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# Most Probable Rate of Horizontal Spread of Phenanthrene in a Sandy Loam Soil Under Natural Environment

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**Abstract:** The rate of horizontal spread of phenanthrene applied in a plot of land of sandy loam texture is determined in a field experiment of four months duration under natural environment. Efforts were made to study the effect of different physico-chemical parameters of the soil sample on the horizontal spread of phenanthrene. The concentration of the hydrocarbon at different points of time was determined by high-performance liquid chromatographic analysis of soxhlet extract of soil samples drawn from different distances from the point of application of the hydrocarbon. Simultaneously, internal samples were also analysed in order to detect the efficiency of the process of extraction and the extent of degradation during the study period.

The ambient temperature of the place of the experiment was in the range of 23.6 to 35.5˚C, which is suitable for microbial activities. From the internal experiment, it was found that there was a 36% loss of phenanthrene which indicates degradation during these four months of the experiment. It has been found that most of the Phenanthrene molecules move a distance of 15 cm from the point of application in the first 60 days. As such the most probable rate of horizontal spread became $2.89 \times 10^{-8}$ m $s^{-1}$.

**Key words:** Natural environment, phenanthrene, porosity, rate of horizontal spread of pollutant, sandy loam.

## Introduction

Pollutants in soil move vertically and horizontally under the influence of natural agents such as wind, rain, etc. unless degraded in a short period. The horizontal distribution of pollutants per unit of time in the soil is the rate of horizontal spread (Rahman et al., 2019). The movement of pollutants in soil is dependent on soil conditions such as soil texture, porosity, soil organic carbon, microbial population, nature of microbes, extent of adsorption, heavy metal concentration, etc. The polycyclic aromatic hydrocarbon (PAH) phenanthrene, EC Number: 201-581-5, CAS number: 85-01-8 is a substance identified as very persistent and very bio-accumulative (REACH, 2018). It is also one of the 16 polycyclic aromatic hydrocarbons (PAHs) identified as carcinogenic or potential carcinogen by the United States Environmental Protection Agency. Due to its persistent nature, it gets the opportunity to undergo vertical as well as horizontal movement in the soil. The extent, to which it will exert its detrimental effect on the environment, is decided by its persistency and subsequent adsorption and movement.

Łyszczarz et al. (2021) studied the role of different soil textures in the distribution of PAHs and trace elements in forest soils. The study confirmed the

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importance of the silt content, followed by soil organic carbon in PAH distribution. Small particles of soil such as silt absorb the PAH and disturb its distribution. Organic carbon content and nitrogen content played a predominant role in controlling microbial activity. Tao (2014) studied the effects of heavy metal cations viz. $Cu^{2+}$, $Cd^{2+}$, and $Ag^{+}$ on the sorption of PAH by phytoplankton. The cation–p interactions between the metal cations and PAHs facilitated the sorption of the PAH. This is increased with the softness order of the metals $Cd^{2+} < Cu^{2+} < Ag^{+}$ and the p donor strength order of the PAHs Phenanthrene < Pyrene < Benzo[a] pyrene. Phenanthrene sorption was facilitated by low concentrations of metal salts. Jia et al. (2018) cited the first-order rate constants for the decay of PAHs on various types of metal ion-containing modified clays where the values for a particular PAH are different for different heavy metal ions. The study establishes the interaction of heavy metal ions and PAH in soil. They also reported that the first-order decay constant of Anthracene decreases in modified clays in the presence of the following heavy meals in the order of $Fe^{+3} > Cu^{+2} > Ni^{+2} > Co^{+2} > Zn^{+2}$. They also established the potential role of metals in the formation and fate of PAH-induced environmentally persistent free radicals at co-contaminated sites. Kaabi et al. (2022) used HPLC analysis of soil extracts in order to find out PAH acenaphthylene concentration in river sediment. The degradation process was reported to follow first-order kinetics.

## Materials and Methods

### Soil Bed Preparation and Study on the Quality of the Experimental Soil

A plot of land with separated multiple plots of $1m^2$ area is made ready by removing the plan residues and tilling a few of them up to a depth of 15 cm. One soil sample of approximately 1kg was drawn by a quartering process in order to determine a few important physico-chemical parameters of the soil. Out of this, one part weighing 20 g was taken for soxhlet extraction and HPLC analysis of the extract in order to detect phenanthrene if any, in the natural sample. Another part weighing 498.75 g of powdered soil was taken in a one litre glass container which is known as an internal plot. The rest part is used to determine the different physico-chemical parameters of the soil. The ambient temperature and rainfall during the period of the experiment were also recorded since the movement and degradation of hydrocarbons in soil under the natural environment are likely to be influenced by these natural factors.

