

ASSESSING THE PERFORMANCE OF *IN-SITU* SOIL ENHANCEMENT TECHNOLOGIES TO IMPROVE GROUND WATER QUALITY

Muhammad Aftab Majeed¹

Abstract

Manufactured gas plants (MGP) produce different contaminants that when released into the environment cause significant soil and ground water pollution. *In situ* enhanced bioremediation techniques are effective in remediation of MGPs particularly for contaminants such as the dense non-aqueous phase liquids (DNAPL), like coal tar. The aim of this study was to assess the potential of *in situ* technologies in a laboratory column experiment. *In situ* amendments of nitrogen, phosphorus and oxygen releasing compound were performed on site. Soil samples were collected from a site and leached daily to equate a 20 years cycle. The biosensor *Escherichia coli* HB101 were used to assess the toxicity of soil leachate while standard chemical analysis was performed. The result of this study suggests that the presence of tar in the soil resulted in an increase in retention time of leachate and water chemistry over time. Nutrients release in the soil leachate was very low which reflected slow *in situ* biodegradation of the contaminants.

Key words: Leachate, Tar, DNAPL, Contaminant, MGP, Retention time.

Introduction

Manufactured gas plants (MGP) produce various toxic compounds and although each MGP site has its own characteristics there are commonalities. For example, halogenated organic solvents (trichloroethene (TCE) and 1,1,1-trichloroethane (TCA)) substituted aromatics, phthalates, monoaromatic and polycyclic hydrocarbons (PAHs), metals and cyanide compounds are common at MGP sites (Owen *et al.*, 1998). Most of these compounds are highly toxic and some are potentially carcinogenic (Mahjoub *et al.*, 2000)

Solid coal tar found in contaminated soils from MGP has been reported as aged coal tar (Bayard *et al.*, 2000). The more water soluble fractions can be slowly released from fresh tar into the subsurface water (Mahjoub *et al.*, 2000) and these compounds increase the density and viscosity of tar that ultimately turning into solid particles, containing high molecular weight PAHs. These solid tar particles have a significant effect on the retention of water soluble pollutants from the aqueous phase (Kopinke *et al.*, 1995). Durnat *et al.*, (1995) reported that naphthalene, phenanthrene and benzene are among the most prevalent constituents dissolving into ground water from the coal tars.

¹ World Bank Scholar, University of Aberdeen, Scotland.

Different technologies have been used to treat MGP wastes including excavation, *ex situ* treatment or *in situ* treatment technologies like ground water pump and treat, soil vapour extraction and DNAPL recovery. The application of these methods and their success largely depends upon the distribution of contaminants in soil, the geology and hydrology of the site. (Owen *et al.*, 1998).

In situ bioremediation is one of the technologies that have been used in MGPs to treat waste such as coal tar. *In situ* bioremediation technique can be used by involving number of methods. This remediation technique requires basic knowledge of the contaminants on the basis of which contaminants are treated.

This study aims to test the potential of enhanced (mixed techniques) *in situ* bioremediation to reduce the level of contaminants at an MGP and provide basic information to design effective bioremediation plan to treat the contaminants from a manufactured gas plant.

Materials and Methods

Soil microcosms

A laboratory experiment was set up with a batch of 30 leaching tubes with a diameter of 3 cm and 14 cm in length (Fig 2). At the base of the leaching tube glass wool was placed, 11.0 cm glass microfibre filter paper Whatman (GF/A Maidstone UK) equivalent to the diameter of leaching column was placed over the glass wool. Soil was added to the leaching column and glass beads were added over the soil sample. To optimise the experimental design a trail of five leaching tubes with 25 grams of soil (w/w) in each of the leaching columns was set up to record the average retention time. In the first test column soil was ground in mortar and pestle. One hundred and seventy ml of deionised water was added. No leaching observed in 24 hours. In the second test 25 grams of soil (w/w) from the same sample was placed in the leaching column without grinding in mortar and pestle in order to maintain the physical characteristics and integrity of soil sample. One hundred and seventy ml of water was added. Water leached down in 3 min. In the third trial 25 grams (w/w) of soil sample were added by partially mixing the soil for few seconds in the mortar and pestle. One hundred and seventy ml of water were added. No leaching observed for three hours. The test was repeated for five times. Different soil samples were tested with various ratios of sand and soil until the required leaching time was obtained.

