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Examining Selected Physico-chemical Properties of Soil under Contaminated and Control Conditions

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Authors' contributions

This work was carried out in collaboration between all authors. Author OIE designed the study, performed the statistical analysis and wrote the first draft of the manuscript. Authors MKCS and AOO managed the analyses of the study, wrote the protocol and managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The processing of oil palm results in the production of contaminated wastewater commonly referred as Palm Oil Mill Effluent (POME), though this is just one of the waste generated during the processing of the fresh fruits. In this study, soil obtained from the POME disposable sites is referred as contaminated soil. This study was conducted across three locations (Vika, Aro and Jubilee) and selected physicochemical properties (pH, conductivity, organic carbon (OC), moisture content (MC), bulk density, cation exchange capacity (CEC), oil and grease of soil from POME contaminated sites and control sites were determined based on the International Organization for Standardization (ISO) standards. Obtained mean values were collated for statistical analysis to obtain mean standard deviations and *p*-values using the ANOVA and student's t-test. Results revealed that the pH of the

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contaminated soil samples across the mills were acidic with a high mean CEC. The moisture content of the contaminated samples was notably high which could be attributed to the high concentration of organic matter in POME to retain water in the soil. Mean conductivity was significantly high alongside oil and grease concentrations at the mills ranging between 105.75 ± 0.03 (Jubilee) and 790.30 ± 1.00 (Vika). The relatively high values of different physicochemical properties of the contaminated soil against the control site present the palm oil mill effluent as a potential material which could be converted into organic manure and applied back to the plantation rather than disposed-off as an agro-pollutant.

Keywords: Oil palm; POME; organic matter; pH; pollutant.

1. INTRODUCTION

Soilis considered as the most important longterm organic carbon (OC) reservoir in terrestrial ecosystems, as it contains more carbon than plant biomass and the atmosphere [1]. POME is considered as one of the most polluting agroindustrial liquid due to its high organic load, in the form of highly concentrated dark brown colloidal slurry of water, oil and fine cellulose materials proceeding from sterilisation and clarification stages [2]. The nature of POME may depend on the discharge limit of the factory, climate and condition of the palm oil processing [3,4]. Raw or partially treated POME has an organic matter which is due in part to the presence of unrecovered palm oil [5] and from the environmental perspective, fresh POME is a hot and acidic brownish colloidal suspension characterised by high amounts of total solids (40,500 mg/L), O & G (4000 mg/L), COD (50,000 mg/L) and BOD (25,000 mg/L) [6]. According to Vairappan and Yen [7] 66.8 million tonnes of POME were generated in Malaysia in 2005, and based on the statistical value of total crude palm oil production (CPO) in May 2011, the production of 985, 065 tonnes of CPO means a total use of 1.5 million m³ of water, and 738,797 m³ was released as POME in one month alone. In general, one tonne of POME is produced from every 2 tonnes of FFB (Fresh Fruit Bunch) processed from the mill [8]. The waste products from oil palm processing consist of oil palm trunks (OPT), oil palm fronds (OPF), palm oil mill effluent (POME), empty fruit bunches (EFB), palm press fibre (PPF), shell palm oil mill sludge (POMS), and palm kernel cake (PKC) [9,10]. In addition, it has been reported that POME also contains certain powerful water-soluble antioxidants like phenolic acids and flavonoids [10]. Muhrizal et al. [11] noted that in POME contains nitrogen, phosphorus, potassium, magnesium and calcium, which are all vital nutrient elements for plant growth and according to Wu et al. [12], POME as a dietary substitute

for pigs, poultry and small ruminants as well as aquaculture organisms is gaining importance. Singh et al. [13] reported that direct discharge of untreated POME into aquatic environments may cause some pollution problems; as its treatment requires efficient and rapid technology. Yusoff. and Hansen [14], noted that the effluent from the industry is known to be an environmental pollutant based on its high compositions of total solids, suspended organic solids and dissolved organic matter. Rupani et al. [15] also argued that among the wastes that are generated from the processing of oil palm fruits, POME is considered the most harmful waste to the environment if untreated dischargeis done. Interestingly, in an effort to utilise the effluent discharge from palm oil industries, citric acid has been produced using POME as a raw material [16]. Therefore, the results are intended to present the negative or positive potentials of POME which is considered by some as a potential pollutant rather than a potential raw material or resource.