### Experiment with Internal Plot

A solution of 1.25g of phenanthrene in methanol solvent was added to the internal plot and mixed thoroughly. The solubility of Phenanthrene in methanol is 0.00543 mol fraction which is 2.4g per 100 ml of methanol of 0.795g $cm^{-3}$ density (Fakhree et al., 2009). The Hydrocarbon concentration applied has become 2500 ppm in the soil of the internal plot. The mass of methanol is not taken into account in this ppm calculation since this being a low boiling liquid, will volatilise from the internal plot quickly. After 1 day, 1 month, 2 months and 3 months gap from mixing of phenanthrene, 20 g of thoroughly mixed soil in each time was taken for soxhlet extraction. Assuming no or negligible loss in one day, the result obtained from the extract of 1-day gap was used to know the efficiency of the extraction process.

### Experiment with Outdoor Plots

About 5 g of phenanthrene was applied at the centre in each of the three different outdoor plots. 20 g of soil were collected from each of these plots by quartering process from circumferences of radii 15, 30 and 45 cm from the point of application. This is done at one month, two months and three months time from the day of application. These samples were soxhlet extracted and extracts were analysed through HPLC in order to determine the phenanthrene concentration.

### Soxhlet Extraction

A total of 23 soil samples drawn by the standard procedure were used for soxhlet extraction. Each such sample was of 20 g mass. The solvent used for the purpose was a 1:1 mixture of acetone and n-hexane since phenanthrene is highly soluble in this solvent system (Haleyur et al., 2016). This solvent system is distilled out later on and dissolved in the residues in HPLC-grade methanol for HPLC analysis.

### HPLC Analysis

The HPLC instrument was calibrated before sample analysis using a standard solution of 50, 100, 150, 200 and 250 ppm phenanthrene in HPLC-grade Methanol. A total of 23 soil sample extracts were analysed in order to estimate the phenanthrene concentration. The particulars of the analysis are --Instrument: LC_DAD (Offline), Peak: Phenanthrene -- ESTD -- DAD: Signal A, 254 nm/Bw:4 nm.

### XRF Analysis

X-ray fluorescent spectroscopic analysis of the soil was done in order to know its elemental composition. Zetium

DY 2942 model instrument was used for the purpose. Interpretation of the concentration of heavy metals was done on the basis of geo-accumulation index ($I_{geo}$), contamination factor (CF) or degree of contamination and pollution load index (PLI). $I_{geo}$ is calculated by using the formula given below (Muller, 1969).

$$I_{geo} = \log_2 \frac{c_n}{1.5B_n} \text{ or } I_{geo} = \frac{\log \frac{c_n}{1.5B_n}}{\log 2}$$

where $C_n$ is the measured concentration of the examined metal (n) in the soil, $B_n$ is the geochemical background value of the metal (n). Factor 1.5 is introduced to minimise the possible variations of the background values due to lithological variations. The world surface rock average data was considered as background concentration (Saikia et al., 2014). The CF values are used to measure the pollution load of the soil related to a particular heavy metal. CF was calculated by the following relation (Manoj et al., 2012).

$$CF = \frac{\text{Concentration of metal}}{\text{Background value of metal}}$$

PLI gives an idea of the overall toxicity status of the sampling site due to heavy metals. PLI was determined by the following equation

$$PLI = \sqrt[n]{CF1 \times CF2 \times CF3.....CFn}$$

where n is the number of metals under study and CF is the contamination factor. PLI >1 indicates polluted and PLI<1 indicates non-polluted conditions (Thomilson et al., 1980).