Fourteen grams of soil sample (w/w) and 26 grams of sand (w/w) gave the assumed retention time of two hours. A mixture of benzene, naphthalene, pyrene and diesel were added to the microcosm at ratio of 10: 10: 10: 70 to a

total amendment concentration of 0.5 g/kg hydrocarbons. Soil samples were leached immediately after adding the hydrocarbons with 340 ml of deionised water in two runs. Leachate from all the six treatments and six replicates (for control four replicates) were collected on daily basis for 16 days and average retention time was recorded. The pH from leachate of first and second run was recorded daily for initial one week and from the second leachate only in the following weeks. 20 ml of leachate was stored in a glass vial for analysis. This 16 day period represented 20 years of rainfall.

Soil Analysis

Twenty ml of deionised water was added to 10 g (w/w) of soil to make a homogeneous mixture and shaken by using overhead shaker for 15 minutes. After shaking, the samples were placed at room temperature for 30 min. A glass calomel electrode was used to measure the pH. Total nitrogen & carbon were measured by combustion using NA 1500 series 2 combustion reactors. A 1 gram aliquot of soil from each of the five samples was placed in aluminium foil dish to oven dry at 80°C for 24 hours. The dry soil was ground by mortar and pestle, weighed and placed in the GC thermal conductivity detector at 1020°C temperature and data were recoded.

Aromatic contact of leachate

UV/Visible spectrometry was used to detect the concentration of aromatic compounds in soil leachate defined by (Tsvetnenko and Evans 2002). A UV/V in GLP with model U-2001 was used for the analysis. Deionised water was used as standard. Soil leachates were placed in the quartz cuvette (Pyeunicam BS38751A, 10 mm F.O) and the concentration of aromatic compounds was measured using standard calibration curve.

Biosensor

The toxicity of soil leachate was determined by expressing the luminescence response as a percentage of the control. *Escherichia coli* HB101 freeze dried vials were resuscitated in 10 ml of 0.1M KCl solution (25 °C at 200 rpm) for 60 min in shaking incubator. A 20 μ l aliquot of resuscitated biosensor were added to each microtitre plate which contained 180 μ l of soil leachate. Samples were exposed for 30 min at room temperature. Lucy Anthos 1 microtitrate luminometer was used to measure the luminescence of soil leachate with each well read for 1 s. (Bundy *et al.*, 2001). The luminescence was expressed as a percentage of the luminescence from control treatments.

Aqueous phase hydrocarbon extraction

Total hydrocarbons in solution were measured by using GC 8000 Top Series. 10 ml of soil leachate was taken and 2ml of hexane was added to the sample. Samples were shaken for 30 min in the end shaker. Top 1 ml of hexane from the shaken samples were taken and put into the GC vial. An aliquot 100 μ l 1 squaline solution was added into the sample. IS squaline solution was prepared by using 1.0313 ml of IS squaline. The amount was added to 250 ml of hexane to make a squaline solution. Samples were analysed by GC –FID (flame ionization detection) defined by (Ong *et al.*, 2003) to detect the total hydrocarbons in the soil leachate.

Soil nutrients leachate analysis

The concentration of nitrates, phosphates and ammonia was determined in soil leachate by flow injection analysis (stannous chloride method) range 0.25 to 5.0 mg/l.

CO₂ Respiration

Level of CO₂ in the soil leachates were measured by using GC Model CP 9001 Chrompack gas chromatograph. A 1ml aliquot of soil leachate was taken in glass vial and placed in the oven at 25°C for 48 hours in order to evacuate CO₂ from the sample. After 48 hours, soil leachates were taken and CO₂ was measured. Standards of 350% ppm, 0.1% ppm and 0.5 % ppm were used. A 5ml aliquot of samples were taken from each of the soil leachates and diluted with nitrogen. Samples were injected in the GC column and data were recorded.

