Desalination and Water Treatment
www.deswater.com
1944-3994 / 1944-3986
doi: 10.5004/dwt.2011.2621

33 (2011) 194–201
September

Biotreatment of oil refinery sludge

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Received 20 January 2010; Accepted in revised form 12 January 2011

ABSTRACT

Utilization of waste materials generated in different industries is important from the points of ecology, economics and conservation of non-renewable resources. One of the main waste materials in petroleum industry is oil sludge, which is produced by the treatment of wastewater, from various refining and tank cleaning processes. Biotreatment or otherwise biodegradation by natural populations of microorganisms is one of the primary mechanisms by which petroleum and other hydrocarbon pollutants can be eliminated from the environment. The biotreatment technology by biopiles, which is applied in Thessaloniki Refinery area of Hellenic Petroleum S.A., for the case of the solid residual material (SRM) (dewatered oil sludge) derived from oil refinery sludge, is presented in this study. Experimental results presented herein demonstrate the fact that the biotreatment method of biopiles in the case of solid residual material stemming from oil refinery sludge can achieve high biodegradation efficiencies for organic substances and low leaching levels for heavy metals. Conclusively, Thessaloniki refinery's biotreatment unit is proven to be one effective and environmentally sustainable treatment option for the oil refinery sludge and its solid residual material, saving valuable natural resources, i.e. non impacted natural soil, which can be used mainly in earthworks or as cover material in landfill sites.

Keywords: Oil sludge; Refinery; Biodegradation; Biopiles; Leaching

1. Introduction

The petrochemical industry generates a series of liquid effluents during petroleum refining process. These effluents must be treated through mechanical and biological processes prior to their final disposal. The oil refinery sludges that result from these processes have a high content of petroleum-derived hydrocarbons; thus it is potentially dangerous waste product, according to EU legislation. Simply disposing these wastes or burning them without any treatment has serious environmental consequences and presents a risk to both ecosystems and human health [1].

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Presented at the 2nd International Conference on Environmental Management, Engineering, Planning and Economics (CEMEPE), Mykonos, Greece, June 21–26, 2009.

Biotreatment or otherwise biodegradation by natural populations of microorganisms represents one of the primary mechanisms by which petroleum and other hydrocarbons pollutants can be eliminated from the environment [2–6]. Biodegradation of organic materials is achieved by assisting the microbial growth and by creating optimum environmental conditions for them to degrade the contaminants mainly into carbon dioxide, inorganic substances, water and to produce microorganisms' biomass. Biotreatment technologies can be broadly classified as ex situ and in situ. Ex situ technologies are those treatments which involve the physical removal of the contaminated material for treatment process. In contrast, in situ techniques involve treatment of the contaminated material in place. Ex situ biotreatment technologies include, among others, biopiles technique, which involves heaping contaminates masses and amendments into piles or cells and stimulating aerobic microbial activity by down-flow pneumatic aeration [7,8].

Biopiles technique [9,10] is considered as one of the most effective biotreatment methods, which is developed to reduce high concentrations of contaminants in sludges to acceptable levels in reasonable time frames and at low cost [11]. Indicative examples of biotreatment techniques that have been demonstrated to function in field pilot or full scale especially for petroleum hydrocarbons are those of:

- refinery sludge treated in Murcia, an north-eastern area of Spain [1], where bioremediation of a refinery sludge containing hydrocarbons in a semi-arid climate is described and the results showed that 80% of the hydrocarbons were eliminated in eleven months, while half of this reduction taking place during the first three months.
- sludge from Duque de Caxias Refinery in Rio de Janeiro, Brazil [12], where biotreatment of oil sludge was taken place and high organic matter biodegradation, i.e. 89% consumption of oil and grease, 99% consumption of n-paraffins and 83% consumption of total polyaromatics, was achieved.
- sludge from Indian Oil Corporation Limited refinery, in Panitat, India [13], where bioremediation of oil sludge was carried out using a selected bacterial consortium that resulted in 97% degradation of the oil content of the oil sludge in a time period of 3–5 months.