## Results and Discussion

### Physico-Chemical Parameters of the Soil

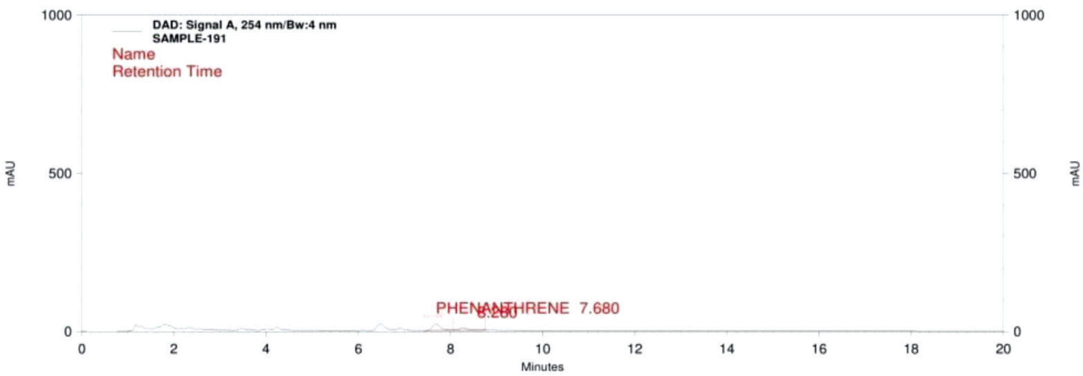
Ten important physico-chemical parameters of the blank soil are selected and their values are determined. These parameters along with their methods of determination are given in Table 1. The percentage composition of the three types of soil particles was found to be sand 71.94%, silt 8.50% and clay 19.56% and hence it belongs to the class of sandy loam texture. The pH of the soil is 6.7, which is very suitable for the growth of most plants because of the lack of its adverse effects on nutrient-supplying power of the soil. On the basis of Dutch standard soil parameters, the maximum acceptable concentration of phenanthrene in the soil is 1.9 $mg.kg_{dwt}^{-1}$ i.e. 1.9 ppm in dry soil (Verbruggen and van Herwijnen, 2011). In the experimental soil, the same has been found to be 3.8 ppm, which seems to be much higher than an acceptable value. The average daily minimum and daily maximum temperature of the place of the experiment was 23.6 and 35.5˚C which is suitable for microbial activities (Khan and Anjaneyulu, 2005). Other parameters do not show any unhealthy condition of the experimental soil.

### HPLC Analysis of Extract of Internal Sample

From the analysis of the blank soil sample, it was found that Phenanthrene appeared at the retention time of 7.68 minutes and showed the concentration to be 3.8 ppm (Figure 1). The same was treated as 4 ppm in calculations later on. Thus, in the internal sample, the initial total concentration of Phenanthrene was found to be 2504 ppm. In the HPLC analysis of the extracts

**Table 1: Physico-chemical parameters of blank soil sample**

| *Sl.No.* | *Parameters/Compound* | *Unit* | *Results* | *Experimental methods* |
|---|---|---|---|---|
| 01 | Colour | - | Brown | Simple observation |
| 02 | Texture | - | Sandy loam | Hydrometer method |
| 03 | Bulk Density (BD) | (kgm-3) | 1028 | Mass/volume calculation |
| 04 | Particle Density (PD) | (kgm-3) | 3135 | Mass/particle volume calculation |
| 05 | Porosity | (%) | 67.21 | Calculation method |
| 06 | Electrical Conduc. (EC) | (S/m) | 0.121 | Conductivity meter |
| 07 | Soil Organic Carbon (SOC) | (%) | 1.10 | Wet Oxidation method |
| 08 | Soil Organic Matter (SOM) | (%) | 1.90 | Wet Oxidation method |
| 09 | pH | - | 6.7 | Digital pH Meter method |
| 10 | Phenanthrene | (ppm) | 3.8 | HPLC analysis of Soxhlet Extract. |




Channel A

| DAD: Signal A, 254 nm/Bw:4 nm Results Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.680 | 554822 | 3.816608 |
| Totals | | 554822 | 3.816608 |

**Figure 1: HPLC chromatogram of blank soil.**

of the internal sample, 2421 ppm was detected on the first day of extract. It was assumed that during this one day, there was no degradation of the PAH. As such, the efficiency of the overall extraction of the hydrocarbon from experimental soil is found to be 96.68% which was taken as 97%. The results obtained for the extraction of phenanthrene from the internal plot after $0^{th}$, $1^{st}$, $2^{nd}$ and $3^{rd}$ month are given in Table 2. Since the process is 97% efficient, hence the actual concentration of the PAH will be obtained by multiplying the chromatogram reported concentration by a factor of 1.03. As such, column IV of the table is obtained by multiplying the data of column III by 1.03. The rate constant for the disappearance of Phenanthrene in the internal plot is calculated as per first order rate equation. Here the initial concentration of the PAH taken is 100 ppm. It has been found that the rate constant decreases in the form of a number in geometric progression series (Figure 2).