2. MATERIALS AND METHODS

2.1 Description of the Study Area

The study was conducted at selected locations Figs. 1-3, in Akwa Ibom and Oyo state in Nigeria. Sites in Oyo state include Aro (7°42'52"N 3°54'37" E) to Jubilee (7°45'26"N 3°54'57" E) and Vika in Akwa Ibom State (4°56'45.0852"N 8°1'55.26"E). Akwa Ibom State has a tropical climate and rainfall is significant in most months of the year followed by a short dry season. The location is classified as Am (Tropical monsoon climate) by Köppen and Geiger and the average annual temperature in Uyo is 26.4°C with an average rainfall of 2509 mm. Ovo state is considered to be Aw (Tropical wet and dry or savanna climate) according to the Köppen-Geiger climate classification with average annual temperature of 26.5°C and an annual precipitation of 1311 mm. The Köppen climate

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classification divides climates into five main climate groups, with each group being divided based on seasonal precipitation and temperature patterns. The five main groups are A (tropical), B (dry), C (temperate), D (continental), and E (polar). All climates are assigned a main group (the first letter) except for those in the E group which are assigned with a seasonal precipitation subgroup (the second letter) [17].



Fig. 1. Satellite image of Vika oil mill, Akwa Ibom state



Fig. 2. Satellite image of Aro oil mill, Oyo state



Fig. 3. Satellite image of Jubilee oil mill, Oyo state

To determine the bulk density, a metallic core with a known volume (h= 20 cm and d = 8 cm) was used to collect soil samples at well-drained areas. Soil samples for other physicochemical analysis were obtained using an auger driven to a depth of 0 - 20 cm to collect soil contaminated by POME, while control samples were collected 200 m away from the contaminated sites. All samples were collected in duplicates, labelled and transported to the laboratory for analysis in accordance with the ISO certified standard as described below.

2.2 pH Determination

The soil pH was determined with 2mm sieved airdried soil at 1: 2.5 soil to water ratio, using a weighing balance (± 0.001 g), centrifuge tubes, pH meter and end-over-end shaker. 10 (\pm 0.1) g of soil samples were weighed and properly labelled into a 50 ml centrifuge tube, 25 ml of distilled water was added to the samples and tubes were capped. Capped tubes on an endover-end shaker were placed and shaken for 15 minutes at 30 revolutions per minute (rpm). The pH meter was calibrated at pH 4.0 and 7.0 using the appropriate buffer solutions and the pH of the samples was measured and recorded.

2.3 Determination of Soil Moisture Content

The soil moisture content was expressed by weight as the ratio of the mass of water present to the dry weight of the soil sample. To determine the ratios for a particular soil sample, the water mass was determined by drying the soil for 24 hours to constant weight and the soil sample mass was measured before and after drying. The water mass (or weight) was the difference between the weights of the wet and oven dry samples. The criterion for a dry soil sample was the soil sample that had been dried to constant weight in an oven at temperature 105°C. The computation was based on the following formula:

 Θ_{dw} = {(weight of wet soil) – (weight of oven dry soil)/ Weight of oven – dry soil}

 Θ_{dw} = {(weight of wet soil/ Weight of oven – dry soil) x-1}

2.4 Bulk Density (pb) Determination

Bulk density determination was based on the core method. A core sampler was used to collect soil samples. The wet soil was transferred to a

tray and weighed and a portion of the sample was placed in a moisture box, weighed and placed in an oven at 105°C for about 24 hours and weighed again. It was crushed with porcelain mortar and pestle, sieved with a 2 mm sieve and weighed. Bulk density was calculated using the formula:

Bulk density = $M_5 / V \text{ gm.cm}^3$

Where the mass of wet bulk soil sample = M_1 gm, mass of the moisture box = M_2 gm, mass of moisture box + wet soil = M_3 gm, mass of moisture box + oven dry soil = M_4 gm, Mass of wet soil = $(M_3 - M_2)$ gm, mass of oven dry soil = $(M_4 - M_2)$ gm, oven-dry mass of bulk soil sample = $(M_4 - M_2) M_1 / (M_3 - M_2)$ gm or = M_5 gm and volume of bulk sample / core sampler = V cm³ or πr^2h cm³.

