, different routes to sludge handling and disposal would become a subject of considerable interest that worth to be further explored.

The oil-containing sludge is the filter cake at biological tank and chemical sludge clarifier in wastewater treatment plant. It is normally wet and has moisture content of about 80 % [2]. The water retained in moist sludge consists of bulk water and bound water. Bulk water is the free water that can be readily removed by mechanical technique and drying. Bound water is the water of hydration or crystallization, which is a motionless thin film of water strongly bonded to the material surface owing to physical entrapment and hydrogen bond [3]. It is stable and does not react and can never be eliminated from the material.

The sludge may result in environmental pollution if it is not properly handled and disposed. The conventional sludge disposal techniques are landfilling and incineration. They are only short-termed solutions because of high-cost and may trigger secondary soil and air pollution [4]. According to the solid waste management hierarchy, such approach is the least preferred and should be the last resort. Therefore, an attempt has been made to exploit the sludge by evaluating its potential for suitable applications. This route falls under the second (reuse) and third (recycling) layer of the solid waste management hierarchy. To unlocking the potential applications, therefore, the present work was embarked to systematically characterize and evaluate the oil-containing sludge and its derivatives. The results were analysed and discussed to shed better insight into future endeavours.

2. Experimental

2.1. Materials and reagents

The sludge was supplied by oleochemical factory in Johore state of Malaysia. Hexane and ethanol for oil extraction were purchased from R&M Chemicals, and the reagents are of analytical grade. Distilled water was also used in oil extraction for comparison.

2.2. Methods

The raw sludge was spread coarsely on a clean surface and weighed. Then, it was oven-dried at 105 ºC to remove excess moisture. After that, it was cooled to room temperature in a desiccator and weighed periodically for 72 h. The moisture ratio (MR) was calculated as:

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$$MR = \frac{M_t - M_e}{M_o - M_e} \qquad (1)$$

where M_t is the moisture content at any time (%, dry basis), M_o is the initial moisture content (%, dry basis) and M_e is the equilibrium moisture content (%, dry basis).

The sludge ash was prepared by carbonizing the dried filter cake at 900 °C for 3 h in a muffle furnace. The final weight of sludge ash was recorded, and the ash content was determined by dividing the weight of ash with the weight of dry sludge. The ash was characterized for morphology and surface mineral composition using FESEM-EDX (Hitachi SU8020) and leaching elements by inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent Technologies).

Elemental analysis was performed using a CHNS analyser (Elementar vario Macro Cube) to estimate the composition of sludge, while proximate analysis was carried out to determine the moisture content and ash content. The morphology of sludge was observed using a FESEM microscope, coupled with EDX to obtain the mineral composition at the surface. The thermal decomposition profile was obtained under N_2 flow at a heating rate of 10 °C/min using a TGA-DTG (TA Instruments Q-500). The calorific value was measured using bomb calorimeter (IKA C3000).

The pH of sludge was determined by boiling 1 g of sample in 100 mL of distilled water for 0.5 h [4]. The mixture was left to cool at room temperature, and the pH was recorded.

Leaching test was conducted by soaking 1 g of sludge in 100 mL of distilled water for one week [4]. The slurry was filtered, and the filtrate was analyzed for ICP-OES analysis. The sludge samples that preceded by acid digestion were also analyzed for leachable components.

Solvents, i.e., hexane, ethanol, and distilled water were used to extract oil from the sludge using a Soxhlet applicator or simple soaking. A 5 g of dried filter cake sludge was placed in a thimble inside the Soxhlet holder, and 600 mL of hexane was added into the round bottom flask. The solvent was allowed to flood the sludge inside the holder and refluxed for 5 h. Then, hexane was recovered to obtain the extract using a rotary evaporator at its boiling point and 20 rpm [5]. Extraction using distilled water and ethanol only involves simple soaking at room temperature. The oil extracts by different solvents were analysed using a GC-MS (Agilent Technologies-5975 with inert mass selective detector). The oil yield is expressed as mass percentage.

3. Results and discussion

3.1. Composition of sludge

The moisture content of oil-containing sludge is 61.3 %, attained after oven-drying for 3 days. Fig. 1 illustrates the drying curve and drying rate curve of raw sludge at 105 °C without air flow with exposed area of about 80 cm^2. Most of the moisture was removed in the first 1.5 h (5400 s) as shown in Fig. 1a. Generally, it contains moisture lesser than other similar counterparts, such as sewage sludge from municipal wastewater treatment (99 %) [6], filter cake from sugar industry (76 %) [7] and sludge from paper industry (76 %) [8].

