



Increasing density and viscosity of light non-aqueous phase liquids are indicators of natural source zone depletion

Jonathan W. N. Smith^{1*}, Emiliano Hinojosa² and Alan F. M. Hill¹

¹ Shell Research Ltd, Shell Centre, York Road, London SE1 7NA, UK

² Equilon Enterprises LLC, 3333 Highway 6 South, Houston, TX, USA

JWNS, 0000-0002-6568-842X

* Correspondence: jonathan.w.smith@shell.com

Abstract: Natural source zone depletion (NSZD) acts to reduce the mass and/or toxicity of light non-aqueous phase liquids (LNAPL) in the shallow subsurface. Several indirect measurement techniques are commonly used to estimate LNAPL mass depletion based on CO₂ generation by hydrocarbon mineralization, heat production by exothermic biodegradation processes, and subsurface gas concentration gradients.

This paper presents data on LNAPL density and viscosity and shows changes that result from NSZD processes. Increasing trends in LNAPL density and viscosity were observed that provided a qualitative line-of-evidence for NSZD. When LNAPL density and viscosity were combined with contemporaneous LNAPL chemistry analysis it was possible to correlate changes in the physical properties to depletion of alkanes from the LNAPL in a manner that would inform a quantitative NSZD assessment. LNAPL density and viscosity measurements are relatively cheap and reproducible, and increasing trends in LNAPL density and viscosity provide additional lines-of-evidence for NSZD.

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Natural source zone depletion (NSZD) is the combination of naturally occurring processes that reduce the mass and/or toxicity of light non-aqueous phase liquids (LNAPL) in the shallow subsurface. The physical, biological and chemical processes that contribute to NSZD have been reviewed (Garg *et al.* 2017; Smith *et al.* 2022) and best practice guidance published (e.g. CRC CARE 2020; CL:AIRE 2024). Several measurement techniques have been developed by which NSZD rates can be estimated, including:

- (1) ground surface CO₂ efflux using dynamic closed chambers, which measure the flux of CO₂ generated by hydrocarbon biodegradation (Sihota *et al.* 2011; Sihota and Mayer 2012);
- (2) time-averaged ground surface CO₂ efflux using sorbent CO₂ traps (McCoy *et al.* 2014);
- (3) soil vapour concentration gradient analysis that considers downward movement and depletion of oxygen, coupled to the upwards movement and depletion of CH₄ and generation of CO₂ (Johnson *et al.* 2006; ITRC 2018);
- (4) temperature gradient analysis ('thermal methods') that evaluate the heat generated from exothermic biodegradation of LNAPL components (Sweeney and Ririe 2014; Warren and Bekins 2015; Sale *et al.* 2018); and
- (5) LNAPL chemical composition change, which relies on the preferential removal of volatile and labile components relative to more conservative compounds in the LNAPL to calculate compound-specific depletion rates (DeVaull *et al.* 2020).

Each of these five methods has its advantages and disadvantages (see table 1 of Smith *et al.* 2022) and, with the exception of the LNAPL chemical composition method, they are all indirect measurements of LNAPL depletion. Consequently a lines-of-evidence approach to estimating NSZD is typically recommended (Wozney *et al.* 2022; CL:AIRE 2024). Such multiple lines-of-evidence approaches have been demonstrated to be effective at both large (Kulkarni *et al.* 2020) and small (Smith *et al.* 2021) LNAPL-impacted sites, and for a range of LNAPLs types (Guan

et al. 2024). Two additional methods of observing the effects of LNAPL NSZD are presented here, based on increases in density and viscosity as NSZD/LNAPL weathering progresses *in situ*.

NSZD of LNAPL typically results in preferential depletion of light and more water-soluble components resulting in enrichment of the heavier and less mobile compounds (Douglas *et al.* 1996; Brown *et al.* 2017). The resulting changes in LNAPL chemical composition typically reduce the environmental risk profile of the LNAPL over time (Thornton *et al.* 2013; CL:AIRE 2014). These NSZD processes would also be expected to change the bulk properties of the LNAPL (Lundy 2014) in a manner analogous to that described for weathering of crude oil and generation of heavy oil and associated natural gas in oil reservoirs where the changes make production of more viscous oils challenging (Head *et al.* 2003). Changes in LNAPL properties are a result of any or all of the aforementioned NSZD/weathering processes and both the relative importance and net effect of these processes are expected to vary spatially. This paper describes an assessment of LNAPL density and viscosity changes as potential indicators of NSZD.

Approach

LNAPL density and viscosity were measured over an eight-year period (2016–24) at a well characterized field site where LNAPL was present at the water table. The site, which is located in northern Europe and described in Statham *et al.* (2023), had a mixed LNAPL comprising refined fuels including diesel, kerosene (jet fuel) and gasoline. Release of these products occurred from several sources, but most notably a buried pipeline carrying refined fuels. The main LNAPL release from a pipeline was reported in 1990. This product then mixed with earlier releases associated with a nearby road tanker loading gantry that occurred in the 1980s. The LNAPL investigated is therefore believed to be around 35 years old, with a component (likely <10%) estimated to be 5–10 years older.