The objective of this study is to present the process characteristics of the biopiles technique for the case of oil refinery sludge from the Thessaloniki refinery of Hellenic Petroleum S.A. along with the biodegradation results for the petroleum waste material. It must be mentioned that technical characteristics and applicability of biopiles technique for oil refinery sludge is presented in a limited number of scientific articles, especially for areas located in southern part of Europe, where weather conditions are considered as favorable for this kind of bioremediation processes.

2. Materials and methods

2.1. Oil refinery sludge

The sources of the oil refinery sludge that was studied here are:

- Tank bottom sludges (European list of wastes – ELW 05 01 03*)
- Sludges from on-site effluent treatment (ELW 05 01 09*)
- Oily sludges from maintenance operations of the equipment of the plant (ELW 05 01 06*)

The above mentioned oil sludges are selected and stored in three tanks with total capacity of 1000 m^3. The sludges are submitted to a heating and centrifugation process that separate three phases, namely:

- *Wastewater*, which goes to the wastewater treatment unit
- *Oil*, that returns as input to the refinery
- *Solid* residual material, which goes to the biotreatment unit

The amount of the sludge treated in the centrifugation unit was 7,722 m^3 for 2007 and after centrifugation this amount was reduced to 262 m^3, i.e. 96.6 % volume reduction. By this process, a semisolid consistency is given in the sludge material. The dry oil sludge, referred as solid residual material (SRM), is produced after the centrifugation process. The principal chemical – characteristics of SRM (yearly average values of three different periods from 2004 to 2008) used in this study are summarized in Table 1.

With respect to the source of the sludge treated and its characteristics, e.g. concentration of hydrocarbons and moisture content, preheating and addition of polyelectroletes [14] are applied in order to enhance sludge dewatering and decrease further moisture content (target value for the moisture content is between 20–30% weight), compared to conventional centrifugations methods. Moisture content has to be fully controlled during biotreatment close to target values in order to assist natural microorganisms to degrade the contaminants. Weather conditions, i.e. rainfalls and temperature, in wider area of Thessaloniki results in moisture within the desired range of values and only during summer, an additional amount of water may be required.

In addition, samples of raw SRM are analyzed according to EN ISO 12457 leaching test procedure [16]. Analysis results of raw SRM show that only DOC concentration is above the limits of 800 mg/kg and 500 mg/kg referred to the Decision 2003/33/EC, concerning disposal as non hazardous material or inert material, repsectively [17]. As a result, DOC is selected as the main chemical parameter

Table 1
SRM characteristics (after centrifugation and prior to biotreatment)

Metals (mg/kg dry weight)											
Cd	Cu	Ni	Pb	Zn	Hg	Cr	As	Se	Ba	Sb	Mo
0.97	286.7	496.7	183.3	1226.7	44.7	123.3	31.7	8	238.3	5.8	25
Moisture* (% w/w)			22–45	Hydrocarbons** (%)			4–34	Solids*** (%)			55–78

*Standard method 2540B [15], **Standard method 5520D. Results in % weight of dry sample [15],
***Standard method 2540G. Results in % weight of wet sample [15]

of concern and the main criterion for the final conclusion drawn about the applicability of biotreatment method.

2.2. Site, waste material preparation and biotreatment process

A site of 3,000 m^2 is used for the application of biopiles technique in the case of refinery waste material. This site is designed for the case of Thessaloniki refinery, assuming average production of oil sludge equal to 6 ton/d and average duration of bioremediation from 1.5 month to 2 months.

SRM properties that are important in influencing the rate and the extent of the biodegradation process include texture and particle size distribution, that are directly affect the density and moisture content of the material. According to the pilot study's results during the initial design of the process, a pre-treatment of the dry oily sludge is required prior to the disposal in the biotreatment cells. Pre-treatment option includes the addition of biocatalytic liquids, stabilizing agents and nutrient formulations, e.g. aqueous solutions of potassium phosphate and nitrate, in the SRM, during a mixing procedure.