Fig. 1b shows the drying rate of sludge. It exhibits three phases, i.e., a short warm-up period (initial rising rate period) and two notable falling rate periods (first and second falling rate period). Similar profiles have been reported for municipal sewage sludge [9] and lignite in thin-layer drying [10]. When the sludge is exposed at 105 °C, the bulk moisture evaporation rate on the surface is greater than the diffusion rate of the internal moisture as the sludge temperature begins to rise to a constant value. This is illustrated by the first rising rate period. The short period of preliminary drying is ascribed to the less amount of free water in the sludge. The drying rate in this phase is governed by temperature and air velocity. In the first falling rate period, the capillary water in the pore network structure evaporates via molecular diffusion through the empty capillaries, suggesting that the drying rate distinctly decreases owing to the increasing inner moisture migration resistance [11]. The temperature of the sludge in this period is constant as the surrounding temperature (105 °C). The drying of sludge then proceeds into the second falling rate period with a mild decrease in drying rate compared to that of the one. This is most likely due to the conversion of capillary water into sorption water (bound water), which is less active and tightly attached to the solid particles, therefore reducing the amount of capillary water.

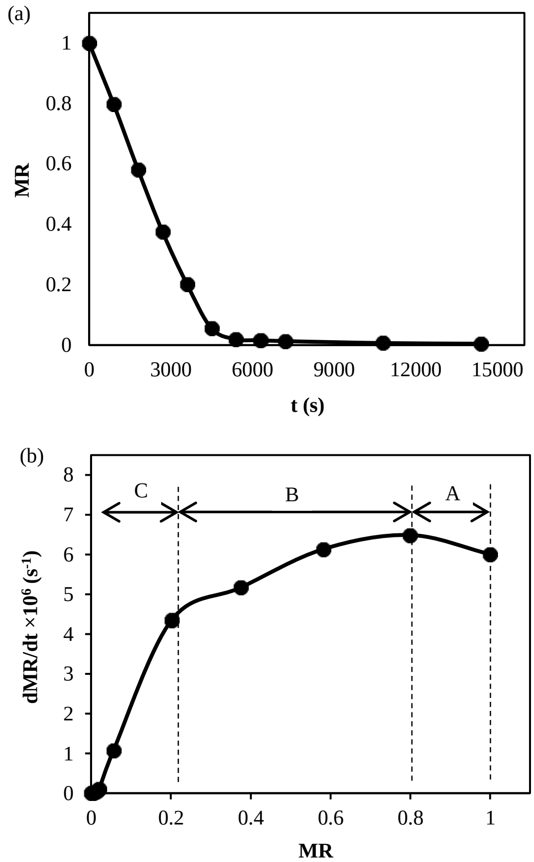

Figure 1. (a) Variation of moisture ratio (MR) with time; (b) drying rate profile of sludge at 105 °C with no air flow (A – the rising rate period; B – the first falling rate period; C – the second falling rate period).

According to elemental composition analysis using CHNS analyser, the dried sludge contains 61.8 % carbon, 9.8 % hydrogen, 0.3 % nitrogen, 0.1 % sulphur and 28 % oxygen (by difference). The high carbon content and small sulphur content are promising traits for making the oil-containing sludge as feedstocks for adsorbent (activated carbon) and solid fuel (briquette). Other industrial sludges have been explored as activated carbon for the removal of dyes, like malachite green [12], reactive red 2 [13] and methylene blue [14].

The ash content in the dried sludge is 10.7 %. The ash constituents present in the sludge as scanned by EDX are mainly calcium and oxygen with more than 40 % each, followed by carbon, iron, and silica (see Fig. 2a). The presence of Fe (6.5 %) and Si (0.5 %) in the sludge is probably associated with the use of silicate-based flocculant and iron-type coagulant in the biological tank. The carbon content decreased from 61.8 % (sludge) to 8.5 % (sludge ash) due to the decomposition of organic matter and fixed carbon upon pyrolysis at 900 °C.