LNAPL was collected and analysed on a quarterly basis from each of the eight modular LNAPL-recovery systems (called 'technical

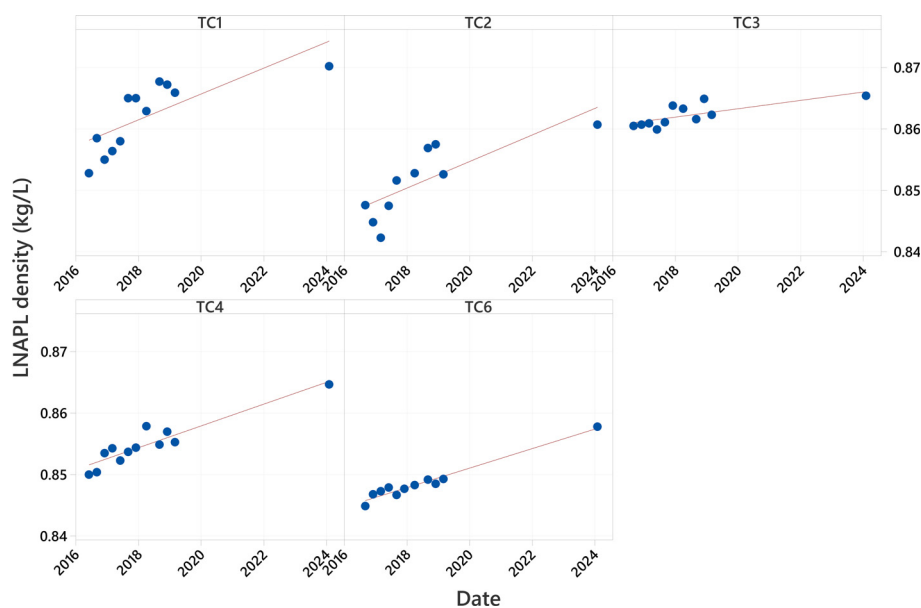


Fig. 1. Temporal change in LNAPL density (at 10°C) in LNAPL recovery technical chambers (TC).

chambers', TCs) present across the site. LNAPL density (kg L^{-1}) and dynamic viscosity (mmPa^{-2}) were measured each quarter between June 2016 and March 2019, and again in February 2024. Kinematic viscosity ($\text{mm}^2 \text{s}^{-1}$) was calculated by dividing the measured dynamic viscosity by the measured density. LNAPL chemical data were also analysed from June 2016, and have continued to be measured quarterly until present (most recent data February 2024) for those TCs that continue to operate. The TCs were established and operated at different times. The first TC established was TC1, which operated from 2015 until 2023. By contrast, TC8 came on-line early in 2022 and continues to be operational.

Methods

The dynamic viscosity and density of recovered LNAPL were measured at Triton Analytics Corp laboratory in Houston, TX, using an Anton Paar Stabinger Viscometer SVM 3000 and following ASTM Method D7042. LNAPL viscosity and density were measured at both 10°C and 30°C.

LNAPL chemistry was analysed at the Shell Global Solutions (US) Inc. environmental forensic laboratory located in Houston, TX, using gas chromatography/mass spectrometry (GC/MS) and

gas chromatography/flame-ionization detection (GC/FID). A direct oil injection method was used, applying a split ratio of 150:1 to ensure that the amount of detected material was within the data acquisition program's range. The method covered the gasoline–diesel range (~C5–C26) and may not be appropriate for the analysis of heavier components.

Selected indicator compounds were identified and reported quarterly as part of the remediation performance monitoring and included BTEX, ether oxygenates, individual C5–C8 alkanes, trimethyl-benzenes and -pentane, as well as indicators of LNAPL age: nC17/pristane and nC18/phytane ratios. The changes of relative concentration of a biodegradable component (nC17, nC18) to a recalcitrant component (pristane, phytane) are often used in forensic investigations to estimate the duration since a release occurred (Christensen and Larsen 1993).

Results

LNAPL density and viscosity

LNAPL density was measured from each operating TC between 2016 and 2024. It can be seen that increases in LNAPL density were

