

Bio-treatment of oily sludge: The contribution of amendment material to the content of target contaminants, and the biodegradation dynamics

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Abstract

The objective was to investigate the aerobic biodegradation of oily sludge generated by a flotation–flocculation unit (FFU) of an oil refinery wastewater treatment plant. Four 1 m³ pilot bioreactors with controlled air-flow were filled with FFU sludge mixed with one of the following amendments: sand (M1); matured oil compost (M2); kitchen waste compost (M3) and shredded waste wood (M4). The variables monitored were: pH, total petroleum hydrocarbons (TPHs), polycyclic aromatic hydrocarbons (PAHs), total carbon (C_{tot}), total nitrogen (N_{tot}) and total phosphorus (P_{tot}). The reduction of TPH based on mass balance in M1, M2, M3 and M4 after 373 days of treatment was 62, 51, 74 and 49%; the reduction of PAHs was 97%, +13% (increase), 92 and 88%, respectively. The following mechanisms alone or in combination might explain the results: (i) most organics added with amendments biodegrade faster than most petroleum hydrocarbons, resulting in a relative increase in concentration of these recalcitrant contaminants; (ii) some amendments result in increased amounts of TPH and PAHs to be degraded in the mixture; (iii) sorption–desorption mechanisms involving hydrophobic compounds in the organic matrix reduce bioavailability, biodegradability and eventually extractability; (iv) mixture heterogeneity affecting sampling. Total contaminant mass reduction seems to be a better parameter than concentration to assess degradation efficiency in mixtures with high content of biodegradable amendments.

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1. Introduction

Oily water from various refining processes, as well as surface water run-off is pre-treated in oil separators. Solids settle down by gravity to form oily sludge. Settling properties of light fractions are improved by using flocculants and by blowing fine air bubbles into the treated water in flotation–flocculation units (FFU), where oil droplets are brought to the surface and skimmed off. FFU sludge contains various fractions of petroleum hydrocarbons (typically 10–15% wt.), solids (6–10% wt.) and water, and is classified as hazardous waste.

Most petroleum hydrocarbons are considered biodegradable [1,2]. Biodegradation of organic matter is achieved by assisting the microbial growth and by creating optimum environmental conditions for them to degrade the contaminants into carbon

dioxide and other gases, inorganic substances, water, and to produce microorganism biomass [1,3]. Biodegradation of oily sludge has been carried out through landfarming and composting [4–6], and using slurry-phase bioreactors [7–10]. Biopile technology involves heaping contaminated masses and amendments into piles or cells and stimulating aerobic microbial activity by up-flow pneumatic aeration [11]. Aerobic biodegradation in static forced air biopile is relatively simple, and offers better control over the process compared to windrow composting. General guidelines for biopile technology recommend that concentration levels of total petroleum hydrocarbons (TPHs) should not exceed 50,000 mg kg⁻¹ DM and toxic metal concentration should be below 2500 mg kg⁻¹ DM [11]. Since the masses are not remixed during the process, the mixture should be as homogeneous as possible before placing it into biopiles.

Due to the high water content and complex composition of oily sludge, composting matrixes require large amounts of amendments to reduce moisture. In composting oil sludge or oil-contaminated soil, different types of bulking material have

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been used with the purpose of adsorbing moisture, enlarging the porosity of the compost mixture, and supporting biological processes. The amount and combination of amendments, as well as the incubation time significantly influence bacterial growth and surface tension [12]. Optimal porosity of 30–35% [13] can be achieved by using inexpensive bulking agents, such as wood chips, sawdust, peat or bark [4,14–16]. Addition of organic matter has been found to enhance the degradation of PAHs during bioremediation of contaminated soils [4,15–21]. Organic matter initially absorbs excessive moisture [13] and release moisture during the composting process, thus compensating the moisture loss to some extent, which occurs in the case of forced aeration [22]. Fresh or partially degraded kitchen compost serves as a thermal source by promoting thermophilic biodegradation. Due to microbial populations organic amendments are also useful for augmentation [22] and it helps to adjust the C:N:P ratio to the targeted 300–100:10:1 [11,13]. Organic material degrades along with target organic compounds, and has been found to interfere with degradation results, thus complicating understanding the treatment efficiency [23].