The FESEM image of dried sludge, as shown in Fig. 2b, shows some rudimentary pores present on the rough and uneven surface. White particles as shown in the morphology possibly reflects the presence of inorganic minerals, specifically calcium compounds. To produce activated carbon out of this material, additional step known as chemical or physical activation is essential to create well-developed and functional pores, for applications such as water and wastewater treatment, air and odour pollution control, and chemical purification processes.

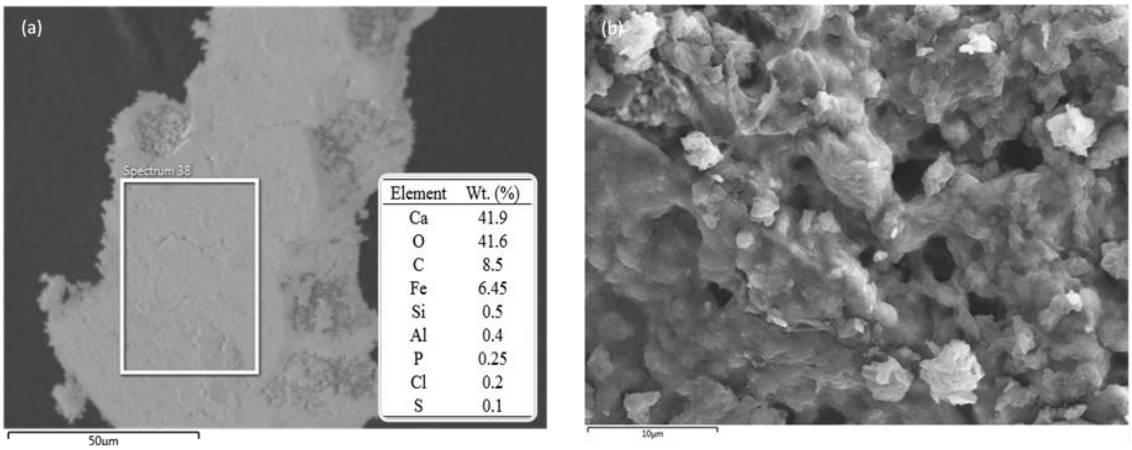

Figure 2. (a) Surface composition of sludge ash; (b) FESEM image of dried sludge.

3.2. Thermal decomposition profile and calorific content

The thermal behaviour and weight loss pattern of dried sludge are displayed in Fig. 3. The ignition temperature of sludge, at which the combustion and decomposition was initiated is at 427 °C, while the burnout temperature, at which the weight of combustible material has achieved 98 % is at 650 °C. The high ignition temperature signifies the unexpected glowing combustion due to inorganic and mineral content. Multiple phase reactions as depicted in Fig. 3 are related to complex sludge composition with high oil content (ca. 68 %). The TGA-DTG curves can be divided into four phases; (i) first phase (0 – 200 °C) is associated with the volatilization and evaporation of unbound water or moisture, (ii) second phase (200 – 400 °C) is ascribed to the combustion of oil, (iii) third phase with a sharp plunge in weight indicates the decomposition of simple structure, thermo-labile organic compounds, and (iv) final phase, wherein the weight loss subsides to a plateau owing to the burning of fixed carbon and decomposition of inorganic compounds like dolomite, kaolin and calcium carbonate [15]. Nearly 80 % of the weight is liberated upon completing all phases. Insignificant weight loss above 650 °C, suggests that the combustion has reached the burnout point. Chen and co-workers [16] reported the multi-stages thermal behaviour of pyrolytic oil droplet from sewage sludge. From Fig. 3, the appropriate carbonization temperature for adsorbent production is 500 °C to allow the decomposition of organic matters to form pores. The TGA- DTG profile is crucial for the characterization of carbonization behaviour because the weight change during pyrolysis could render insight into operating temperature, carbon yield, and textural properties of the resultant adsorbent.

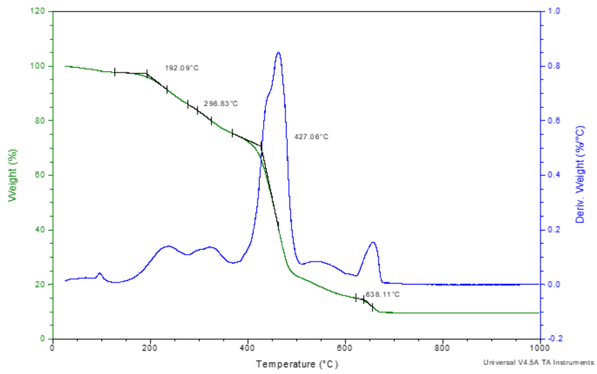

Figure 3. TGA-DTG curves of oil-containing sludge.

