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Evaluating the effect of insensitive high explosive residues on soil using an environmental quality index (EQI) approach

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HIGHLIGHTS

- Environmental Quality Indexes developed for Insensitive High Explosives (IHE).
- Impacts of IHE mixture residues on soil environmental status established.
- EQI of the training range soil was reduced by >24 % after 1 month.

GRAPHICAL ABSTRACT

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ABSTRACT

The environmental impact of Insensitive High Explosive (IHE) detonation residues to soil quality was assessed using a series of outdoor soil mesocosms. Two different soils were used including a pristine sandy soil and a land-degraded soil collected from a training range. Both soils were spiked with an IHE mixture comprised of 53 % NTO, 32 % DNAN and 15 % RDX at three different concentrations 15, 146 and 367 mg/kg respectively. The concentration levels were derived from approximate residues from 100 detonations over a 2 week training period. A set of five physico-chemical and biological indicators representative of the two soils were selected to develop environmental quality indexes (EQI). It was found that none of the concentrations tested for the pristine soil affected the chemical, biological and physical indicators, suggesting no decrease in soil quality. In contrast, the EQI for the degraded soil was reduced by 24 %, mainly due to a decrease in the chemical and biological components of the soil. Therefore, it is concluded that depending on the soil health status, IHE residues can have minor or severe consequences on soil health. Further studies are needed to determine the environmental impact of IHE on soil and water especially in the case where a larger number of detonations are more likely to be carried out on a training range.

1. Introduction

Environmental contamination of live-fire military trainings areas has been recognised as a worldwide problem, ranked as the second-largest anthropogenic source of environmental pollution after mining activities (Jenkins et al., 2006; Pichtel, 2012; Tauqeer et al., 2020; Walsh et al., 2005). In addition, new generation explosives, such as Insensitive High Explosives (IHE), are of increasing concern due to their toxicity and early indications that increased quantities will be deposited on soil compared to legacy explosives (Johnson et al., 2017; Taylor et al., 2017). IHE formulations consist of combinations of legacy explosives such as 2,4-dinitroanisole (DNAN) and previously unused energetic materials such as 3-nitro-1,2,4-triazol-5-one (NTO) (Lent, 2019). This mixture is replacing

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the use of Comp B as it less sensitive to unintentional shock with DNANs replacing TNT as it safer during the manufacturing processes (Braida et al., 2012; Singh et al., 2010).

Deposition and accumulation of energetic chemical compounds in soil can occur at live fire training ranges due to repeated field detonation of Insensitive Munitions (IM) (Hewitt et al., 2005; Walsh et al., 2012; Zentelis et al., 2017). In a previous study, we quantified the residual concentration of explosive compounds in soil after three full-order detonations of a 155 mm shell filled with a melt-cast mixture of 53 % NTO, 32 % DNAN and 15 % RDX (Persico et al., 2022). The residual concentrations were then extrapolated to predict the highest residual concentration from 100 detonations, which was estimated to be 370 mg/kg. The three components characterising the explosive mixture, NTO, DNAN and RDX, have a calculated acute LD_{50} (lethal dose estimated to kill 50 % of the population) of respectively >5000 mg/kg, 199 mg/kg and 59 mg/kg respectively (Lent, 2019), Justifying the need to pre-evaluate any explosive residue in soil to pre-empt any potential impact and avoid severe consequences to environmental receptors.

Previous studies have demonstrated the environmental impacts of traditional explosives, such as 2,4,6-trinitrotoluene (TNT) and hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) on soil and water ecosystem functioning and biodiversity (Chatterjee et al., 2017; Stanley et al., 2015; Tauqeer et al., 2020; Travis et al., 2008). In contrast, environmental impact studies for IHE remain scarce. Missing ecotoxicological values and limited understanding of the mechanisms effective for evaluating soil changes in the environment has left a gap in research, and a suitable standardised procedure has not yet been developed.

The consequence of contaminantion on the soil environment is usually evaluated by assessing soil quality, although currently procedures focus on crop production for agricultural land purposes rather than environmental impact (Moebius-Clune et al., 2016; Purakayastha et al., 2019; USDA, 2019). Soil, "*represents the difference between survival and extinction of most land-based life*" and is a "*vital living system*" (Doran and Zeiss, 2000) where the physical, chemical, and biological properties ensure that the system properly functions. Soil Quality Indexes (SQIs) have been developed specifically for this purpose to score the 'healthiness' of the soil (Amacher et al., 2007; Andrews et al., 2002; Chaves et al., 2017). Soil Quality Indexes (SQIs) assess and apply weighting to the physical, chemical, and biological parameters that are essentialto maintain soil integrity for its intended purpose I.e., agriculture, storage etc… (Amacher et al., 2007; Chaves et al., 2017). SQIs use a selection of indicators currently designed to evaluate soil health for land management (Chaves et al., 2017). Each indicator is scored using principal component analysis (PCA); the indicators that are given higher weighing are then scored and used to calculatate the SQI (Mukherjee and Lal, 2014). Therefore, SQI values calculated for different landscapes are not comparable because the indicators are chosen based on the different land management. Therefore, the aim of this work was to evaluate the consequences of IHE residue deposition on soil environmental status using a series of soil mesocosms exposed to outdoor conditions, evaluating soil changes by using an EQI approach. For this work a specific set of soil indicators representing the physical, chemical, and biological parameters of the soil were scored by giving the same relevance to each parameter and avoiding PCA analysis. Ultimately, a new Environmental Quality Index (EQI) approach was developed where all the indicators are scored and considered to quantify the quality of soil to enable comparison between sites to ensure a more comprehensive assessment when different areas are evaluated.