**XRF Analysis**

During XRF analysis quantities of oxides of ten elements and another eight heavy metals/trace elements are detected (Table 3). The calculated $I_{geo}$ values are given in Table 4. The CF values with respect to Ni and Zn are less than 1, which is considered a low contamination factor. For the other metals under investigation, the same value lies in the range of equal or greater than 1 but lower than 3. These metals have

**Table 2: Concentration of residual phenanthrene after uniform gap of one month in the internal sample**

| *Time after which soxhlet extraction was carried out* | *Recovered amount of Phenanthrene [as per HPLC chromatogram]* | *Recovered* | *Residual concentration of Phenanthrene* | *Rate constant for disappearance of Phenanthrene as per first order reaction kinetics* | *Relationship between rate constants* |
|---|---|---|---|---|---|
| (*month*) | (*ppm*) | (%) | (%) | (*month*-1) | |
| *Col. .I* | *Col. .II* | *Col. .III* | *Col. .IV [Col.III x 1.03]* | *Col.. V* | |
| 0 | 2421 | 97 | 100 | -- | |
| 1 | 1817 | 73 | 75 | K1 = 0.2876 | |
| 2 | 1607 | 64 | 66 | K2 = 0.2077 | K1=1.3847 K2 |
| 3 | 1560 | 62 | 64 | K3 = 0.1487 | K2=1.3968 K3 |

moderate contamination factors (Hakanson, 1980). It has been found that the $I_{geo}$ value for most of the metals studied is negative, that is, below zero. As such, with respect to these metals, the soil is unpolluted. These metals are Co, Cr, Cu, Mn, Ni and Zn. It indicates that these metals are not supposed to influence the adsorption of PAH. Consequently, the horizontal rate of spread is not likely to be influenced by the cited heavy metals. On the other hand, metals such as Al, Fe, Pb and Ti have $I_{geo}$ values in the range of 0 to 1 and they are stated to be in the range of unpolluted to moderately polluted levels (Muller, 1969). The three heavy metals viz. Fe, Pb and Ti are likely to take part in the absorption of PAH in soil. The pollution load index with respect to the heavy metals is found to be 1.15. Since the PLI value is higher than 1, the soil is polluted with respect to heavy metals. Consequently, it is implied that the rate of horizontal spread is slightly retarded by heavy metals.

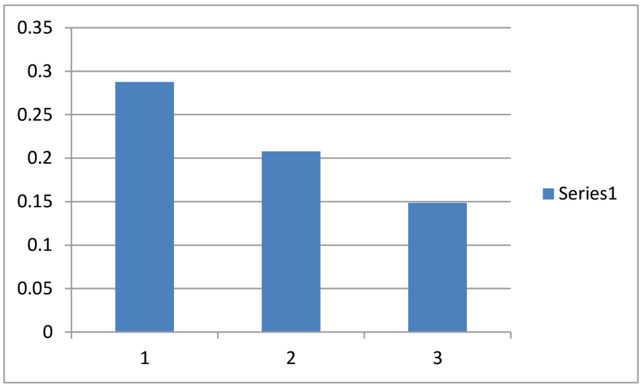



**Figure 2: First order rate constant of phenanthrene disappearance with respect to time in months in the internal plot.**

**Table 3: Results of chemical analysis of soil sample by XRFS**

| *Sl No.* | *Major & minor element oxides ( weight percent)* | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$(T) | MnO | MgO | CaO | $Na_2O$ | $K_2O$ | $TiO_2$ | $P_2O_5$ |
| 2 | 51.91 | 28.48 | 13.33 | 0.117 | 1.34 | 0.25 | 0.23 | 2.56 | 1.45 | 0.17 |
| | *Concentration as element (ppm-calculated)* | | | | | | | | | |
| 3 | Si | Al | Fe | Mn | Mg | Ca | Na | K | Ti | P |
| 4 | 242247 | 150776 | 93310 | 906 | 8040 | 1786 | 1706 | 21243 | 10875 | 742 |
| | *Heavy metals/Trace Elements (ppm)* | | | | | | | | | |
| 5 | As | Co | Cr | Cu | Ni | Pb | Ti | | Zn | |
| 6 | 5 | 17 | 82 | 32 | 44 | 46 | 10875 | | 72 | |

**Table 4: Calculation of $I_{geo}$ values of the metals**

| *Sl No.* | *Metal* | *Concentration detected ($C_n$)* | *Background concentration ($B_n$)* | $CF = \frac{C_n}{B_n}$ | $\frac{C_n}{1.5B_n}$ | $\log \frac{C_n}{1.5B_n}$ | $I_{geo} = \frac{\log \frac{C_n}{1.5B_n}}{0.301}$ |
|---|---|---|---|---|---|---|---|
| | | (ppm) | (ppm) | | | | |
| 1 | Al | 150776 | 69300 | 2.1757 | 1.4505 | 0.1615 | 0.5366 |
| 2 | Co | 17 | 13 | 1.3077 | 0.8718 | -0.0596 | -0.1980 |
| 3 | Cr | 82 | 71 | 1.1549 | 0.7700 | -0.1135 | -0.3771 |
| 4 | Cu | 32 | 32 | 1.0000 | 0.6667 | -0.1761 | -0.5850 |
| 5 | Fe | 93310 | 35900 | 2.5992 | 1.7328 | 0.2388 | 0.7932 |
| 6 | Mn | 906 | 750 | 1.2080 | 0.8053 | -0.0940 | -0.3124 |
| 7 | Ni | 44 | 49 | 0.8980 | 0.5986 | -0.2229 | -0.7407 |
| 8 | Pb | 46 | 16 | 2.8750 | 1.9167 | 0.2826 | 0.9387 |
| 9 | Ti | 10875 | 3800 | 2.8618 | 1.9079 | 0.2806 | 0.9321 |
| 10 | Zn | 72 | 127 | 0.5669 | 0.3780 | -0.4225 | -1.4037 |