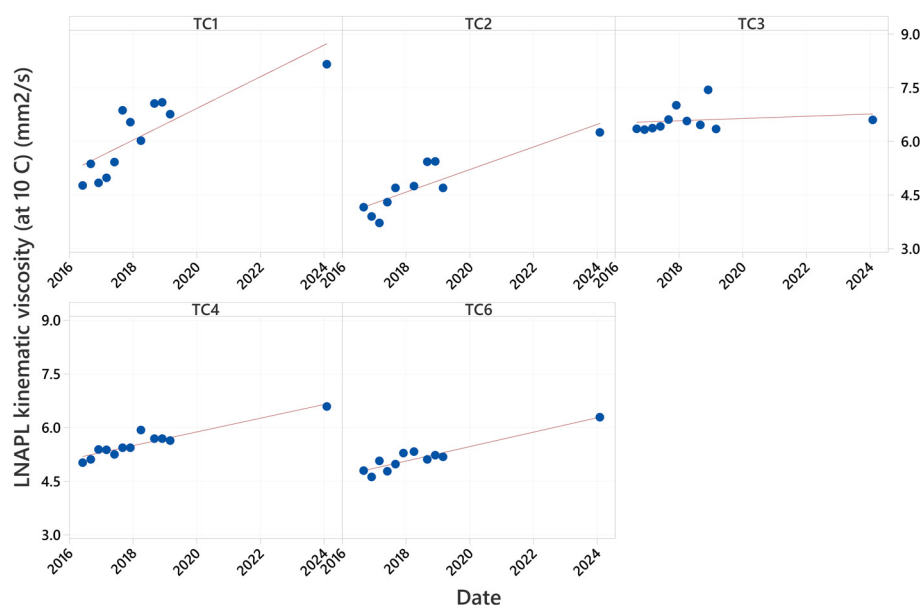


Fig. 2. Temporal change in LNAPL kinematic viscosity (at 10°C) in LNAPL recovery technical chambers (TC).

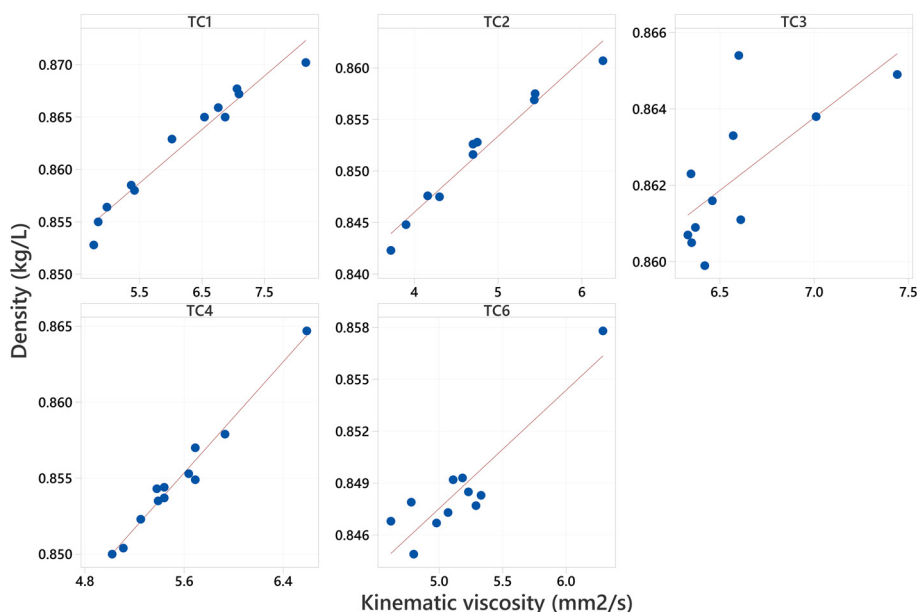


Fig. 3. LNAPL density–kinematic viscosity relationship in LNAPL recovery technical chambers (TC).

observed in all TCs over the period (Fig. 1). Density was measured at both 10°C and 30°C. Measured LNAPL densities were slightly (about 2%) lower at 30°C, but the trends shown in Figure 1 were replicated for 30°C data. Density increased by between 0.7 and 2.2% during the eight-year monitoring period, with R^2 values for the TCs between 0.55 (TC3) and 0.96 (TC6). An implicit assumption of linearity was made in generating R^2 values. For reference, the reported densities fall in the range of API gravity of 31.8° (density of 0.87 kg L⁻¹ at 10°C) to 37.6° (density of 0.84 kg L⁻¹ at 10°C) when converted to API standard temperature (60°F or 15.6°C).

Dynamic viscosity was also measured at 10°C and 30°C, and kinematic viscosity calculated. Figure 2 shows the trend in increasing LNAPL kinematic viscosity at 10°C. Data for kinematic viscosity at 30°C and dynamic viscosity at both 10°C and 30°C showed very similar trends (not presented). LNAPL kinematic viscosity increased by between 4 and 71% during the eight-year monitoring period in the TCs, and R^2 varied between 0.69 (TC1) and 0.88 (TC6), excepting TC3 (0.03). No data were available for the density of the original (pre-release) products, but literature data (TPHCWG 1998; CL:AIRE 2014) suggest diesel has a density in the range of 0.83–0.87 kg L⁻¹, kerosene 0.75–0.84 kg L⁻¹ and gasoline 0.67–0.8 kg L⁻¹. The earliest field density measurements from 2016 lie between 0.84 and 0.86 kg L⁻¹.

Density and kinematic viscosity were observed to be closely correlated in most TCs, with $R^2 > 0.96$ in TCs 1, 2 and 4 (Fig. 3).