2.5 Determination of Organic Carbon

Organic carbon determination was based on the wet oxidation method through acid-dichromate oxidation of soil organic matter and the back titration of unused dichromate with a standard ammonium ferrous sulphate solution. And by this, oxidisable organic carbon was determined. However, computations were based on the equation as shown:

% Org C =((A) - (B) x N x 100 x 0.003 x 1.33/ weight of soil sample)

Where:

A	= Titre value of blank
В	= Titre value of sample
Ν	= Normality of titrant (0.5 N)
Organia matter	- Total argania C x 1 704

Organic matter = Total organic C x 1.724

2.6 Determination of Soil Conductivity

This procedure covered the determination of soluble salts in soil by measuring the electrical conductivity (EC) of a 1:2 soil-water suspension. The electrical conductivity of the soil suspension increased proportionately with salt concentration. A suspension of soil in water (1 part soil-2 parts water) was placed in a conductivity cell, and the electrical conductivity was measured using a soil scoop calibrated to hold 10.0 g of light-coloured silt loam soil and a conductivity meter with an operating range between 0.001-20 dS/m and 50 ml beaker. Soil conductivity was obtained by dS/m = mm hos/cm

2.7 Determination of Cation Exchange Capacity

A standard curve was prepared as per the procedure given in the test for sodium, 6 gm of coarse-textured soil passing through 2 mm sieve was weighed into 50 ml centrifuge tubes. 33 ml of 1 N sodium acetate (pH 8.2) was added and triturated with a glass rod for 5 minutes and the tubes centrifuged at 2000 to 2440 rpm for 10 minutes till supernatant liquid became clear, placing the tubes opposite to each other the supernatant liquid was decanted. The procedure was repeated twice to complete the process of saturation of the exchange complex with sodium. Soil saturated with sodium was washed in an identical manner with three 33 ml of 99% isopropyl alcohol washings. This was done to remove soluble sodium acetate in soil without causing any hydrolysis of exchangeable sodium. The concentration of sodium was determined in CEC extract as per the procedure given for determination of sodium. This is as follows:

Meq of Na / litre in CEC extract = ppm Na in CEC undiluted extract / Eq. Wt. of Na

Meq of Na / 100 gm of soil = (Meq of Na/litre in CEC extract x 100 / 10 x soil in gm taken for preparation of CEC extract)

2.8 Determination of Oil and Grease

Oil and grease were determined using the Soxhlet extraction apparatus. 25 g (+/- 0.5 g) of wet sludge with a known dry-solid weighed and placed in a 150 ml beaker. It was acidified to a pH of 2 with approximately 0.3 ml concentrated HCI. Anhydrous sodium sulfate was added and stirred until the sample appeared dry. The paste was spread on the sides of the beaker to facilitate drying and was allowed to stand until the substance solidified. It was quantitatively transferred to the sample and the paper extraction thimble. The thimble was filled with glass wool and extracted in Soxhlet apparatus using methylene chloride at a rate of 20 cvcles/hr for a minimum of 6 hours or until the solvent became colourless. Using grease-free glass wool, the extract was filtered into a pre-weighed 250 ml boiling flask using forceps to avoid adding fingerprints to the flask. The flask and glass wool were rinsed with solvent. The boiling flask was connected to the distilling head and the solvent was evaporated by immersing the lower half of the flask in water at 70°C. When the temperature in the distilling head reached 50°C

or the flask appears dry, the distilling head was removed. To remove solvent vapour, the flask was swept out for 15 seconds with air by inserting a glass tube that was connected to a vacuum source. Immediately the flask was removed from the heat source and the outside wiped to remove excess moisture and fingerprints. The boiling flask was placed in a desiccator until cool and weighed. Oil and grease were calculated as a percentage of the total dry solids:

% oil and grease = 100*G+D

Where:

 $D = (W * 100) \div (100+ (100*(W-D) \div D))$ G = Gain in weight of the flask D = Dry weight of sample in grams W = Wet weight of sample in grams

Additionally, to ascertain whether or not there were significant differences in their physical and chemical properties, samples were compared using Student's t-test and ANOVA.