One major concern related to compost mixtures is that if the attempted biological treatment proves to be unsuccessful, mixing non-contaminated amendments to contaminated material generates a far greater quantity of contaminated masses [15]. Eventually, some bulking agents can be recycled and used in subsequent runs, which decreases waste production and provides acclimated microbial inoculate for subsequent bio-treatment of contaminated masses.

In this paper, the treatability of FFU sludge mixed with four different amendments is compared in four 1 m³ pilot bioreactors. The degradation rate of TPH and 16 US EPA priority PAHs was targeted during a relatively long (one year) monitored treatment. Special attention was given to the effect of different amendments on the TPH and PAH concentration reduction in relation to total dry matter – DM (mg kg⁻¹) versus mass reduction (remaining mass compared to the initial mass of the contaminant).

2. Material and methods

2.1. Pilot reactors

Four 1 m³ polyethylene containers were adapted to work as bioreactors and simulate static forced air biopiles. A slotted ventilation tube (Ø 100 mm) was placed onto the bottom of each container. Airflow of 30 m³ min⁻¹ was equally divided among the four reactors. The flow was adjusted according to the oxygen content of the mixture in the reactor M3. Oxygen content and temperature were measured hourly using 1-m long probes (Umwelt Elektronik GmbH & Co). The containers were thermally insulated by heaping shredded waste wood around them.

2.2. Sampling

The treatment was monitored during about one-year (from Nov 2003 to Nov 2004). At day zero, random composite samples consisting of ten sub-samples taken from each amendment

and mixture were analysed. At days 104, 183, 278 and 373, samples with about 500 g of each mixture, covering the whole profile, were collected from the centre of each container, using a 100 mm diameter tube drill. Composite samples were carefully mixed to achieve maximum homogenisation. The samples were stored at +5 °C until delivered to the laboratory within 2–3 days. In the present investigation, large particles in each sample were finely shredded before analysis to guarantee that even TPH and PAH not easily available for biodegradation were included in the analyses. The characterization of sludge, amendments and mixtures based on physical and chemical variables was conducted by ALcontrol AB, Sweden, according to standard methods (Swedish Board for Accreditation and Conformity Assessment, SWEDAC). The specific surface of the amendments was determined in three replicates according to standard methods [24].

2.3. Analytical procedure – TPH and PAH analyses

For extraction of TPH, 10 g of the wet homogenized sample was agitated during 16 h with 40 ml acetone/*n*-hexane (60:40), spiked with *n*-decane as internal standard. To the mixture sample and acetone/*n*-hexane, 20 ml of a 4% NaCl-solution was added and the resulting *n*-hexane phase was taken care of. The volume obtained was reduced in a Zymark Turbo Vap # 2 and the final volume was adjusted to 1000 µl. The hydrocarbons were analyzed by GC/FID. The TPH extracts were analyzed using a HP 6890 gas chromatograph equipped with flame ionization detector, an auto injector using the split mode and a DB5MS capillary column (30 m, 0.25 mm, 0.25 µm) and helium as carrier gas. The temperature program was: 60 °C for 2 min, 15 °C/min to 330 °C, hold for 10 min. The total area between *n*-C10 and *n*-C40 was integrated and normalized to the internal standard for the quantification.

For extraction of PAHs, a wet homogenized sample (5 g) was agitated during 2 h in a glass bottle with 20 ml of acetone and cyclohexane (60:40), spiked with d₁₀-pyrene and d₁₂-perylene as internal standard. After extraction, 20 ml of distilled water was added and the mixture was centrifuged at 2000 rpm during 5 min. From the *c*-hexane phase, 1 ml was taken for the GC/MS analysis. The PAH extracts were analyzed with GC/MS using a HP 6890 gas chromatograph equipped with a HP 5973 mass spectrometer running in the SIM mode, a PTV injector and a DB5MS capillary column (30 m, 0.25 mm, 0.25 µm) having Helium as carrier gas. The temperature program was: 60 °C for 2 min, 10 °C/min to 180 °C, 15 °C/min to 300 °C, 30 °C/min to 340 °C, hold for 3 min.