Results and Discussion

Retention time

After adding one hundred and seventy ml of deionised water into the leaching columns average leaching time was recoded. For the initial three days the average retention time of all the 30 replicates were approximately 2.5 to 3 hours. On the 4th day of replicate number five (R5) of sample (49 ORC) took more than 12 hours to leach. Leaching time of replicate number (R6) from soil leachate (74 Nil) increased to 4 hours on the fifth day. The pH from these two soil leachates were measured from the first leachate only. On day six retention time of replicate (R2, R4, and R6) from soil leachate (76 Mix), replicate (R1, and R4) from soil leachate (49 ORC) and replicate (R2, R5, and R6) from sample

number 74 Nil were increased to more than 24 hours and this resulted in water clogging of samples. About 40-50 % of soil samples from each of the treatments took more than 7 hours to leach down during their first run. On the second run leaching time further increased to 12 hours. A considerable change and variation in the pH was also observed from these samples.

Replicate (R3) from control soil leachate (74 Nil) also resulted in reduced hydraulic conductivity on day seven. Replicate (R3) from sample (49 ORC) was removed from the column and soil was remixed. No significant change in the retention time occurred. Replicate (74 Nil), (R5) and Replicate (49 ORC) (R4) were mixed with 15 g (w/w) of sand. Replicate (74 Nil) (R6) was remixed with 10 g of sand for the second time. On day seven Replicate R1, R3, R4, R5 of sample (49 ORC), replicate R2, R4, R6, of sample (76 Mix) and replicate R2, R3, R5, R6 of sample (74 Nil) were remixed with 10 grams (w/w) sand and filled back into the columns and run again. In few of the sample filter paper was also changed. One hour difference of increase in retention time were observed after remixing the sample with sand. On day eight sample (76 Mix) replicate (R4) and sample (74 Nil) (R1) took more than 24 hours to leach down. An increase of 4 hours was observed in sample (37 SPS) (R2). Some of the replicates were remixed twice by adding 10 g of sand and a sudden decrease in leaching occurred. Sample (76 Mix) R6 and (49 ORC) (R5) were leached down in 40 min. Two days after the remixing of sand a further increase in retention time of 2 hours observed in most of the samples.

About 50 % of the replicates from each of the treatments were clogged and retention time of these replicates increased to 24 hours and in some cases to 48 hours. Increase in retention time of 5-6 hours was observed in the soil column (49 ORC) and (74 Nil) on day 3 three, more than 50 % of the soil column resulted in increased residence time. About 80 % of the replicates of (49 ORC) were amended with sand and few replicates were mixed twice. On day six 40 % of replicates from column (21 Urea), 50-60% of replicate from soil column (76 Mix) and 30% of replicates from soil column (37 SPS) resulted an increase in retention time of 7- 8 hours. Kopinke *et al.*, (1995) suggested that solid coal tar particles in MGP contaminated soils have a significant effect on the retention of water soluble pollutants from the aqueous phase. The tar particles entrapped into the pores and blocked the mobility of the pollutants. One reason of clogging may be the non uniform packing of the soil (Townsend *et al.*, 2003). Haeseler *et al.* (1999) showed a clear relationship between soil polluted with coal tar from a former MGP and reported that coal tar polluted soils have high concentration of PAH in the form of tar particles further verifying this observation. Adsorption of these soil particles has been suggested as a limiting factor in mobility and

biodegradability of the pollutants in MGP soils (Alexander, 1995). Mahjoub *et al.*, (2000) studied the effect of tar in column experiment with similar methodology adopted in this experiment and showed that tar has a great resistance in leaching pollutants and have prolonged contact with water. The pollutant transfer was rapid during the initial two days of the experiment after adding deionised water. Dissolution of pollutants decreased by 60 % after 3 days of experiment. The reason may be the low water solubility and biodegradability of a number of its constituents present in tar. Contact with water also modifies the viscosity of tar. This characteristic of tar affects the pollutant transfer. One of the properties of tar is that it forms a "interfacial" membrane with in few days of contact with water that resist the transfer of pollutants (Nelson *et al.*, 1996). Addition of hydrocarbons may not necessarily increase the concentration of pollutants. A finding of a study suggests that artificial spiking of naphthalene into the soil column did not give rise to the naphthalene concentration in the aqueous solution. Variation of nutrients in the soil leachate might be one of the time depending factors and may have relevant to the hydraulic conductivity.