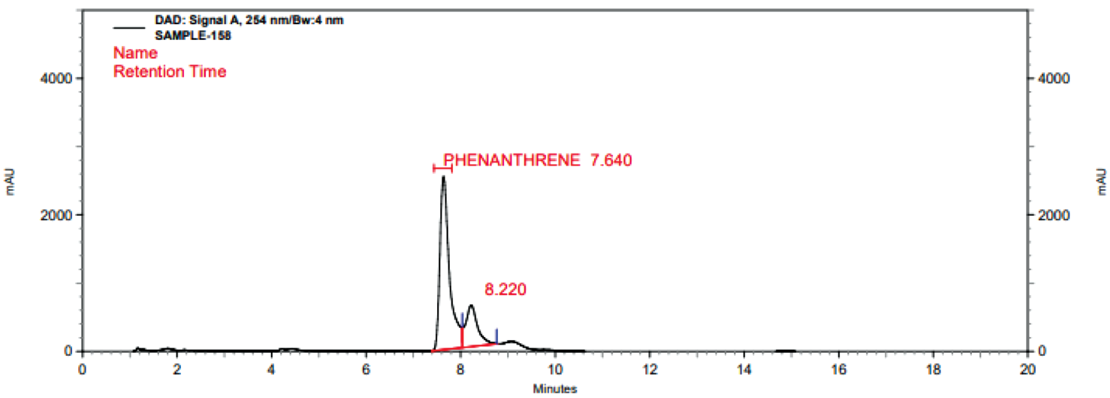
**Porosity and Phenanthrene Movement**

The results of HPLC analysis of the soxhlet extracts of contaminated soil samples collected from the 15, 30 and 45 cm circumferences from the point of application of phenanthrene on the outdoor plots are given in Table 5. These samples were drawn after 1, 2 and 3 months of application in three plots having different porosities numbered as plot no. 1, 2 and 3. Figures 3 to 8 are a few of the HPLC chromatograms of samples from plot no. 1 at a distance of 15, 30 and 45 cm after one, two and three months of placement. The graphical representation of the data on the horizontal rate of spread is given in Figures 9 and 10.

It has been found that the porosity during this four months experimental period was not fixed. This is quite natural. The experiment was carried out during the summer monsoon season. As such, there were rains. Clay particles undergo expansion in contact with water (Meshram et al., 2021). Since the experimental soil contains 19.56% clay, the decrease in porosity with the progress of time is supposed to be due to the expansion of soil particles. The average porosity was 44.7%, 42.4% and 41.4% in plots 1, 2 and 3 respectively. It has been found that the untilled soil (plot no. 3) with a minimum initial porosity of 42.3% has recorded the highest horizontal distribution (638 ppm) during the

**Table 5: Concentration of phenanthrene at 15, 30 and 45 cm circumference of the point of application in experimental plots of different porosities**

| *Sample Plot No.* | *Porosities at interval in months* | | | | *Average porosity* | *At circumference of 15 cm* | | | *At circumference of 30 cm* | | *At circumference of 45 cm* | *Series for graphical representation↓* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0(Initial) (%) | 1 (%) | 2 (%) | 3 (%) | (%) | 1m (ppm) | 2m (ppm) | 3m (ppm) | 2m (ppm) | 3m (ppm) | 3m (ppm) | |
| 1 | 69.6 | 45.3 | 30.2 | 33.6 | 44.7 | 531 | 1173 | 649 | 404 | 496 | 544 | 1 |
| 2 | 45.4 | 45.2 | 36.4 | 42.7 | 42.4 | 243 | 1210 | 631 | 599 | 561 | 487 | 2 |
| 3 | 42.3 | 41.5 | 37.6 | 44.1 | 41.4 | 638 | 695 | 254 | 789 | 098 | 276 | 3 |
| Grouping for graphical representation→ | | | | | | Set 1 | Set 2 | Set 3 | Set 4 | Set 5 | Set 6 | |