Biocatalytic liquids [18] are oxygen release redox compounds and applied for the stimulation of aerobic biodegradation through controlled-release oxygen and nutrient delivery. This kind of materials is designed specifically for the in-situ and on site treatment of petroleum-based hydrocarbon contamination or any aerobically degradable substance. Usually it is a fine, powdery material that is typically mixed with water and pressure injected into the subsurface or sprayed on the contaminated material. Once hydrated, the material releases up to 10% of its weight as molecular oxygen. This release of oxygen is governed by specialized ORC chemistry which allows for a gradual, controlled release of oxygen over periods of up to 12 months. The available oxygen is then utilized by indigenous microbial populations to naturally degrade contaminants into harmless end products like CO_2 and water.

On the other hand, stabilizing agents [19–22] refer to techniques that chemically reduce the hazard potential of a waste by converting the contaminants into less soluble, mobile, or toxic forms. The physical nature and handling characteristics of the waste are not necessarily changed by stabilization. Stabilizing agents commonly used include Portland cement, cement kiln dust (CKD), lime, lime kiln dust (LKD), limestone, fly ash, slag, gypsum, bentonite and phosphate mixtures, and a number of proprietary reagents. Due to the great variation of waste constituents and media, a mix design should be conducted on each subject waste. At the specific site, in case the inorganic content, e.g. Cd, exceeds the limits of inert waste characterization then some additional stabilizing agents were applied as bentonite.

With respect to the moisture and total organic content of the waste material, a proper amount of fine or medium grained gravel is also added. The above mentioned pretreatment procedure results in significant increase of the effective porosity of the sludge and optimization of aeration conditions [7].

After the pre-treatment procedure, the dewatered oil sludge (SRM) is transferred in the biotreatment unit. Biotreatment process applied in Thessaloniki refinery is an engineered bioventing technique and it is technically described as biopiles technique. Biotreatment unit consists of totally twenty (20) independent cells, divided in two segments. These biotreatment cells are constructed above an artificial impermeable liner in order to achieve a hydraulic coefficient value of the basis lower than 1×10^{-9} m/s for the protection of soil and groundwater, in agreement with technical requirements for disposal in landfills. Oxygen supply is required and is provided by an extended soil-venting system, due to the fact that hydrocarbon biodegradation is primarily an aerobic process. This system consists of 20 independent units with blowers and activated carbon filters, which are connected with the biotreatment cells by means of an extended slotted pipeline network placed on the basis of each cell. Furthermore, the entire biotreatment system includes a dense drainage system that enables the collection of any wastewater, produced mainly from the runoff within the treatment area. The collected wastewater is re-injected into the biotreatment cells in periodic intervals in order to control the moisture of the material, especially in the summer period. Any excess of the collected waster water above the necessary amount for the moisture control is led