2.4. Characterization of FFU sludge and amendments

FFU sludge generated by Shell AB refinery in Gothenburg, Sweden, was mixed with four low-cost amendments. The properties of FFU sludge have been previously described [25,26], indicating the presence of two main dominant bacterial species (based on denaturing gel electrophoresis of 16S rDNA fragments) as an important characteristic, which suggests that no further inoculation is needed.

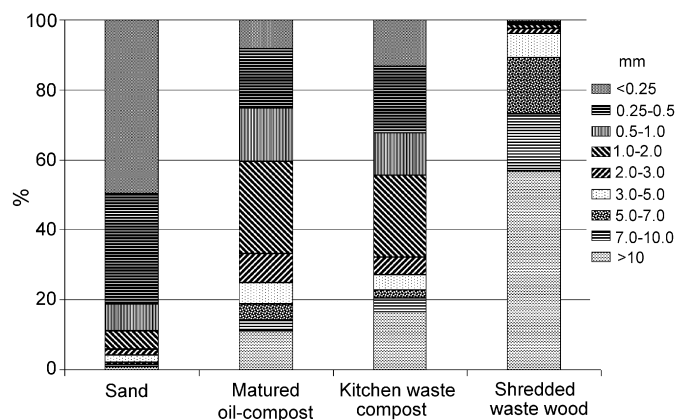


Fig. 1. Particle size distribution in the amendments: sand (used in mixture M1), matured oil-compost (M2); kitchen waste compost (M3) and shredded waste wood (M4).

The physical properties of the amendments varied largely, as illustrated by the particles size distribution in Fig. 1. Sand contained 88% of particles <1 mm. Mature oil-compost and kitchen waste compost both contained 40% of fine particles <1 mm, 30–35% particles with 1–3 mm, and 25% particles >3 mm. The shredded waste wood had coarse nature, containing 60% of coarse particles >10 mm, 34% of particles with 5–10 mm, and only 10% of fine particles <5 mm. The characterization of the FFU sludge and amendments used for preparing the mixtures is shown in Table 1. It was found that not only the FFU sludge but also the amendments contributed to the total content of TPH and PAHs, where sand (amendment in M1) contributed the least.

2.5. Preparation of mixtures

The mixtures were prepared as following: M1 received a mixture of sand and FFU sludge (6:1 ratio); M2 mature oil compost and FFU sludge (4:1 ratio); M3 kitchen waste compost and FFU sludge (3:1 ratio); and M4 shredded waste wood and FFU sludge

(3:1 ratio) in kg kg^{-1} (wet wt.). To adjust the C:N:P ratio to values close to 100:10:1, 1 kg of Hydro Agri AB super phosphate P20 (containing 20.5% of water and acid soluble P; 17.9% of water soluble P; and 1.9% S) and 12.5 kg of Hydro Agri AB Calcinit 15–0–0 (14.4% nitrate; 1.1% ammonium; 19.0% elemental calcium; and 26.5% CaO) were added to mixtures M1, M2 and M4. The FFU sludge and amendments were carefully mixed with the help of a front-end loader. Mixing was not stopped until a visual inspection indicated the best mixture was achieved. Each mixture was weighed before and after the experiment. Based on moisture monitored values, from time to time, water was manually added to balance the moisture content of the mixtures.

3. Results and discussion

3.1. Initial composition of mixtures

Table 2 shows the composition of mixtures with samples collected just after mixing FFU with the amendments. The measured concentrations of different variables (e.g. TPH, PAHs, C, N, P) in the mixture slightly differed from the expected concentrations based on the FFU sludge to amendment ratio in each mixture (Table 2). This could be attributed to the heterogeneity of the amendments themselves and/or incomplete mixing. The initial amounts of total TPH and total PAHs in M4 were substantially higher than in all other mixtures.