The low concentration of nutrients into the aqueous solution may be due to the hydrophobic properties of the pollutants. Tar polluted sub soils or soils had a great effect on the pollutant transfer in ground water and this phenomenon also affect the efficiency of remediation particularly the techniques like biological treatments (Mahjoub *et al.*, 2000). The addition of sand increased the porosity of the soil for a while but did not last for longer period. This has been observed by remixing the 10 grams of sand into the columns that have shown increased retention time. A day after mixing sand did not showed a significant change in retention time. Retention time increased to 4- 5 hours in most of the amendments. Bardos *et al.* (2000) reported that hydraulic conductivity depends upon the particle size and its distribution.

This increase in retention may have an effect on the toxicity, pH and nutrients in the soil leachate. Mobility of the contaminants in soil depends upon the interaction between the compounds present in the soil. These interactions makes the biological processes more complex (Bardos *et al.*, 2000)

pH Value

The high pH value at the start of the experiment in different soil leachates was caused by a cement amendment. A slight decrease in pH over time has been observed in the soil samples (Fig 2) shows a clear decreasing trend over time. The change in pH was related to the retention time of the leachate. Retention time of added deionised water in the columns increased over the duration of the experiment. Supplemental nutrients in the amended soil may also

have effect on the redox potential and the pH value. The redox potential defines the electron availability and efficiency of degradation.

Toxicity test of soil leachate

The *lux* marked metabolic biosensor *Escherichia coli* was used to assess the toxicity of the soil leachates. This biosensor was chosen because of its sensitivity and its luminescence across a wide range of pH values (Sinclair *et al.*, 1999). This sensor is responsive to a spectrum of hydrocarbons (Bundy 2001) significantly different response has been observed by the biosensor over the different treatments (Fig 3). Soil leachate (37 SPS) and (49 ORC) displayed a sudden increase in toxicity on day 5. Soil leachate (74 Nil Control) tends to be more toxic compared to the soil leachate with added nutrients. The difference in the toxicity may be due to the difference in concentration and type of pollutants in the soil leachate and the leaching capacity of the pollutant. In some cases the soil leachate has been taken for analysis from first leach only due to the reason of increased retention time, as the water stays for longer period of time in the leaching column when compared to other samples. Change in luminescence might be the cause of change in the chemistry of first and second leachate. The variable response of biosensor may be one of the reasons that can be related to the chemistry of first and second leachate.

A sudden decrease in luminescence has been observed in soil leachate (37 SPS). This soil column maintained an average of three hours in retention time during 70% of the experiment duration. Two of the replicates were mixed with sand in the last week of experiment. Addition of sand decreased the retention time of the soil column and changed the chemistry that resulted in more toxicity. Sudden decreased in luminescence in soil leachate (49 ORC) is due to the difference in soil leachate. 50 % of the soil columns were stuck and data were recorded from first leachate only. This shows more toxicity in first leachate compared to the second. Soil leachate (74 Nil) tends to be more toxic compared to the others. This is due to the remixing of sand in clogged samples that resulted in decrease in retention time.

Concentration of Hydrocarbons

Aromatic hydrocarbon concentration in leachate decreased over time. A sudden increase in the concentration was observed in the last week (Fig. 4) this may be due to the remixing of sand in the columns. On day 13 about 50 % of the soil samples with in all the five treatments in columns that resulted in increase in retention time were mixed with sand. The trapped hydrocarbons in the pores may have been leached down after stirring the soil in mortar and pestle. Temperature of incubator may be one of the contributing factors.