The calorific values of the filter cake (wet sludge) and the dried one are 19 MJ/kg and 33.8 MJ/kg, respectively. As the apparent moisture has been initially removed, the dried sludge exhibits a high calorific mainly because the carbon content is more than 60 %. The magnitude of calorific value of dried sludge in this work is twice that of activated sludge (14 MJ/kg) [17], and comparable to carbon fuel (34.1 MJ/kg). It signifies its promising use as an economical and environmentally benign fuel source. George and co-workers [18] reported that the filter cake sludge of sugar factory can be utilized as carbon fuel. Nonetheless, the main hurdles surrounding the application of sludge as solid fuel and briquette, namely the burning retention and probable toxic gas release should be clearly addressed prior to implementation.

3.3. Leaching test

The oil-containing sludge endows a mild acidic pH of 5.5. The test was aimed to detect possible release of minerals and contaminants from different sludge samples, i.e., raw sludge (RS), sludge ash (SA), and oil-containing sludge (SO) by (extreme) acid digestion, and sludge ash by (natural) distilled water (SMW). Generally, calcium was found to be the most abundant mineral, followed by iron, sodium, and titanium (trace, negligible) as displayed in Table 1. Acid digestion is effective to leach mostly all minerals out of the SA, with high concentrations of Ca (6189 mg/L) and Fe (948 mg/L). As the ash content in the dried sludge is only 10.7 %, the fractions of minerals in RS and SO are therefore undoubtedly smaller.

Although Ca and Fe are abundant in SA ash, the poor leaching under natural environment suggests the stability of the minerals and their insolubility in water. The sludge rich in calcium is a good feedstock for concrete blend and clinker in cement manufacturing. Robl et al. [19] patented a hybrid cement clinker that is formulated using industrial sludge, which is more economical and possesses comparable performance to conventional ferrite alite - calcium sulfoaluminate cement [19].

Table 1. Mineral concentrations in four sludge samples (by ICP-OES).

Sample	C_{av} (mg/L)					C_T (mg/L)
	Ca	Fe	Na	Si	Ti	
RS	383	93.9	2.95	9.89	0.12	490
SA	6189	948	3.02	6.52	3.71	7150
SO	94.4	93.9	0.87	5.74	0.00	195
SMW	0.452	0.357	0.318	0.004	0.000	1.13

C_{av}: average concentration of elements; C_T: total concentration of all elements.

3.4. Oil composition

The oil-containing sludge is rich in carbon content. However, part of it is attributed to the presence of oil in sludge. Herein, the oil from the filter cake sludge was extracted using solvents based on the nature and polarity of oil constituents. The solvents are distilled water (very polar, μ (dipole moment) = 1.87 D), ethanol (polar, μ = 1.66 D), and hexane (non-polar, μ = 0.08 D) [20]. Hexane extraction was performed in a Soxhlet apparatus, while extraction by water and ethanol involves soaking at ambient temperature because of high boiling points of ethanol (78 ºC) and water (100 ºC) may trigger accelerated side reactions and degrade certain components during extraction and solvent recovery [21].

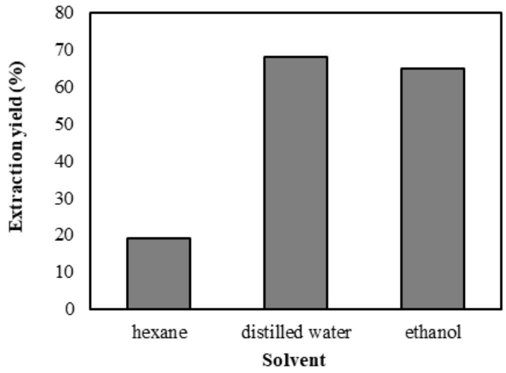

Figure 4. Yield of oil extracts by different solvents.