3.2. Monitoring parameters

The temperature monitored during the first nine months (Day 0–264) is shown in (Fig. 2) In the beginning of the experiment, the temperature in all mixtures slightly increased above ambient temperature, indicating the start-up of microbial activities, regardless the low ambient temperatures and cooling due to frequent aeration with cold air. The ventilator was controlled by the oxygen meter and it was switched on every time the oxygen

Table 1
Initial composition and characteristics of FFU sludge and amendments (day 0)

Variable	Unit	FFU sludge	Sand	Matured oil compost	Kitchen waste compost	Shredded waste wood
pH		8.0	6.6	7.8	7.8	5.7
Dry matter, DM	%	9.0	96.5	62.7	44.1	74.3
TPH	mg kg^{-1} DM	130,000	11	2400	4300	4400
Sum of carcin. PAHs	mg kg^{-1} DM	14	<0.3	5.3	0.74	4.4
Sum of other PAHs ^a	mg kg^{-1} DM	120	<20	<20	<20	51
Total carbon C_{tot}	% DM	26.5	<0.3	14.8	31.2	50.7
Total nitrogen N_{tot}	% DM	1.6	<0.3	1.3	2.9	0.7
Total phosphorus P_{tot}	g kg^{-1} DM	1.8	0.32	2.3	6.7	0.13
Specific surface	$\text{m}^2 \text{g}^{-1}$	n.d.	26	224	313	337
Structure ^b	–	n.d.	Fine	Fine-medium	Fine-medium	Coarse
TPH-PAHs added	–	Yes	No	Yes	Yes	Yes
Augmentation ^c	–	Yes	No	Yes	Yes	No
Moist. holding capac. ^d	–	^e	Low	High	High	Low

^a Meaning sum of 16 USEPA priority PAHs minus sum of carcinogenic PAHs among them.

^b Based grain size evaluation (details in Fig. 1).

^c Previously stimulation of microorganisms' growth, meaning augmentation to the mixture.

^d Based on visual inspection.

^e Chemically conditioned by flocculants; n.d.: not determined.

Table 2
Initial composition (day 0) of mixtures: FFU sludge + indicated amendment

	Mixture: Amendment:	M1 Sand	M2 Matured oil compost	M3 Kitchen waste compost	M4 Shredded waste wood
Sludge: amendment ratio	kg kg ⁻¹ wet wt.	6:1	4:1	3:1	3:1
pH		8.1	7.8	7.4	7.5
Dry matter, DM	%	77.6	50.3	43.8	52.2
TPH	mg kg ⁻¹ DM	3700	9900	13,000	22,000
Sum of carcinog. PAHs	mg kg ⁻¹ DM	0.51	6.7	4.6	7.6
Sum other PAHs ^a	mg kg ⁻¹ DM	<20	<20	<20	35
C _{tot}	% DM	1.1	16.6	26.6	29.2
N _{tot}	% DM	<0.3	1.8	2.6	1.9
P _{tot}	g kg ⁻¹ DM	0.67	3.2	2.7	4.8
C:N:P ratio ^a		100:14:6	100:11:2	100:10:1	100:7:2
C:N:P adjustments		Yes	Yes	No	Yes

^a Recommended ratio 300–100:10:1.

level in the compost fell under 82% of the oxygen level in the ambient; it switched off when the level exceeded 85% the ambient level. The average ambient temperature during winter and early spring (Day 0–180) was 8.3 °C. During spring–summer (Day 180–250) the temperature in all mixtures followed the air temperature pattern, remaining slightly cooler than the air.

During seven watering events, 215 l of water were added manually by pouring it on surface of M1, M2 and M3 and 330 l were added to M4. It was observed that M1 and M4 dried faster than M2 and M3. Through the experiment, the pH in all mixtures remained within the range 7.2–8.2.

3.3. Mass balance and TPH and PAHs biodegradation dynamics

Table 3 shows the mass balance in all mixtures. The initial error during preparation of mixtures was estimated to be ±20 kg, and in the end of the experiment, ±2.0 kg.