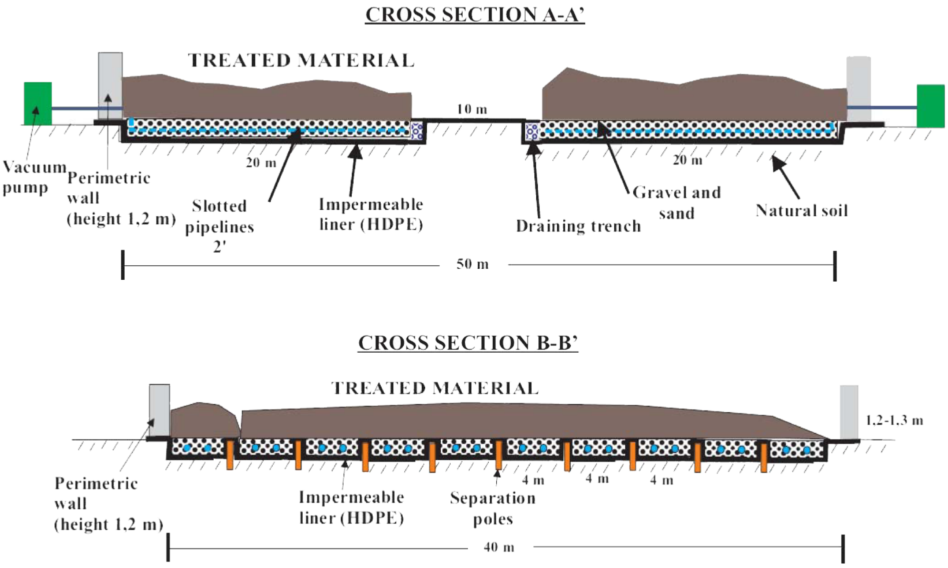

Fig. 1. Cross sections drawings of the bioremediation treatment area.

into the wastewater management system of the refinery. The biotreatment unit is presented graphically in Fig. 1.

2.3. Sampling and chemical analysis

Samples of treated SRM (Fig. 2) were collected periodically during previous years (several times in each month) from different cells of the biotreatment unit and are analyzed according to EN ISO 12457 leaching test procedure, which also includes measurement of TOC and DOC [16]. Sampling methodology is based on international standard of EPA SOP 2017 – Waste pile sampling [23] and Standard EN 14899 [24] of Greek organization for standardization.

During sampling, the operators use the hand auger to bore a hole to approximately 0.3 m above the desired sampling depth. Next, a slide hammer-type hand sampler, lined with brass sleeves (two 6-inch sections) is used to collect a core sample. Upon labelling the samples, they complete chain-of-custody form and they place the sample in a cooler chilled with artificial ice. Upon completion of sampling, they transport samples to a test lab.

Apart from the chemical analysis of the treated material after biotreatment, the groundwater and the subsoil of the biotreatment unit's area were periodically monitored with the help of sampling and drilling devices. In more details, a special sampling tube is used to collect soil gas samples for field measurements. The soil gas samples are obtained from the soil gas monitoring points. Soil gas can be either pumped into the sampling tube (or special bags) using an inert pump or pulled into it by connecting the bag directly to the sample line and then placing the bag in a portable vacuum chamber. The soil gas samples are analyzed on site using hand-held gas analyzers once a month for the presence of volatile organic compounds

Fig. 2. Solid residual material (SRM) prior (left) and after (right) bioremediation treatment.

(VOC) and other selected physicochemical parameters, i.e. conductivity, pH, temperature, free oxygen along with concentration of total petroleum hydrocarbons (TPH).

3. Results and discussion

3.1. Bioremediation results

Leaching test results are presented in Table 2 and they are compared to limit values proposed by Decision 2003/33/EC [17] on establishing criteria and procedures for the acceptance of waste at landfills pursuant to article 16 of and Annex II to Directive 1999/31/EC [25].

It is clearly demonstrated in Table 2 that the product of the biotreatment technique not only fulfils the criteria of acceptance of non hazardous waste materials at landfills, but also the parameters' values are lower than the acceptance criteria for inert materials, with the exception of upper range values for Cd and TDS. The slightly longer period of operation, the addition of new biocatalyst and stabilizing agents has resulted in faster biodegradation of organic materials as long as avoidance of inorganic salts leaching.

Furthermore, total organic carbon (TOC) and dissolved organic carbon (DOC) analysis was conducted for the case of the product of the biotreatment process. TOC value varied from 0.3% to 1.4%, which is lower than the limit of 5% referred to Decision 2003/33/EC [17]. In addition, DOC values vary from 45 mg/kg to 135 mg/kg, which are also lower than the limit of 500 mg/kg referred to the same Decision [17].

Table 3
Physicochemical parameters of groundwater

Parameter	Values
TPH, mg/l	0.15–2
pH	6.6–7.3
Temperature, °C	16.7–25.1
Oxygen, mg/l	7–8.1
Conductivity, ms/cm	2.99–3.83

Regarding analysis of VOC in the recovered air from soil (soil–gas), as part of the groundwater and the subsoil of the biotreatment unit's area, obtained results are constantly equal to zero values. Moreover, the analysis results of the selected physicochemical parameters of the groundwater are presented in Table 3. Obtained results show that groundwater condition is within the normal range of the wider area of Thessaloniki. The applied monitoring process in the soil and groundwater proves the proper installation and operation of the impermeable liner and guarantees the immediate detection of any possible failure in its condition.