3. RESULTS AND DISCUSSION

3.1 Soil pH

The pH of contaminated samples across the oil palm mills was more acidic than that of the control samples as seen in Tables 1-3. Md Din et al. [18], observed that POME is low in pH because of the production of complex organic acids during fermentation process, and the value could be about 4.5 which is suitable to be used as carbon source. The mean soil pH recorded across the entire mill ranged between 4.53 ± 0.20 in Aro and 5.16 \pm 0.08 at Vika oil mill, which showed that the soil was within the acidic range and this could be due to frequent palm oil inundation from spills or direct discharge. This result is close to the findings of other authors who reported that the pH range of POME is in between 4 to 5 [15] and at 4 to 9 [6] after understudying some selected physicochemical and heavy metal properties of palm oil mill effluent. According to Hemming [19], the mean pH of contaminated soil samples was significantly different at (P < 0.05) than that of the control sample, and this low pH of the POME may have been influenced by the corrosion of iron used in processing and organic acids found in fresh fruit [20]. Hemming [19] also noted that when POME is discharged, the pH is acidic but seems to gradually become alkaline as

biodegradation takes place. The value of pH recorded in this study is, however, lower i.e. more acidic than the [21] guideline value (pH 6 to 9) for effluent from vegetable oil processing. According to Okwute and Isu [22], when the untreated POME is discharged into the soil, it affects the nutrient availability of the nearby plants because the most of the plants grow and do better within a pH range of 6.5 - 7.5 [23]. This may be a possible reason for the absence of plants at the disposable site. However, treated POME can be converted into organic fertilizer due to the presence of high total solids in it [24]. From the analysis, the pH of POME contaminated soil was more acidic compared to the control site.

3.2 Soil Conductivity (µS/cm)

The conductivity (µs/cm) values observed from this study (Tables 1-3) indicated that contaminated soil sample from Aro oil mill had the highest mean conductance of 85.15 ± 25.95 . The lower mean soil conductivity from the control samples implies that there were low salts and organic matter concentrations. The mean EC value compares well to the values obtained by Eze et al. [25] and Nnaji et al. [26] who found higher EC values in soil samples contaminated by POME than in control samples as obtainable for this study, where the means of the contaminated soil are relatively high across the study sites. It may be inferred that the higher mean EC value in the oil mill samples reflects a higher deposition of dissolved ions from POME discharge [26] which supports the high values in the contaminated site. Additionally, the increase in conductivity in the POME contaminated sites could be as a result of the loss of weight and release of another mineral salt such as phosphate and ammonium ions through the decomposition of organic substances [27]. The result from Table 1 shows that soil conductivity in POME contaminated soil was higher than the control.

3.3 Soil Organic Carbon (%)

Mean soil organic carbon between contaminated (17.16 ± 3.95) and control (0.92 ± 0.51) soil samples at Vika oil mill was significantly different (P < 0.05). There was no significant difference (p > 0.05) between mean organic carbon of contaminated and control soil samples at Jubilee oil mills (p = 0.542). At Aro oil mill, there was a significant difference (P < 0.05) between the contaminated (12.53 \pm 1.47) and control sites (1.61 ± 0.20). Generally, the mean soil organic carbon values were higher across the contaminated samples than that of the control and this conforms to a past study done by Nnaji et al. [26] at various palm oil mills in Umuahia, Abia State, Nigeria. The study showed that the mean soil organic carbon concentration in the oil mill samples were significantly higher (P < 0.05) compared to the control sample. The higher soil organic carbon from the oil mills in this study can higher palm oil mill be attributed to effluent (POME) discharge and contamination of the soil surrounding the oil mills. Habib et al. [28] showed that POME raises the level of organic carbon of the soil to the range of maximum nutrient availability. And this is in line with the earlier reports of Khalid and Mustafa and Rupani et al. [29,15] who observed that since no chemicals are added during the oil extraction process, POME is considered as non-toxic because it contains substantial amounts of Na, P. K. and Ca which are vital nutrient elements for plant growth. The higher organic carbon value for the POME soil can also be related to the constituents of raw and untreated POME. It is possible that a slow decomposition of organic matter in POME under water-saturated particularly conditions. when mean soil temperature is low [30] contributed significantly to the higher organic carbon of the POME soil. On the other hand, there was no vegetation at the POME disposable site, hence growth performance cannot be established in the present study.