The biggest wet weight loss, dry matter loss as well as settling were observed in mixture M3, where non-mature kitchen waste compost with highly biodegradable organics was used as amendment. M2 and M4 had similar weight losses and compaction. M1 settled basically due to compaction of sand. The initial and final concentrations of TPH (in mg kg⁻¹ DM) differed largely among the mixtures (Table 3). The final mass reduction/loss of TPH (Table 3) was 62, 51, 74 and 49%, respectively. These values are

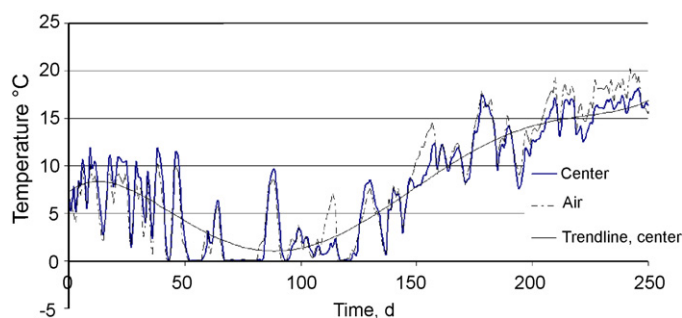


Fig. 2. Temperature profile in M3 (FFU sludge + kitchen waste compost): day 0 to day 250.

higher than the reduction based on concentration in mg kg⁻¹ DM (59, 38, 51 and 18%, respectively). As expected, higher the organic content in the mixture, bigger the difference, due to faster degradation of easier biodegradable organics, which increases the relative concentration of the target recalcitrant contaminants.

In order to estimate the degradation of TPH against time, the TPH concentration (mg kg⁻¹ DM) measured along the monitoring period in each reactor were divided by the initial TPH concentration TPH₀ (Fig. 3).

The fluctuations of total TPH values observed in M2, M3, and M4 is affected by: (i) different degradation rates for TPH compared to easily degradable organics, as described above; (ii) the release of compounds measured as TPH existing in the amendment material itself; (iii) adsorption–desorption mechanisms involving hydrocarbons and organic matrixes; and (iv) the heterogeneity of contaminated masses. All these mechanisms are dependent on organic matter content in the mixture and therefore, irrelevant in M1.

The % of degradation in time for each mixture based on concentration decay is shown in Fig. 4. In M1 (sand as amendment), decrease in TPH concentration was detected already at day 104. In this mixture, degradation occurred linearly in time and fitted to a linear equation which never gave negative degradation (meaning increase). With regard to M2 (oil compost) and M3

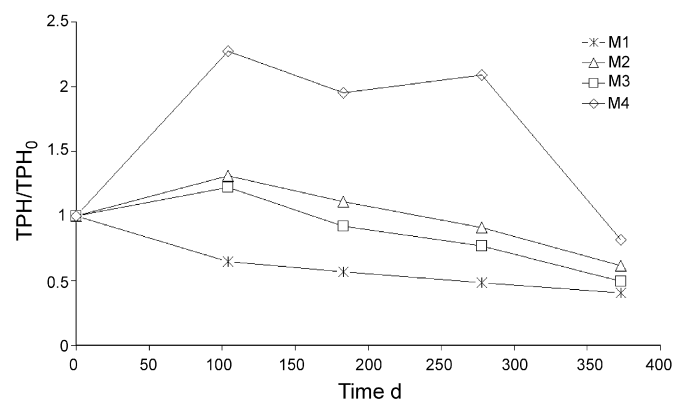


Fig. 3. TPH concentrations in mg kg⁻¹ DM divided to the initial concentration TPH₀.