Concentration of nutrients

Over all concentrations of nitrates and phosphates in the soil leachate were low in few of the soil leachates. Maximum nutrients leached down during the first two days of experiment. Soil leachate (76 Mix, 49 ORC and 37 SPS) were more in nitrates. An increasing and decreasing trend in the concentration of nitrates has been observed over time. Concentration of nitrates in the leachate was more compared to phosphates (Table 3& 4). Literature shows that nitrates are more mobile than phosphates. Rosenbeg *et al.* (1996) showed low concentration of nitrogen when washed with sand compared to the nitrogen level in the soil only. The higher concentration of nitrates compared to the phosphates may be due to the mobile nature of nitrates. The other reason may be the contact of nutrients to the pollutants. An increasing retention time might have affected the leaching potential of the nutrients. Soil leachate (37 SPS) has the average retention time of three hours. Results show the continuous concentration of nitrates, which declined at the end as the column resulted an increase in retention time. Soil leachate (49 ORC) showed a low concentration on day three. This may be due to increase in retention time and clogging of 50-60% of soil columns. Increase in the concentration of nitrates on day seven may be related to the remixing of the soil columns with sand. Conversely the concentration of phosphates has been found very low in the leachate (37 SPS). The higher concentration of phosphate in soil leachate (49 ORC) has clearly shown the effect of residence time. Replicate (R4) were mixed with sand twice that affected the retention time which was decreased to 50 min.

Percentage of nitrogen and carbon in the soil were found very low (Table 2). Soil sample (74 Nil) was more in carbon concentration compared to the others.

TPH concentration

Soil leachate (49 ORC) and (76 Mix) were more in concentration compared to the other treatments. Soil leachate (21 Urea) was found in very low concentration (Table 5).

CO₂ Respiration

The amount of CO₂ in the soil leachate was very low. Soil leachate (21 Urea) and (49 ORC) were found more in concentration compared to the other leachates (Table 5).

Conclusion

Residence time has been observed as the main factor in controlling the soil leachate chemistry. Five treatments of soil responded differently. The experimental method did not produce to expected results to assess the effectiveness of *in situ* bioremediation technique. The mixing of cement changed the hydraulic conductivity of the soil and the existing properties of pollutants particularly the tar, which is the main factor of increased residence time. The biosensor response to the soil leachate was not constant but varied with time. Nutrients release was very low partly due to the elevated pH of the system. The presence of pollutants in the soil particularly the tar, probably has changed the pollutant transfer and might also be the cause of slow transfer of nutrients to the soil leachate. The pH has decreased over time. Residence time, soil properties and presence of organic compounds in the form of pollutants are the main factors that determine the rate of pollutants transfer into saturated and unsaturated zone. The short duration of this experiment may be one of the factors limiting a full assessment of *in situ* bioremediation. *In situ* bioremediation needs long term studies to assess the potential of effective bioremediation plan but novel methods employed here may develop to sustainable technologies in future:

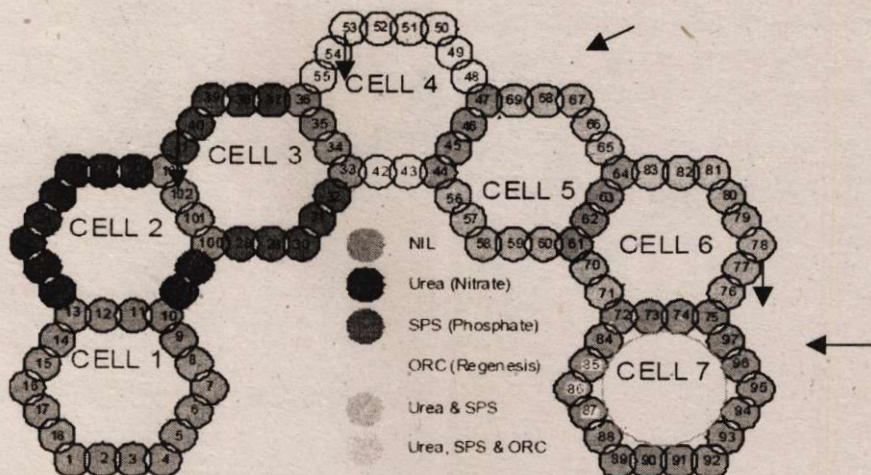


Fig 1. Cells with columns marked with sampling locations

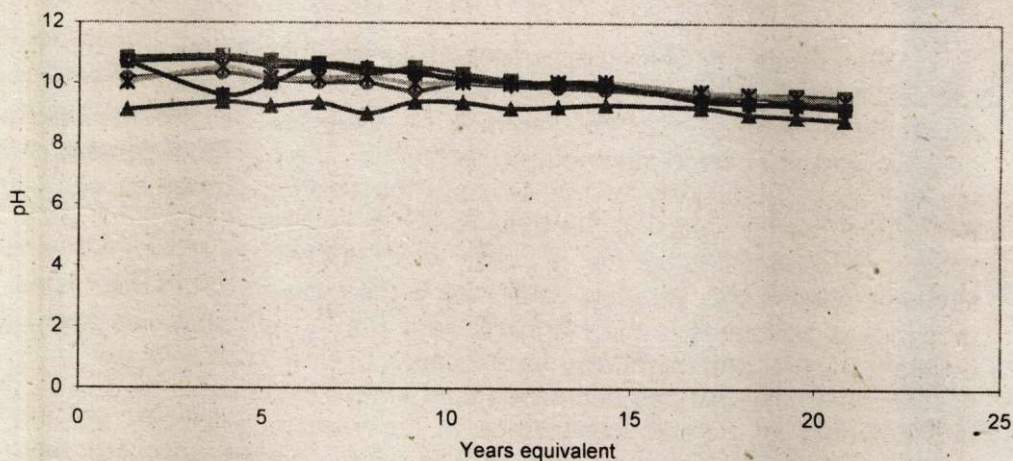


Fig 2. pH recorded from soil leachates over time ♦ 21 Urea, ■ 37 SPS, ▲ 49 ORC, ● 76 Mix, * 74 Nil and + Control (74 Nil)

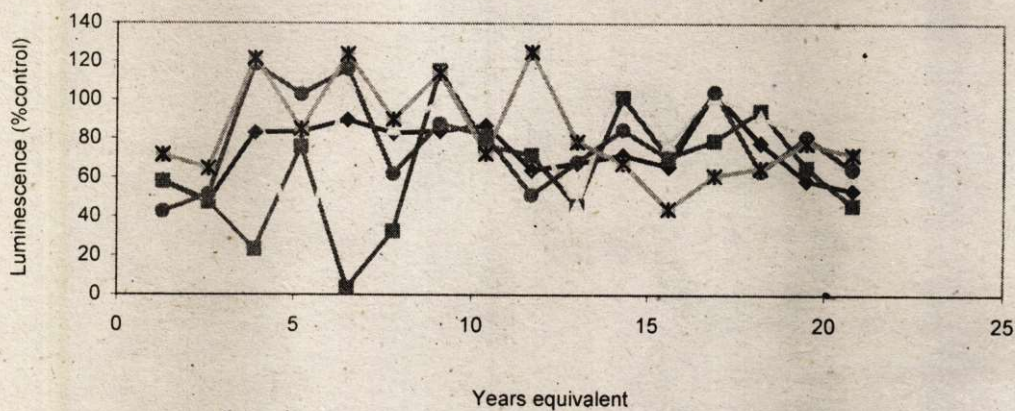


Fig 3. Response of metabolic biosensor *E. coli* HB101 to soil leachates as a percent of the control value. ♦ 21 Urea ■ 37 SPS ▲ 49 ORC ● 76 Mix * 74 Nil

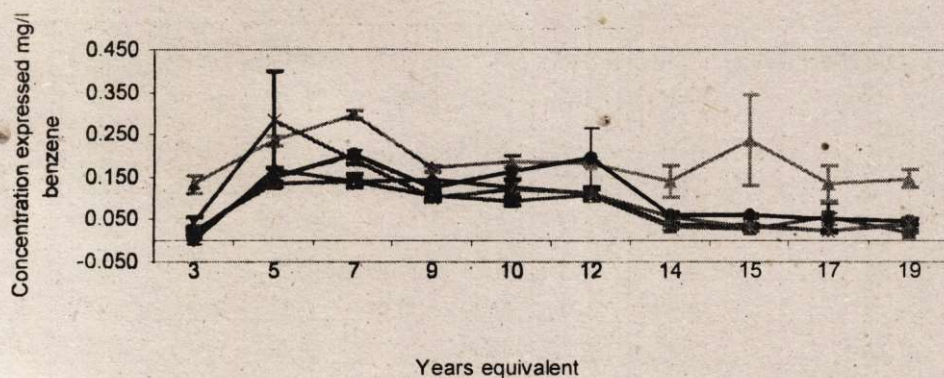


Fig. 4 Concentration of aromatic hydrocarbon showing soil leachate
 ◆ 21 Urea, ■ 37 SPS, ▲ 49 ORC, ● 76 Mix and * 74 Nil

Table 1. Details of soil samples

Sample ID	Type
21 Urea	Soil column mixed with Urea
37 SPS	Soil column mixed with super phosphate (SPS)
49ORC	Soil column mixed with Oxygen Retention Compound (ORC)
76 Mix	Soil column mixture of Urea+SPS+ORC (Mix)
74 Nil	Nothing added (Nil) – control

Table 2. Carbon and nitrogen percentage in the soil samples.