Chemical composition

The change in LNAPL chemical composition over time was considered, by comparison of gas chromatograms (Fig. 4), and measurement of the wt% concentrations of the various LNAPL components. Observations of the gas chromatograms identified sequential depletion of lighter hydrocarbon fractions in LNAPL samples collected from TC1 in 2016, 2021 and 2024, which is consistent with typical LNAPL weathering patterns (Brown *et al.* 2017). The results are illustrated with TC1 data (Fig. 5), which show how certain parameters are depleted from the LNAPL over time (e.g. n-heptane, n-octane, methylcyclohexane, 2,3,4-trimethylpentane, ethylbenzene, o-xylene and p- and m-xylenes, 1,2,4-trimethylbenzene), whilst others are absent from the NAPL (<limit of reporting, LOR) for all or most of the monitoring period (e.g. ETBE, isooctane, benzene) in this location. The nC17/pristane and nC18/phytane ratios decrease (through preferential loss

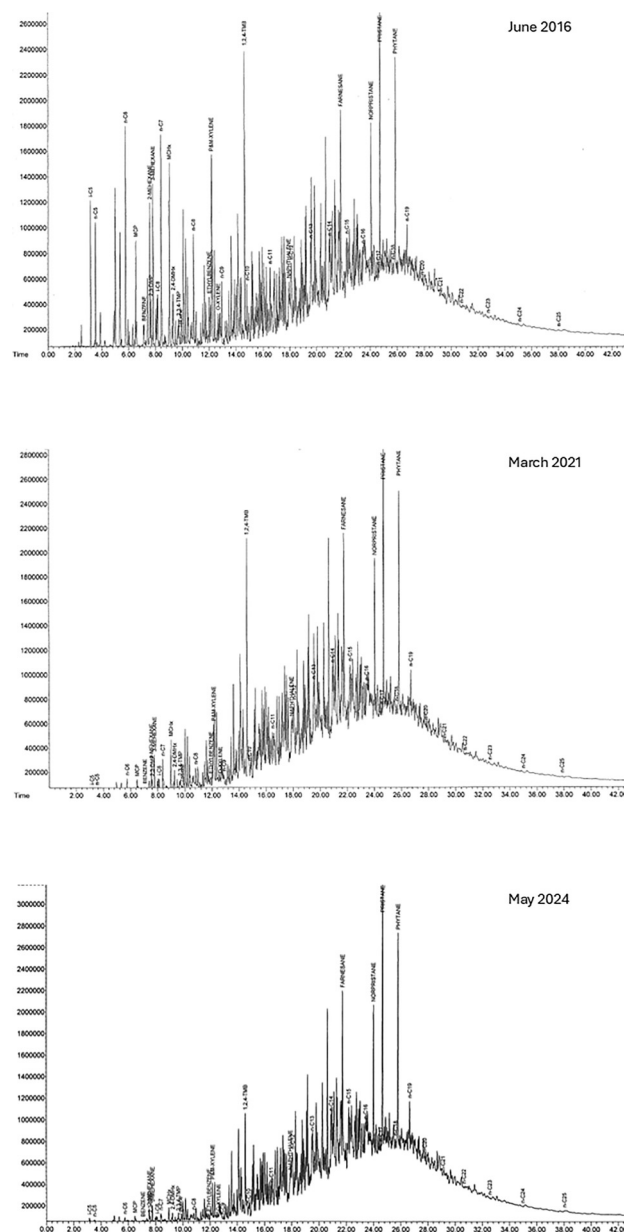


Fig. 4. Gas chromatograms of LNAPL from TC1 sampled in 2016, 2021 and 2024, showing the effects of weathering.

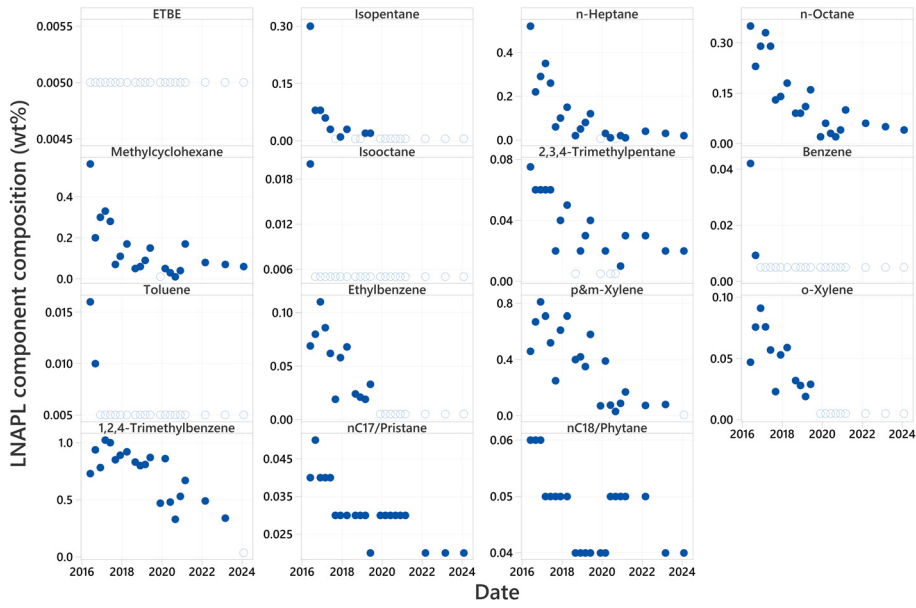


Fig. 5. LNAPL chemistry evolution illustrated for TC1 (wt%, or fraction for nC17/pristane and nC18/phytane ratios). Solid circles are data \geq LOR, open circles $<$ LOR.