Table 3
Mass balance in the mixtures: initial (day 0) and final (day 373)

	M1		M2		M3		M4	
	Initial	Final	Initial	Final	Initial	Final	Initial	Final
Wet weight (kg)	1293	1047	801	531	613	281	517	339
Moisture content (kg)	290	105	398	213	345	139	247	170
Dry matter content (kg)	1003	942	403	318	269	142	270	169
Wet weight loss (%)	19		34		54		34	
DM (dry matter) loss (%)	6		21		47		37	
Compaction (%)	19		14		33		16	
TPH (mg kg ⁻¹ DM)	3700	1500	9900	6100	13,000	6400	22,000	18,000
TPH (kg)	3.71	1.41	3.99	1.94	3.49	0.91	5.93	3.04
TPH mass loss (%DM)	62		51		74		49	
PAHs (g)	10.55	0.33	6.73	7.63	3.92	0.30	11.49	1.42
PAHs mass loss (%DM)	97		13		92		88	
C _{tot} (kg)	11.04	10.37	66.91	39.76	71.45	32.48	78.73	28.70
C _{tot} mass loss (%DM)	6		41		55		64	
N _{tot} (kg)	1.51	1.41	7.25	5.09	6.98	3.70	5.12	2.53
P _{tot} (kg)	0.67	0.55	1.29	1.08	0.73	0.54	1.29	0.76

(non-mature kitchen waste compost), the fitted curve crossed the x-axis, meaning that in the beginning of the process the readily biodegradable organics were oxidized into CO₂ and H₂O in a faster degradation rate than TPH compounds. However, after 225 days for M2, and 180 days for M3, the TPH was below the initial concentration and degradation rate was even faster than the one observed in M1. Regarding M4, two curves were fitted to the data points. One included all sampling points, and the other excluded the point corresponding to day 278, considered as an outlier. Based on either curve, organic compounds in shredded wood waste are likely to be degraded faster than TPH and/or wood waste is likely to release compounds measured as TPH before the second sampling at day 104. The fitted dashed line in Fig. 4 shows that close to day 350, degradation resulted in a TPH concentration below the initial concentration; when the outlier is included, this was reached around day 400. According to van Gestel et al. [14], adsorption of oil onto large particles has found to be negligible, thus large wood chips could be removed during sampling preparation. In the present study, however it

was found that mixing FFU sludge with amendments resulted in covering wood chips with a sludge film, which was not possible to separate before analytical procedures by sieving.

Regarding the dynamic of PAHs (Fig. 5), a linear decrease in concentration (mg kg⁻¹ DM) from day 104 to day 373 fitted to M1, M3 and M4. The final reductions in these mixtures were in the same range (88, 79 and 80%, respectively). As observed for TPH, in M4 an initial increase of total PAH was registered at day 104. The reduction of total PAHs in M1 was faster than in the other mixtures and 83% of reduction was already observed at day 183. As discussed previously for TPH, the degradation of organics in the amendments in all mixtures but M1 and slower degradation of the target contaminants affects the contaminant concentration, which was confirmed by analysis of each individual PAH (not shown here) revealed that fluctuations in M2 were mostly due to 5–6 ring PAHs, the most persistent PAH compounds. The data also suggests that the remediated oil-contaminated soil (matured oil compost) added to M2 was not stabilized yet. The 5–6 ring PAHs in M3 and M4 also fluctuated,

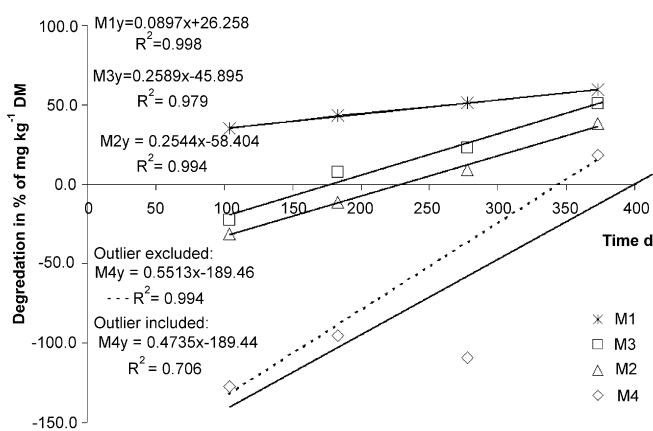


Fig. 4. Reduction (in %) of TPH compared to the initial concentration in mixtures M1, M2, M3 and M4 (negative values means increase in % compared to the initial value).