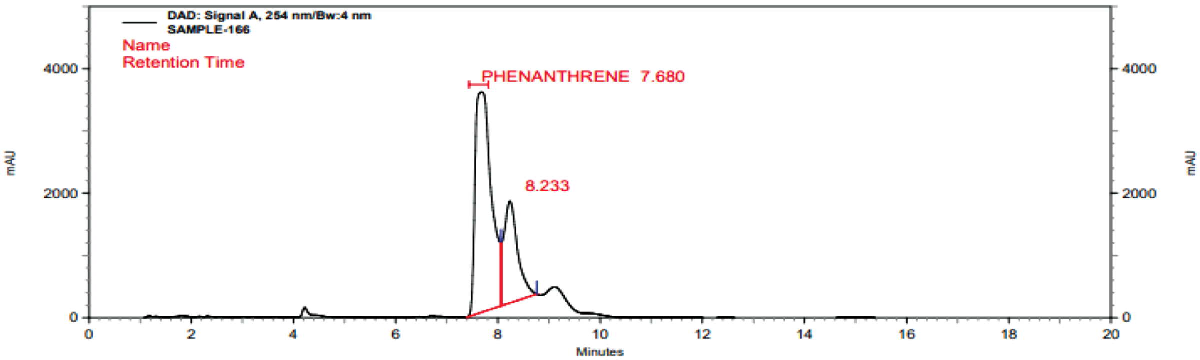



Channel A

**DAD: Signal A, 254 nm/Bw:4 nm Results**

| Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.640 | 74952201 | 515.594486 |
| Totals | | 74952201 | 515.594486 |

**Figure 3: HPLC chromatogram of 15 cm radius sample from plot 1 in 1 month.**

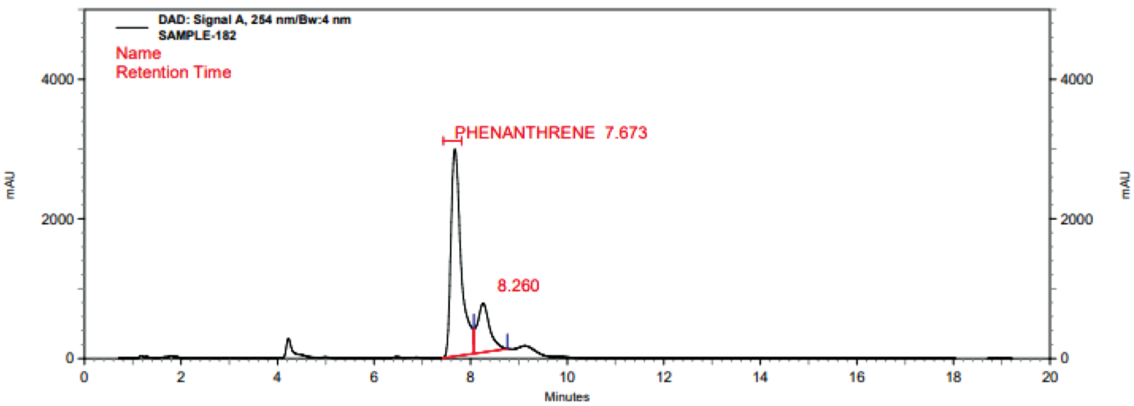



Channel A

**DAD: Signal A, 254 nm/Bw:4 nm Results**

| Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.680 | 165522421 | 1138.624969 |
| | | | |
| Totals | | 165522421 | 1138.624969 |

**Figure 4: HPLC chromatogram of 15 cm radius sample from plot 1 in 2 months.**



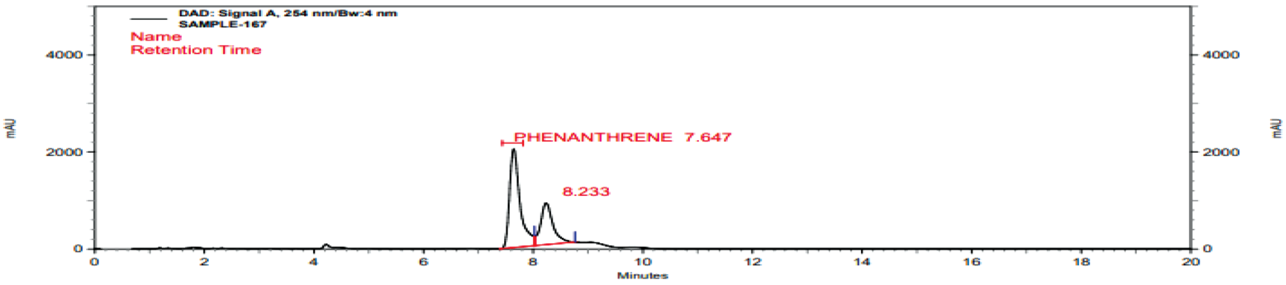

Channel A

**DAD: Signal A, 254 nm/Bw:4 nm Results**

| Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.673 | 91603810 | 630.140525 |
| | | | |
| Totals | | 91603810 | 630.140525 |