3.1. Progress and duration of biotreatment process

The duration of the bioremediation process depends

Table 2
Leaching analysis results and waste limit values of Decision 2003/33/EC

		Inert materials	Non-hazardous waste materials	Product of biotreatment process
Parameter	Method of analysis	L/S = 10 l/kg (mg/kg dry)	L/S = 10 l/kg (mg/kg dry)	L/S = 10 l/kg (mg/kg dry)
Arsenic (As)	EN ISO 11969 D18	0.50	2	<0.001
Lead (Pb)	EN ISO 11885	0.50	10	0.06–0.17
Cadmium (Cd)	EN ISO 11885	0.04	1	<0.003–0.05
Chromium (Cr)	EN ISO 11885	0.50	10	<0.005
Copper (Cu)	EN ISO 11885	2.00	50	0.05–0.07
Nickel (Ni)	EN ISO 11885	0.40	10	0.04–0.08
Mercury (Hg)	NF T90-113	0.01	0.2	<0.001
Zinc (Zn)	EN ISO 11885	4.00	50	0.01–0,9
Barium (Ba)	EN ISO 11885	20.00	100	0.09–0.13
Molybdenum (Mo)	EN ISO 11885	0.50	10	<0.01
Antimon (Sb)	DIN 38405 D32	0.06	0.70	<0.01
Selenium (Se)	DIN 38405 D23-2	0.10	0.50	<0.01
Chloride (Cl^-)	EN ISO 10304-1 D19	800	15,000	140–410
Fluoride (F^-)	EN ISO 10304-1 D19	10	150	4–15
Sulphate $(SO_4)_2^-$	EN ISO 10304-1 D19	1,000	20,000	30–110
Total dissolved solids (TDS)	DIN 38409 H 1-2	4,000	60,000	4,520–7,860

strongly on the pollutant concentrations [26], the mechanical and geochemical characteristics of the SRM in the bioremediation area and the climatic conditions, i.e. temperature and moisture [7–9]. Based on the obtained biotreatment results, the average time that is required for the completion of the biodegradation procedure varies from 21 to 70 days. For example, during September of 2006, 3,060 ton of refinery sludge were treated in the unit and 43.3 days were required for the completion of the biodegradation procedure.

During year periods of high temperatures and average humidity in Greece, i.e. summer and autumn, the bioremediation process is completed more quickly than year periods of low temperatures and high humidity, i.e. winter and spring. This performance of the biotreatment process is due to the fact that the favourable conditions for the microorganisms are between 12 and 30% w/w, and between 25–30°C, for air humidity and ambient temperature, respectively. This performance is clearly demonstrated in Fig. 3.

In addition, a weak correlation is noted between the duration of the biotreatment process and the mass of sludge treated. This is explained by the fact that the best aeration conditions are achieved in the low thickness cells (< 0.40 m) in contrast to cells where the high amounts of sludge do not allow air to come into the main body of the waste material. This weak correlation between experimental data is presented in Fig. 4.

The main criterion that was selected for the final decision of the disposal or utilization of the biotreatment product is the DOC concentration, which is the main chemical parameter of concern. In Fig. 5, the progress of the biodegradation procedure is shown. DOC concentration, which was measured once a week during a summer period of time, was constantly decreasing to values significantly lower than the limits of the Decision 2003/33/EC for the disposal of inert materials. The progress of the biotreatment process is shown also in Fig. 6, where the concentrations of VOC, CO_2 and O_2 in gaseous emissions from a specific biotreatment cell were measured for a period of two winter months.