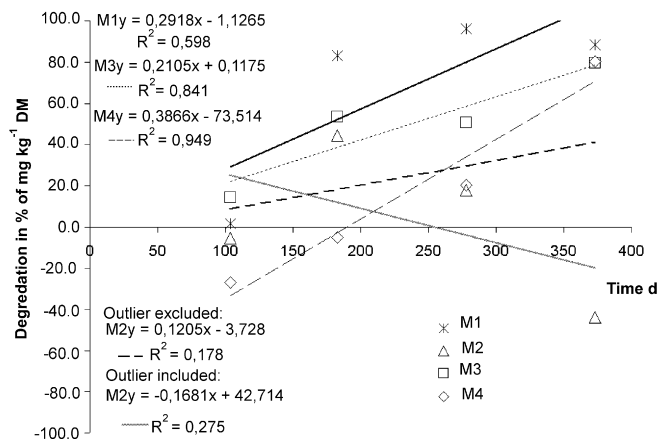


Fig. 5. Reduction (in %) of total PAHs compared to the initial concentration in mixtures M1, M2, M3 and M4 (negative values mean increase compared to the initial value).

suggesting a slight relative increase trend during the studied period. M1 (sand as amendment) was the only mixture where 5–6 ring PAHs decreased consistently with 2, 3 and 4-ring PAH decrease.

A combination of different mechanisms might play an important role to explain the results, such as: (i) not only the oily sludge, but also amendments in M2, M3 and M4 might release compounds measured as TPH and PAHs as observed in the analyses of amendments; (ii) composting of organic matter frequently generates aliphatic hydrocarbons of microbiological origin during the process [27], which was probably the case for M2, M3 and M4, but not M1; (iii) since the target compounds have degradation rates slower than the biodegradable organics added with amendments, the relative concentration of TPH and PAHs is affected by the reduction of the dry mass content, transformed in CO₂ and H₂O. Similar phenomenon has been described for heavy metals [28] remaining in composting piles meanwhile the organics degrade; (iv) the slower is the degradation of the organic material (e.g. wood waste compared to kitchen waste), the longer is the time it takes to start reducing TPH and PAHs concentrations; (v) there is a close relationship between asphaltenes found in crude oil/oily sludge and increase of PAHs. In nature, asphaltenes are hydrogenated into high molecular weight polycyclic aromatic hydrocarbons. They differ, however, from polycyclic aromatic hydrocarbons by the presence of oxygen and sulphur in varied amounts [29]; (vi) high heterogeneity of contaminated masses and sampling preparation needs to be properly addressed, since the results depend upon which portion of the mixture is analysed. In the present investigation, large particles in each sample were finely shredded before analysis. This might increase the content of certain compounds in the compost mixture, which were not affected by the biodegradation process.

4. Conclusions

Oily sludge with high water content can be aerobically composted in biopiles or solid phase bioreactors if mixed with large amounts of amendments that reduce moisture and promote biodegradation. Organic amendments have been said to bring additional benefits, but since their organic content is degraded along with target contaminants in oily sludge, optimal sludge-to-amendment ratio is needed to ensure that the degradation of target contaminants in FFU sludge is achieved. The properties of the amendment material must also be carefully determined before setting up the bio-treatment, since their contribution to the final content of target contaminants must be accounted. When inert inorganic amendments are selected, the benefits added by organic amendments are lost and when microbial community already exist in the contaminated medium (as the case of FFU sludge), careful adjustments in C:N:P ratio might be enough to optimize conditions for microbial metabolism; a positive aspect is that degradation of the target contaminants in oily sludge is explicitly and equally monitored by either mass loss or concentration reduction, since the inert material does not degrade and usually does not bring additional mass of target contaminant. However, when large amounts of biodegradable amendments

are used, calculation of remaining total mass of target contaminant along the bio-treatment period seems to be more accurate way to estimate the degradation rate than concentration reduction – which is the indicator usually used. Further investigations are needed to promote homogeneity or improve sampling representativeness when using organic amendments, since they are often highly heterogeneous by nature.

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