Sample ID	%Nitrogen	%Carbon
21 Urea	0.183	1.795
37 SPS	0.115	1.971
49 ORC	0.156	3.637
76 MIX	0.151	2.863
74 NIL	0.166	5.110

Table 3. Average concentration of nitrates in the soil leachate over the years (mg/l)

Years equivalent	1.30	2.60	3.90	6.50	9.10	13.00	15.60	19.50
21 Urea	64.68	20.78	0.00	36.68	36.47	34.50	7.60	3.17
STDV	0.04	0.02	0.00	0.01	0.01	0.02	0.00	0.00
SE	0.04	0.02	0.00	0.01	0.01	0.01	0.00	0.00
37 SPS	72.78	19.00	2.65	45.85	29.08	29.10	5.32	1.27
STDV	0.01	0.01	0.01	0.04	0.00	0.01	0.00	0.00
SE	0.03	0.01	0.00	0.01	0.01	0.01	0.00	0.00
49 ORC	72.50	15.72	0.00	20.50	46.80	26.62	6.97	4.43
STDV	0.02	0.01	0.00	0.01	0.02	0.01	0.00	0.00
SE	0.03	0.00	0.00	0.01	0.00	0.01	0.00	0.00
76 Mix	78.27	21.63	0.00	60.33	86.27	51.73	8.36	9.50
STDV	0.02	0.00	0.00	0.02	0.08	0.02	0.00	0.01
SE	0.03	0.01	0.00	0.02	0.02	0.02	0.00	0.00
74 Nil	67.10	14.63	2.18	50.00	68.50	50.75	9.50	11.40
STDV	0.01	0.01	0.01	0.05	0.02	0.01	0.01	0.02
SE	0.03	0.01	0.01	0.00	0.04	0.02	0.00	0.00
Control	0.00	0.00	0.00	70.00	69.97	65.77	30.26	2.11
STDV				0.04	0.01	0.01	0.05	0.00
SE				0.03	0.04	0.03	0.01	0.00

Table 4. Average concentration of phosphates in soil leachates over the years (mg/l)

Years equivalent	1.30	2.60	3.90	6.50	9.10	13.00	15.60	19.50
21 Urea	7.2	0.0	2.8	2.9	2.2	9.0	19.8	79.3
STDV	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1
SE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
37 SPS	3.8	0.0	32.2	0.0	0.0	0.0	23.8	37.3
STDV	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0
SE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
49 ORC	21.2	3.9	10.6	36.5	64.1	123.9	159.8	192.5
STDV	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.1
SE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
76 Mix	22.4	0.0	0.0	0.0	23.2	61.9	65.8	158.7
STDV	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1
SE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
74 Nil	25.7	0.0	14.2	0.0	14.9	30.1	40.8	95.7
STDV	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
SE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Control	0.0	0.0	0.0	32.2	0.0	40.3	59.5	135.3
STDV				0.0	0.0	0.0	0.0	0.1
SE				0.0	0.0	0.0	0.0	0.0

Table 5. Concentration of total polyaromatic hydrocarbons (TPH) in soil leachate and CO₂ Respiration

Leachate ID	TH-C (mg ml ⁻¹)			mg/L CO ₂ in original sample		
	Mean	SDTV	SE	Mean	SDTV	SE
21 Urea	1.12	0.0	0.0	1.00	1.314	0.536
37 SPS	71.64	0.1	0.0	3.52	3.231	1.319
49 ORC	0.065	0.1	0.1	2.74	1.695	0.692
76 Mix	125.24	0.2	0.1	3.19	2.520	1.029
74 Nil	13.59	0.0	0.0	2.99	1.650	0.674

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