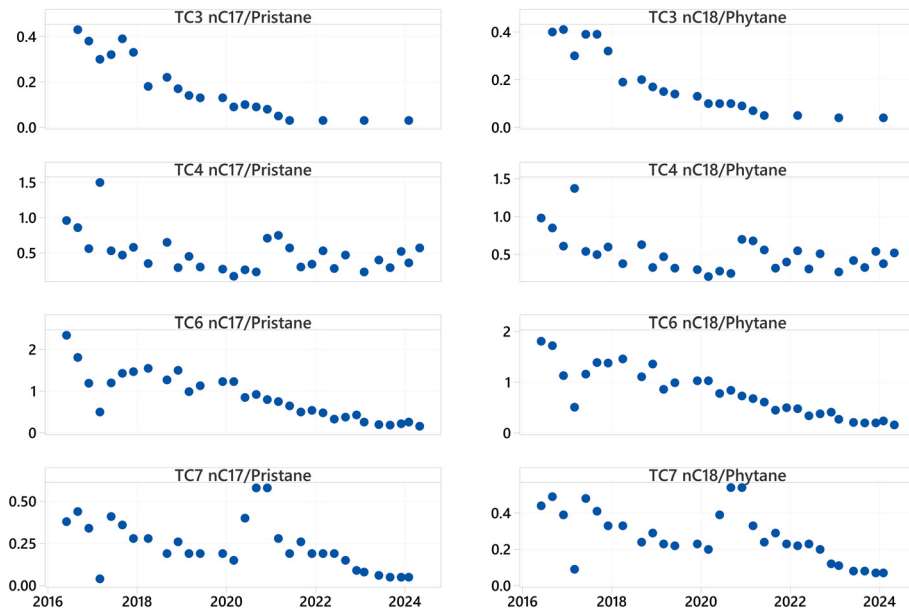


Fig. 6. nC17/pristane and nC18/phytane ratios over time.

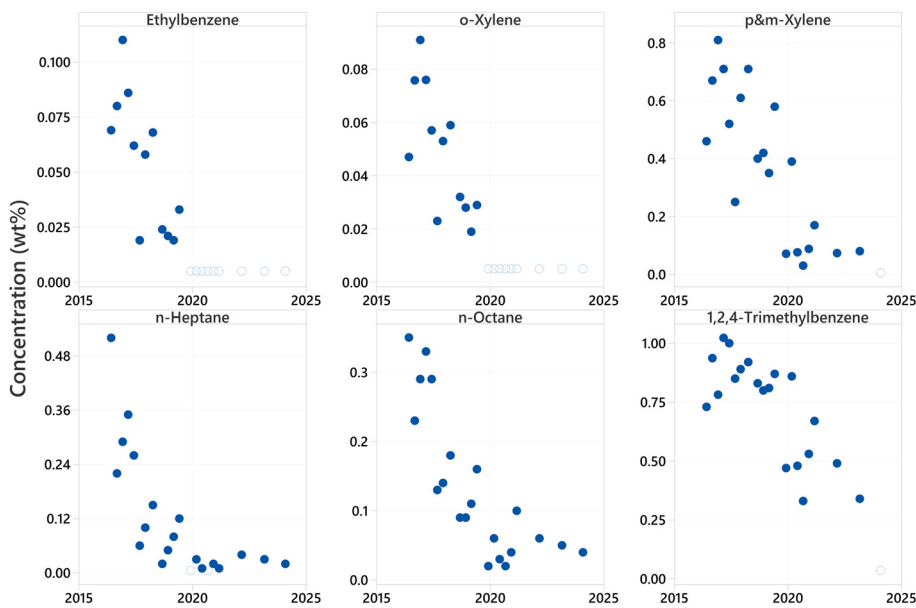


Fig. 7. LNAPL chemical composition changes, TC1. Solid circles are data \geq LOR, open circles $<$ LOR.