Parameter	Contaminated sample	Control	t	df	P-value
pН	4.88 ± 0.20	5.16 ± 0.08	5.959	2	0.027
Conductivity (µs/cm)	73.05 ± 1.77	9.89 ± 0.04	50.513	2	0.001
OC (%)	17.16 ± 3.95	0.92 ± 0.51	5.767	2	0.029
MC (%)	47.89 ± 0.43	2.92 ± 0.10	143.69	2	0.001
Bulk density (g/mL)	1.13 ± 0.37	1.76 ± 1.44	7.857	2	0.004
CEC (meg /100 g)	3.87 ±0.41	3.16 ± 0.32	6.231	2	0.002
Oil & Grease	790.3 ± 1.0	112.65 ± 1.06	660.533	2	0.001

 Table 1. Physico-chemical parameters of soil at Vika oil mill

Parameter	Contaminated samples	Control	t	df	P- value
рН	4.53 ± 0.20	5.06 ± 0.04	3.692	2	0.066
Conductivity (µs/cm)	85.15 ± 25.95	7.73 ± 1.56	4.212	2	0.042
OC (%)	12.53 ± 1.47	1.61 ± 0.20	10.42	2	0.009
MC (%)	68.84 ± 1.31	14.99 ± 0.01	58.22	2	0.001
Bulk density (g/mL)	0.58 ± 0.49	1.40 ± 0.99	3.962	2	0.019
CEC (meq /100 g)	4.65± 0.31	3.14± 0.10	5.321	2	0.030
Oil & Grease	330.20 ± 0.04	130.95 ± 0.01	2.98	2	0.029

Table 2. Physico-chemical parameters of soil at Aro oil mill

3.4 Soil Moisture Content (%)

There was a significant difference (P < 0.05) in moisture content mean soil between contaminated and control soil samples at all the oil mills (Tables 1- 3). This corroborates to the mean soil moisture content (20.9 ± 0.80 - 40.8 ± 0.10) reported by Enyoh et al. [31] at Ihube community, Okigwe, Imo State, Nigeria. Higher concentration of soil moisture content at the study site may be due to increased decomposition or microbial activities in POME. The oil mills had mean soil moisture content ranging from 2.92 ± 0.10 to 68.84 ± 1.31. The soil type (loamy) was also an important factor for the high moisture content at the oil mills. Loamy soil generally contains more nutrients, moisture, and humus than sandy soil with better drainage and infiltration of water and air than silt and clayrich soil. Notably, POME application to soil can result in some beneficial soil characteristic like increased organic matter content and water holding capacity [14] which is evident in the result. The major factors for the high moisture content were unrecovered oil and cellulose fruit debris (higher organic matter) of the POME soil when compared to the control site [32]. Notably, the absence of vegetation at the disposal site can be explained from the fact that untreated POME soil's ability to retain water could cause clogging of soil pores and hence waterlogging of the soil [33]. This excess water in soil restricts microorganisms and their activities by preventing oxygen movement into and through the soil.

3.5 Soil Bulk Density (g/mL)

A bulk density (BD) estimate is required to calculate soil organic carbon stocks in tonnes of carbon per hectare. Soil tests for organic carbon normally report a percentage of total soil organic carbon (%), using a measure of bulk density to estimate the amount of carbon per hectare in a given depth of soil. Mean soil bulk (g/mL) across three density mills are represented in Tables 1-3. Mean values obtained for the sandy loam soils from contaminated and control samples were significantly different (P < 0.05) and ranged between 0.56 ± 0.44 and 1.76 ± 1.44. According to the USDA guideline for estimating bulk density, the range for soils classified as sandy loam falls within 1.50 -1.60 g/mL [34]. This confirms that mean values obtained for the contaminated samples (0.56 ± 0.44) were lower when compared to the textural class and Bauer and Black [35] explained that soil bulk density decreases with increasing organic carbon concentration which explains the reason for lower bulk density values in the soil due to POME. Across the three oil palm mills. bulk density at the POME contaminated soil was lower than that of the polluted sites, based on the fact that organic soil materials have lower bulk density because it is less dense and has a lower degree of compaction [36]. However, POME contains organic /debris from the oil processing mill.