It is demonstrated in Fig. 6 that the progress of the bioremediation is a typical one for this kind of process. In more details, the volatile organic compounds in the recovered air from soil (soil-gas) are reduced while DOC values are also reduced according to leaching analysis results. Moreover, the increase of CO_2 concentration, the main product of biodegradation, indicate that the hydrocarbons are eliminated by natural microorganisms. This process is also shown by increase of the O_2 concentrations, which is not used by microorganisms for the biodegradation process.

It must be mentioned that the climate conditions in Greece, in contrast to other countries in central and northern Europe, contribute importantly to the fact that the required time for completion of biodegradation procedure keeps approximately constant during the time period of one year.

3.2. Evaluation of the bioremediation treatment method

According to analysis results of treated waste material as well as analysis of recovered air and groundwater, the effectiveness of bioremediation process for the treatment of oil refinery sludge is demonstrated in this work. It is shown that biodegradation by natural microorganisms is achieved and petroleum hydrocarbons can be eliminated by this kind of techniques. The only factor that must be careful controlled is temperature and humidity, which affect the progress, e.g. duration, DOC concentration, of the bioremediation process. Based on the obtained results, meteorological conditions in Greece and Thessaloniki seem to be the favorable ones for bioremediation process.

Regarding moisture content, it is noted that the contaminated solid material must contain enough moisture to encourage growth of the hydrocarbon degrading microorganisms, but not so much as to reduce soil permeability.

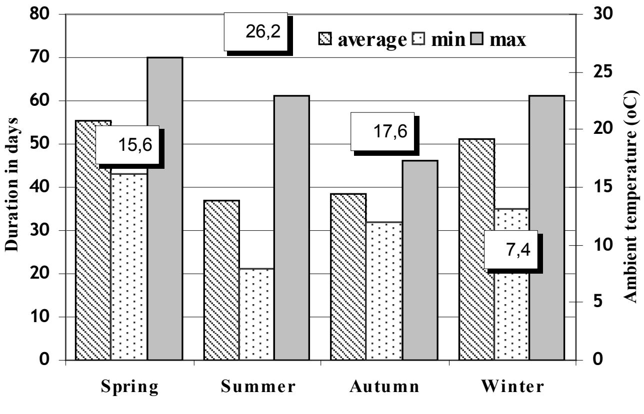

Fig. 3. Duration of bioremediation process vs. year periods (ambient temperature values in labels).

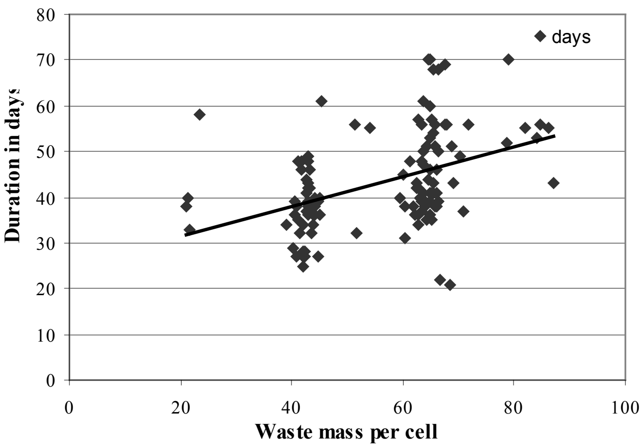

Fig. 4. Duration of bioremediation process vs. waste mass per cell.