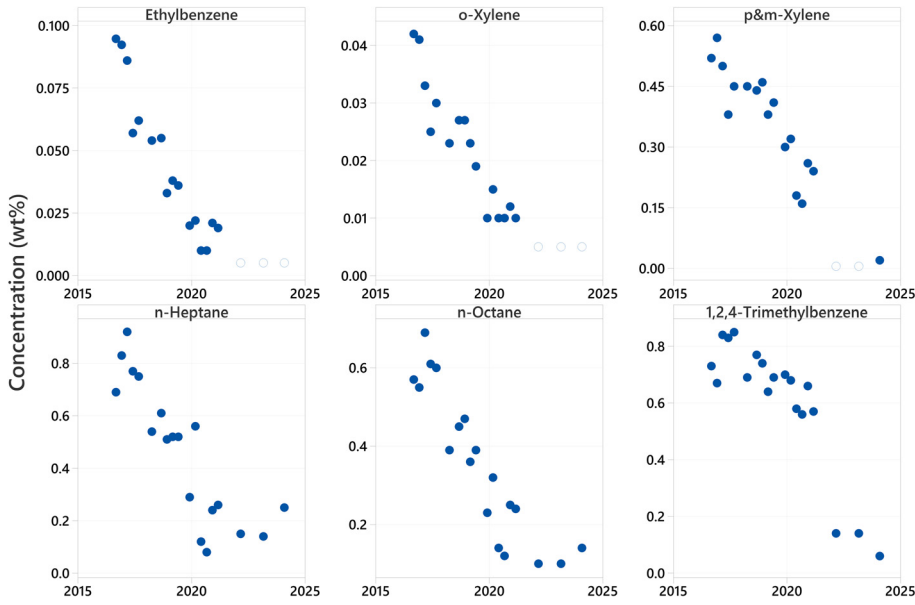


Fig. 8. LNAPL chemical composition changes, TC2. Solid circles are data \geq LOR, open circles $<$ LOR.

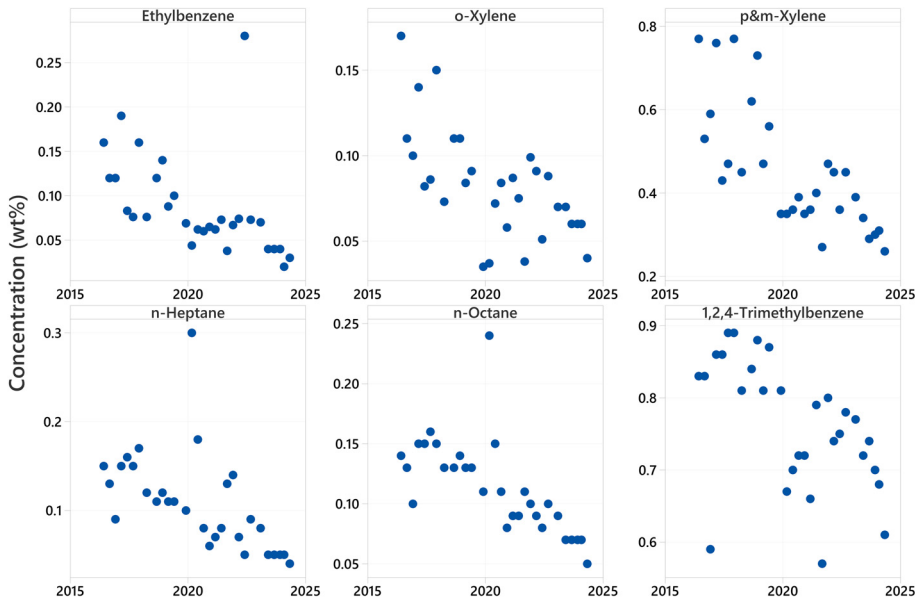


Fig. 9. LNAPL chemical composition changes, TC4. Solid circles are data \geq LOR, open circles $<$ LOR.

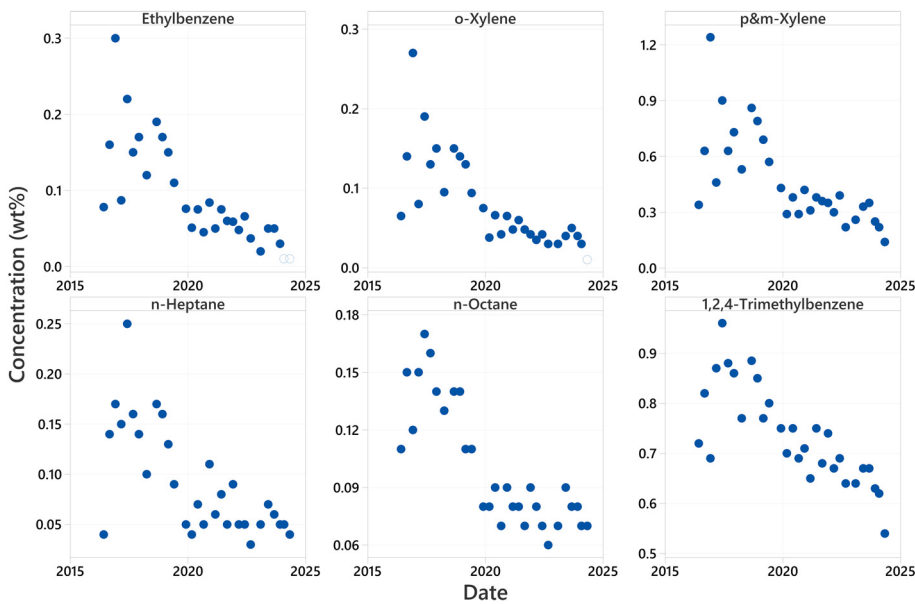


Fig. 10. LNAPL chemical composition changes, TC6. Solid circles are data \geq LOR, open circles $<$ LOR.