**Figure 5: HPLC chromatogram of 15 cm radius sample from plot 1 in 3 months.**

first month. This might be due to superficial movement along with surface runoff. Soil in plot no.1 is highly porous (69.6%), which was a tilled soil. In this sample, vertical movement of the PAH was expected, but the runoff water is supposed to bring a large amount of PAH (531 ppm) to a distance of 15 cm in the first month. In all the above plots, it has been found that most of the molecules move 15 cm in the first 60 days. The

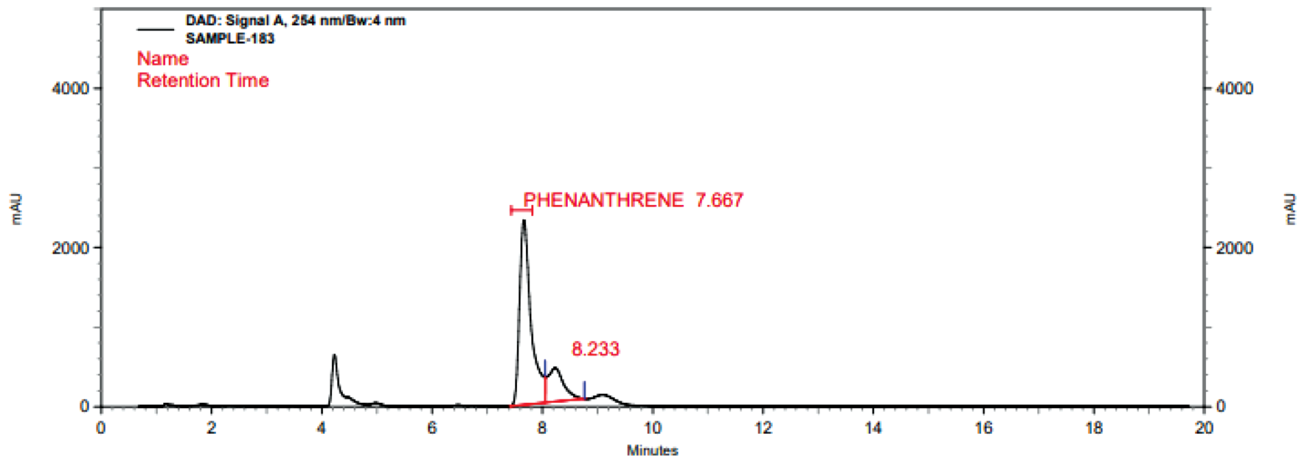



Channel A

**DAD: Signal A, 254 nm/Bw:4 nm Results**

| Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.647 | 56975639 | 391.934125 |
| Totals | | 56975639 | 391.934125 |

**Figure 6: HPLC chromatogram of 30 cm radius sample from plot 1 in the second month.**



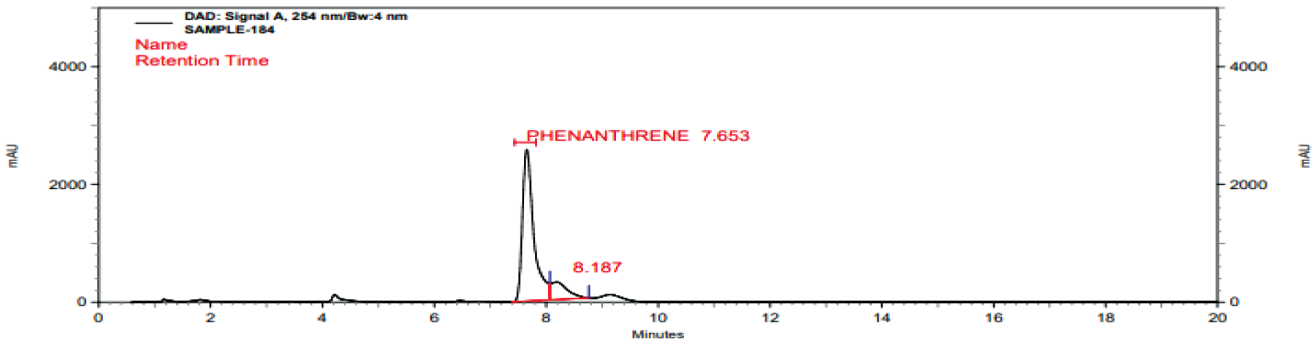

Channel A

**DAD: Signal A, 254 nm/Bw:4 nm Results**

| Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.667 | 71045102 | 488.717641 |
| Totals | | 71045102 | 488.717641 |