Fig. 4 shows that water and ethanol render considerably high extraction yields of 68 and 65 %, respectively, while hexane is only 16 %. The high extraction yield could be associated to the polarities of oil constituents that are prone to interact well with polar solvents (water and ethanol) compared to the non-polar one (hexane). Water and ethanol hold hydroxyl groups to allow oil compounds containing hydrocarbon molecules (fatty acid, ester, etc.) to dissolve easily due to strong hydrogen bonding and dipole-dipole interaction. Fauzi and co-workers [5] reported similar results, whereby the oil yield by methanol extraction is higher (67 %) than acetone (33 %) and hexane (13 %). Methanol (μ = 2.87 D, relative polarity = 0.762) is a polar organic solvent, while acetone (μ = 2.69 D, relative polarity = 0.355) is partially polar [20, 22]. In a different work, a 5.16 % of lipid is extracted using methanol/hexane mixture compared to only 1.18 % by acetone/hexane mixture [23].

GC-MS analysis reveals the composition of volatiles with different chemical groups in each oil extract owing to the properties of solvents tied-up with the solubilities of compounds therein. In general, all extracts contain mainly pyridinemethanol (monoaromatic C_6H_9NO) and 2-1-phenyl ethylidene-hydrazono-3-methyl-2,3-dihydrobenzothiazole (polycyclic aromatic, $C_{16}H_{15}N_3S$). Zhou et al. [24] reported the presence of pyridinemethanol in sewage sludge from wastewater treatment plant. Water extraction renders oil with higher concentrations of 7-chloro-4-methoxy-3-methylquinoline (polycyclic aromatic, $C_{11}H_{10}ClNO$), furoic acid (carboxylic acid, $C_5H_4O_3$) and nitrogenated compounds including ethylacridine, acetamide, hexahydropyridine and ethylamine, but trace amount of quinolinone (ketone, C_9H_7NO). 7-Chloro-4-methoxy-3-methylquinoline has been reported in bottom ash of municipal solid waste incinerator [25] and the bark of *Trichilia gilgiana* [26].

Significant fraction of carboxylic acids (85.2 %) was found in oil extract using hexane compared to that by water (17.6 %) and ethanol (21.8 %). Moreover, hexane extracts more tetradecanoic acid (myristic acid, $C_{14}H_{28}O_2$) and hexadecanoic acid (palmitic acid, $C_{16}H_{32}O_2$) than ethanol, but less fractions of ester (4.38 %), aromatic compounds (3.3 %) and nitrogenated compounds (1.32 %). The composition of hexane oil extract implies that it can be used as low-grade oil to produce odourless soap.

On the other hand, ethanol oil extract has higher concentration of ester than hexane oil extract. It indicates the possible esterification reaction that may as well take part during the ethanol extraction. Thus, the extract could be further exploited for biodiesel or liquid fuel production. For comparison, oil extract from anaerobically digested sewage sludge for biodiesel production contains 5.2 % of carboxylic acids, 7.3 % of esters, 7.5 % of nitrogenated compounds and 29.8 % of monoaromatic compounds, wherein the carboxylic acids constitute of hexadecanoic acid (3 %), tetradecanoic acid (0.6 %), octadecanoic acid (0.8 %) and metiltetradecanoic acid (0.6 %) [27].

4. Conclusions

Oil-containing sludge requires preliminary characterization to unlock its possible applications in embracing “waste-to-wealth” towards sustainable environment and circular economy. From the characteristics already presented, the sludge can be turned into viable commodities.

i. The carbon content of sludge is high at 62 %, making it a potential candidate to produce adsorbent or activated carbon. This is backed by the presence of rudimentary pores on its surface. For this reason, the carbonization temperature is estimated at 500 ºC to create high surface area.
ii. The dried sludge exhibits a high calorific content of 33.8 MJ/kg, comparable to carbon fuel (34.1 MJ/kg), signifying that it can be used as solid fuel (briquette) that is economical and environmentally friendly (small fractions of nitrogen and sulphur).
iii. The sludge is rich in calcium. Despite the presence of metallic minerals in ash, the constituents are stable from leaching under natural (water) environment. Iron and calcium in sludge making it a viable feedstock for concrete blend and clinker in cement manufacturing.
iv. Extraction yields by distilled water, ethanol and hexane are 68 %, 65 % and 16 %, respectively. The oil extract can be safely used as low-grade oil to produce odourless soap and biodiesel.

Conflict of interest

Authors declare no conflict of interest.

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