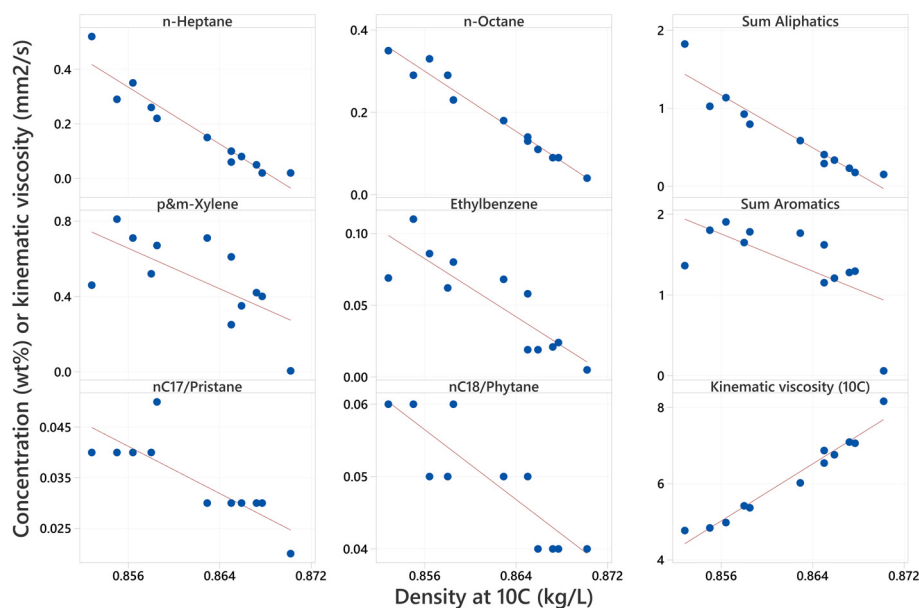


Fig. 11. LNAPL density plotted against chemical concentrations (wt%), nC17 and nC18/conservative marker ratios (unitless) and kinematic viscosity ($\text{mm}^2 \text{s}^{-1}$), TC1.

of the linear alkane) in the case of TC1 but are not suitable for deriving a rate of reduction; in some other TCs, much clearer trends in nC17/pristane and nC18/phytane were observed (Fig. 6).

Comparing the chemical composition changes across all TCs it was identified that the most consistent reductions were observed in n-heptane, n-octane, ethylbenzene, xylenes, and 1,2,4-trimethylbenzene concentrations (Figs 7–10), and in the ratios of nC17/pristane and nC18/phytane (Fig. 6).

Physical and chemical parameters were compared to assess whether changes in viscosity or density could be used to generate a quantitative assessment of NSZD, based on chemical composition change. Illustrating the relationships with data from TC1, it was observed that concentrations of aliphatic compounds most closely correlated with increasing density (R^2 for n-octane = 0.97, for n-heptane = 0.91 and for the sum of six aliphatics = 0.90) (Fig. 11). The six aliphatic compounds were isopentane, n-heptane, n-octane, isooctane, methylcyclohexane and 2,3,4-trimethylpentane. Correlation with aromatic compound concentrations was poor ($R^2 = 0.75$ for ethylbenzene; otherwise <0.45). Similarly, n-octane and n-heptane were the most strongly correlated with both density and viscosity across the other TCs.

An equivalent analysis for LNAPL kinematic viscosity to LNAPL chemistry (Fig. 12) showed similar albeit slightly less strong correlations (R^2 for the aliphatics were: n-heptane 0.91; n-octane 0.94; sum aliphatics 0.80).

Discussion

Data collected from an LNAPL-impacted site showed that LNAPL viscosity and density increased over time. Measurements of density and dynamic viscosity were taken on LNAPL samples at both 10°C and 30°C under laboratory conditions, and kinematic viscosity calculated. Measurements at 10°C and 30°C resulted in slightly differing values (lower viscosity and density at 30°C), but the trends were very similar and generated no additional insight into understanding the NSZD process from undertaking measurements at both temperatures. Measurement of LNAPL density over time generated data that indicated increasing LNAPL density, consistent with NSZD occurring. The increase in LNAPL density and viscosity in TC3 was the lowest of the TCs (Figs 1 and 2), yet estimation of NSZD rates in TC3 by Statham *et al.* (2023) identified this as the highest NSZD rate measured. The LNAPL physical