**Figure 7: HPLC chromatogram of 30 cm radius sample from plot 1 in the third month.**

concentration of phenanthrene is found to decrease at 30 and 45 cm distances from the point of application. As the distance increases, the hydrocarbon spreads to more and more areas of the soil layer and encounters contact with more and more natural agents which accelerates its degradation. These natural agents may include microorganisms, photons etc. The experiment in the internal plot also indicates a natural loss of 36% in three months' time, since there is no opportunity for the hydrocarbon to move horizontally and vertically. Nutrient availability in more and more areas of soil will accelerate biodegradation.




**Channel A**

**DAD: Signal A, 254 nm/Bw:4 nm Results**

| Name | Time | Area | Amount |
|---|---|---|---|
| PHENANTHRENE | 7.653 | 76754522 | 527.992611 |
| Totals | | 76754522 | 527.992611 |

**Figure 8: HPLC chromatogram of 45 cm radius sample from plot 1 in the third month.**

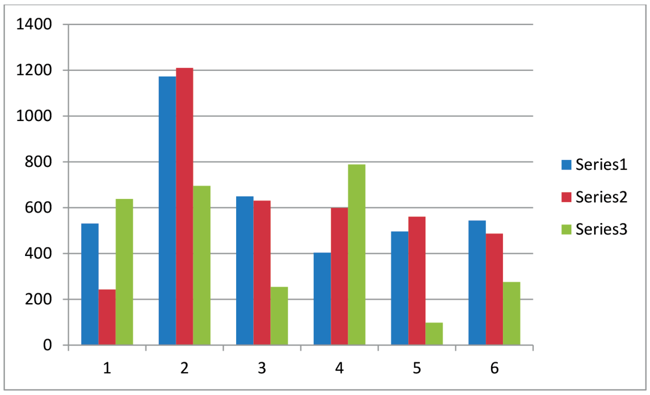



**Figure 9: Bar diagram of concentration of phenanthrene at distances 15, 30 and 45 cm.**

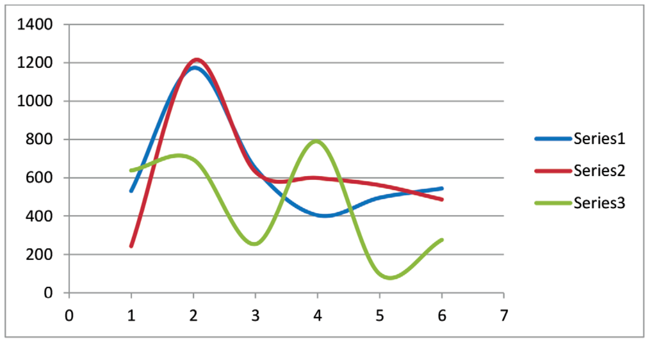



**Figure 10: Line diagram of concentration of phenanthrene at distances 15, 30 and 45 cm.**

It has been found that in each of the experimental plots, the Phenanthrene concentration is highest at a 15 cm radius in the second month of application. As such, the most probable speed of the Phenanthrene molecules is 0.25 cm per day which is $2.89 \times 10^{-8}$ m $s^{-1}$.

## Conclusion

In spite of having doors open to all natural processes such as degradation, adsorption in soil particles, interaction with heavy metals, vertical movement and surface runoff; most of the Phenanthrene molecules move a distance of 15 cm in 60 days. As such, the most probable rate of horizontal spread of Phenanthrene is 0.25 cm per day ($2.89 \times 10^{-8}$ m $s^{-1}$) in the experimental soil of sandy loam texture. Phukon et al. (2023) reported (in a 12-month experiment) that fluorene moves mostly at a distance of $1.45 \times 10^{-8}$ $ms^{-1}$ in a sandy loam under similar physicochemical conditions. The solubility of phenanthrene in water is 0.42 mg/L at 8.5°C, 1.6 mg/L at 15°C, 0.82 mg/L at 21°C, 1.10 mg/L at 25˚C , 1.3 mg/L at 30°C (Yalkowsky et. al. 2010). It is evident that during this period, a part of Phenanthrene dissolves in soil water. Since the experiment was carried out in the summer-monsoon season, both the vertical movement and surface runoff are possible processes. Out of these processes involving rain water, the surface runoff will expedite the horizontal movement where as the vertical movement will lower it. Although phenanthrene is a persistent PAH, the experiment on the internal plot shows that there was 36% degradation during this period of four months. This process has also lowered the speed of horizontal movement.

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