Parameter	Contaminated samples	Control	t	df	P-value
рН	4.92 ± 0.06	5.16 ± 0.07	3.748	2	0.064
Conductivity (µs/cm)	27.52 ± 12.42	8.75 ± 3.40	2.061	2	0.015
OC (%)	4.89 ± 2.68	3.47 ± 0.62	0.728	2	0.542
MC (%)	24.48 ± 1.03	19.20 ± 0.47	6.622	2	0.022
Bulk density (g/mL)	0.56 ± 0.44	1.22 ± 0.89	3.990	2	0.028
CEC (meg /100 g)	4.60 ± 0.23	3.80 ± 0.03	2.410	2	0.003
Oil & Grease	130.95±13.76	105.75 ± 0.03	3.051	2	0.038

Table 3. Physico-chemical parameters of soil at Jubilee Oil mill

3.6 Soil Cation Exchange Capacity (meq/100 g)

Cation exchange capacity is the number of exchangeable-cations adsorbed in colloid surface and represents the ability of the soil to supply nutrient or gross guide about the type of mineral clay and the presence of organic material [37]. Mean soil cation exchange capacity across the mills is presented in Tables 1-3. Mean values obtained were higher in the contaminated samples at $(4.65 \pm 0.31, 4.60 \pm 0.23 \text{ and } 3.87 \pm 0.23 \text{ and } 3.8$ 0.41) than in the control $(3.14 \pm 0.10, 3.80 \pm 0.03)$ and 3.16 ± 0.32). In a similar study [38], the results showed an overall increase in the CEC of POME soils especially at the area close to the source of the POME and agree with the observation in this study. Organic matter plays a role in the CEC of a soil and it implies that low organic matter in the soil can give rise to equally low CEC values which could be the possible reason for the low CEC values at the control site, with very low organic carbon contents [39.40]. The increase in CEC could be attributed to the increase in the pH-dependent charge as well as the addition of organic matter from the effluent [41]. CEC from the POME contaminated sites have slightly higher values than soil at the control site and can be related to past studies which reveal that soil with the higher organic matter have higher CEC and vice-versa [42].

3.7 Soil Oil and Grease Concentration

Mean soil oil and grease concentrations at the oil mills ranged between 105.75 ± 0.03 (Jubilee) to 790.30 ± 1.00 (Vika). These high values are supported by Ma [6] who reported the high concentration of oil and grease level in POME ranging from 4000- 6000mg/l. The higher soil oil and grease concentrations in this study is attributed to higher palm oil mill effluent (POME) discharge to soil as a result of probable increased oil palm milling activities. The highest mean soil oil and grease concentration (790.3 ± 1.00) was obtained from the contaminated site at Vika oil mill. This results from the fact that POME is disposed of in a shallow pit which stagnates permanently, hence the values. On the contrary, the discharge point for other mills (Aro and Jubilee oil palm mill) were runways,/water ways and as a result, the POME discharged was diluted or washed away by running water. Hajek et al. [23] reported that oil and grease concentration was significantly lower in the control sample compared to the contaminated samples. Result obtained showed that POME is

acidic and contains residual oil that cannot be easily separated which is evident in the contaminated sample having higher values than the control samples. However, the regulatory discharge limit of oil and grease in POME is 50 mg/l [5] though Jones [20] recommended a lower value of 10 mg/l as the limit of oil and grease from vegetable oil processing effluent. From the data presented in Tables 1-3, it is clear that oil and grease are higher in POME contaminated soil than at the control sites across the three sites.

4. CONCLUSION

The major objective of this study was to shed more light on the potentials of POME as a nutrient or pollutant. Past studies have shown that POME is considered as non-toxic because it contains substantial amounts of Na, P, K, and Ca which are vital nutrient elements for plant growth. The POME contaminated site was observed to be bare without vegetation while the non-POME site was grown with weeds. Results obtained from the physicochemical analysis showed that soil samples from the POME contaminated sites contained a high amount of organic matter. Additionally, high moisture content in soil samples from the oil mills can be attributed to high concentration of organic matter especially from palm oil mill effluents which may help to retain water in the soil. Therefore the study suggests future research towards converting POME and other oil palm processing waste to meaningful organic materials.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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