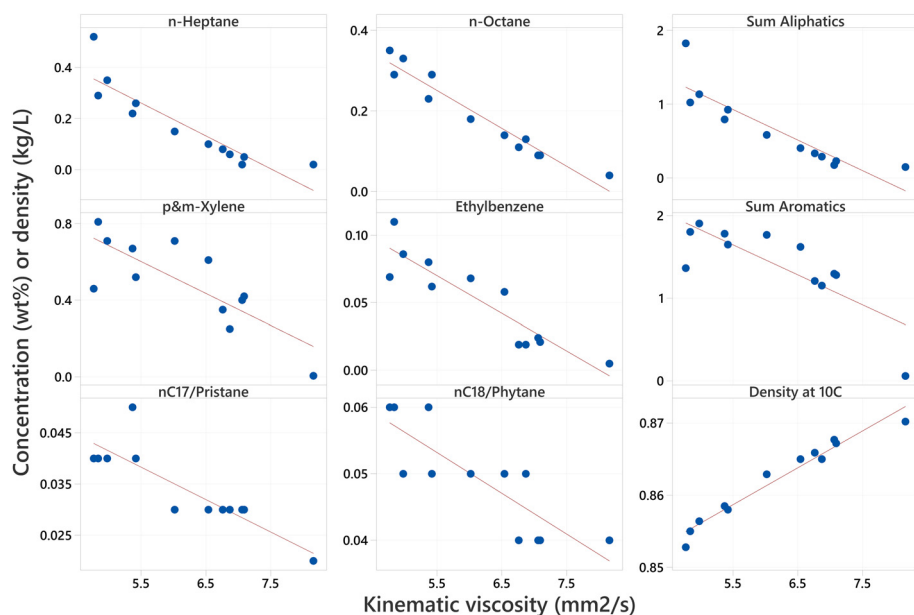


Fig. 12. LNAPL kinematic viscosity plotted against chemical concentrations (wt%), nC17 and nC18/conservative marker ratios (unitless), and density (kg L^{-1}), TC1.

properties may under-represent the effects of NSZD in some circumstances and are best considered a line-of-evidence alongside other data.

Laboratory measurement of LNAPL viscosity also demonstrated increasing viscosity over time. Increases in LNAPL viscosity and density were closely correlated. It was observed that the relative increase in LNAPL viscosity (4–71%) was much greater than the relative increase in LNAPL density (0.7–2.2%) and suggest that if only one measurement is made, it may be easier to see variation and trends in LNAPL viscosity data as the relative change is greater.

Measured alone (i.e. without corresponding chemical analysis), LNAPL density and LNAPL viscosity increases provided qualitative evidence for NSZD that may be included in a NSZD lines-of-evidence analysis.

Decreases in selected chemical concentration in LNAPL were also compared to concurrent LNAPL density and kinematic viscosity increases. Close correlations between the increase of LNAPL viscosity and the depletion of certain n-alkanes were observed. Correlation with aromatic compounds was weaker. In the dataset presented, measurement of LNAPL viscosity and density acted as a quantitative proxy for n-alkane depletion from LNAPL. Landon (1993) reported a similar correlation between physical properties and loss of C4–C12 compounds from a crude oil at the Bemidji spill site in Minnesota, USA. Transfer to other sites would, however, need monitoring of both physical and chemical data to demonstrate similar correlation.

The GC analysis of the LNAPL identified a typical weathering sequence of the lighter component depletion, and a decrease in the height of peaks.

Finally, it was observed that the ratios on nC17/pristane and nC18/phytane showed steady decreases over the eight years of data collection. Pristane and phytane are typically assumed to be conservative markers in fuel (i.e. they are recalcitrant), so the decreasing ratios imply depletion of the nC17 and nC18 hydrocarbons. Whilst data from TC3 and TC6 showed a broadly linear decrease, the results from other datasets are more difficult to estimate a NAPL depletion rate from, so we consider this to be another potential qualitative line-of-evidence, and a topic for future research into the temporal variation in NSZD rates.

Conclusions

LNAPL density and viscosity data measured over a period of eight years are presented for an LNAPL-impacted site. Increases in both LNAPL density and viscosity demonstrated the value of these easily collected physical properties as qualitative indicators of NSZD. Comparison of LNAPL viscosity to depletion of chemicals in the LNAPL also showed close correlation between viscosity increase and alkane depletion from NAPL, such that a quantitative estimate of alkane depletion could be derived based only on viscosity data for the study site.

Whilst LNAPL density increased, it remained well below 1 kg L⁻¹ and the remaining LNAPL body would be expected to continue to be a light NAPL. We do not anticipate that further depletion would cause the residual NAPL to become denser than water, and consequently become a DNAPL. The increased viscosity (and associated surface tension; Hollebhone 2011) over time also means that the oil mobility would also likely decrease, such that the NSZD processes would have the contemporaneous effects of:

- decreasing LNAPL mass;
- decreasing LNAPL mobility; and
- depleting soluble and volatile components that typically drive risk via subsurface pathways.

Applied at other NSZD sites, measurements of LNAPL viscosity and density over time have the potential to provide additional

evidence for NSZD, and with suitably designed investigations may allow quantitative estimates of the depletion of certain LNAPL components.

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Author contributions JWNS: conceptualization (equal), data curation (lead), formal analysis (lead), methodology (lead), project administration (lead), writing – original draft (lead); EH: data curation (supporting), writing – review & editing (equal); AFMH: conceptualization (equal), writing – review & editing (equal).

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Competing interests This work was conducted by employees of Shell companies, which have responsibility for managing a number of LNAPL impacted sites.

Data availability All data generated or analysed during this study are included in this published article (and if present, its supplementary information files).

Correction notice Figures 6 to 10 were attributed to the wrong figure numbers. These figures have now been placed in the right order with their correct caption.

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