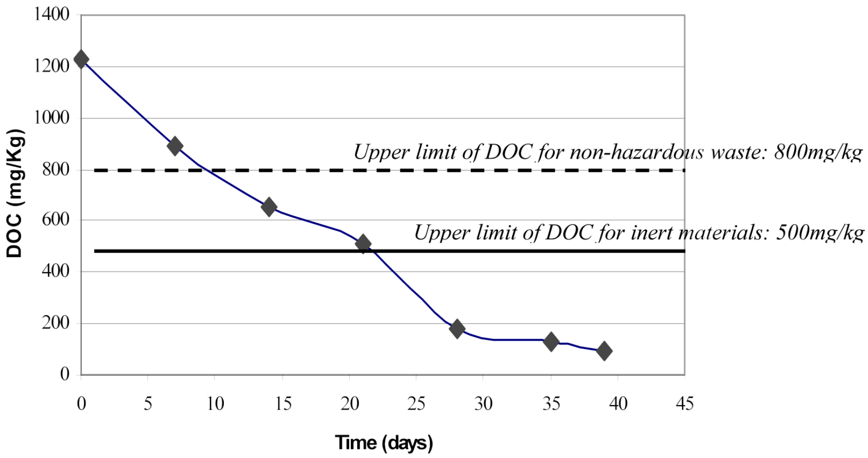

Fig. 5. DOC analysis results – dry base (biotreatment period 19/6/06–28/7/06).

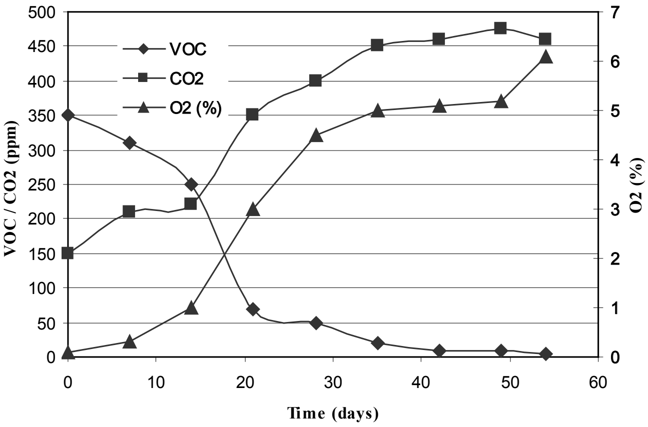

Fig. 6. Monitoring results of biotreatment process – gaseous emissions measurements (biotreatment period 05/01/07 –28/2/07).

Water is essential for biological processes because it not only provides the transport medium for the chemicals that supply energy and nutrients to the microorganisms but also enables the metabolic processes to proceed. However, excessive moisture will fill the pores in the soil pile and reduce soil permeability, making it difficult to aerate the biopile. That is the reason why the results did not show a strong correlation between the biodegradation rate and moisture content although a slight increase in biodegradation with increasing moisture was detected. Nevertheless, microorganisms will effectively degrade hydrocarbons over a wide range of moisture contents.

Concerning the quality of the final product of the bioremediation treatment process, this is controlled and certified in order to be disposed safely in an appropriate receptor area or to be used in earthworks or as cover material in landfill sites. This is clearly demonstrated by the results of chemical and leaching analysis tests presented in this study. Alternatively, product of bioremediation process can be used as a primary source for the cement production [27] or as constructing material [28–30].

In addition, wastewaters and gas emissions from the biotreatment unit are fully controlled by the application of wastewater treatment techniques and active carbon filters, respectively. However, experimental results show that no harmful concentrations of gaseous or liquid pollutants are produced. The final products of these techniques are either safely disposed in the environment, i.e. water and air substances, or thermally treated in waste to energy facilities, i.e. spent activated carbon filters.

4. Conclusions

Experimental results, which are presented in this study, demonstrate the fact that the biotreatment method of biopiles for the case of the Solid Residual Material derived from oil refinery sludge can achieve high biodegradation efficiencies for organic substances and low leaching levels for heavy metals. Thus, the utilization options for the treated SRM include various disposal or recovery methods, such as disposal in a landfill as non hazardous or inert material, use as cover material of municipal waste or use in various earthworks as backfill material. Other utilization options of this final solid material may include its usage as primary source for the cement production, which can be examined by the refinery based on the requirements of the cement industry and the material's chemical composition

Conclusively, comparing to other methods applied for the treatment of oil refinery oil sludge, e.g. incineration, biodegradation in biopiles seems to be a more simple and low cost alternative. Thessaloniki refinery's biotreatment unit is proven to be one effective and environmentally sustainable treatment option for the oil refinery sludge and its Solid Residual Material, saving valuable natural resources, i.e. non impacted natural soil, which can be used mainly in earthworks or as cover material in landfill sites.

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