2. Methodology

2.1. Soil characterization and soil preparation

To ensure a comparison between a pristine and a representative soil, a sandy loam soil (Soil A) purchased from SureGreen and a loamy sand soil from an active military training area in the UK (Soil B) were used. Both soils were homogenised by screening through a 2-mm sieve and air dried for 2 weeks at ambient room temperature. Soil B was collected using the multi-increment sampling methodology as it is, to date, the most representative collection technique for a non-homogeneous contamination (ITRC, 2012). Both soils were characterised (Supplementary material 2 - S1) using particle size distribution according to STM D 2487-11, soil pH and Electrical Conductivity were measured on a Jenway 3540, Carbon (organic and total) (BS 7755-3.8:1995, ISO 10694:1995), total hydrogen and total nitrogen (BS EN 16168:2012) were measured on an Elementar vario EL cube (Temple et al., 2019).

2.2. Preparation of soil

Both air dried soils were spiked with an IHE solution at either low, medium, or high concentration as determined in Persico et al., 2022 (Table 1). Briefly, IHE flakes (12.5 mg (low), 1250 mg (medium) and 3375 mg (high)) were dissolved in 5.8 L of distilled water at pH 7 for 2 weeks using a Heidolph MR3002 agitator with a magnetic bar. The volume of water for dissolving the IHE flakes was determined by the solubility of RDX (66 mg L^{-1}) at the maximum concentration of the experiments, being the compound with the lowest solubility (Lent, 2019). The soil (8 kg) was then added to the IHE solution and frequently mixed over 3 weeks under a fume cupboard in black containers until excess water had evaporated. The soil mixture was prepared in black containers away from direct sunlight to limit any photodegradation of the explosive mixture.

2.3. Mesocosm experiments

Sealed buckets were used to prepare the mesocosms by cutting a hole in the lid (20.5 × 15.4 × 5.0 cm) to accommodate commercially available black plastic seed trays, which were sealed with silicon to ensure any collected rainwater first passed through the soil (Supplementary material 1).

Each tray (36 in total) was first filled with 0.5 cm of damp inert quartz sand to avoid contact between the soil and the bottom of the tray and minimise any soil loss through the holes at the bottom of the trays. Spiked soil (8 kg) was then poured into each tray to a depth of 5 cm (Supplementary material 1) and final weight recorded. The depth of soil was determined by the depth of the top layer of soil (5 cm), as this is where any change to soil properties due to surface contamination is expected to be observed (Blume et al., 2016). For low, medium and high contamination 5 contaminated samples, and 3 positive controls (clean soil) were prepared.

Samples were sacrificed at day 0, 1, 7, 14, and 36 and prepared for analysis. Leachate volume was collected in triplicate using 50 mL amber glass tubes to avoid photodegradation of the IHE. For biological analysis, soil (5 g) was collected from each mesocosm in triplicate using a 5 mL sterile vials and 20 μL of glycerol was added to preserve the soil for biological analysis. Soil samples were stored at −70 °C until biological analyses were carried out. The remaining soil (1 Kg for each mesocosm) was stored at −18 °C for physical and chemical property analysis and leachate was collected and stored at 4 °C until chemical analysis was carried out.

2.4. Weather condition data collection

The mesocosms were set up during the UK summer months (July–August) and weather data was recorded during this time from the closest

Table 1
Measured spiked concentration of low, medium and high concentrations used for the soil mesocosms.

	Soil A			Soil B		
	Low	Medium	High	Low	Medium	High
53 % NTO (mg/kg)	8.1	82	194.9	8.1	81.1	196.7
32 % DNAN (mg/kg)	4.9	49.5	117.7	4.2	49	118.8
15 % RDX (mg/kg)	2.3	23.2	55.2	2.3	23	55.7
IHE total concentration in soil (mg/kg)	15.3	154.6	367.7	15.3	153.1	371.2

weather station (Marlborough, 22 km from the sample collection area). The data was recorded daily and included minimum and maximum air temperatures, rainfall in mm, wind speed and direction, evapotranspiration, solar energy, direct sunshine, humidity, and temperature of the first 5 cm of the soil. The data is publicly available at https://www.windrushweather.co.uk/station/.

2.5. Soil analyses

2.5.1. Explosive extraction and analysis

Soil collected from the mesocosm was air dried. From each collected mesocosms triplicate soil samples (10 g) were collected using multi-increment sampling. The explosive was extracted from the soil using the Temple et al. (2019) method. Briefly, a mixture of acetonitrile/water (1,1) (20 mL) was added to soil (10 g) and shaken for 18 h at 180 rpm in 50 mL amber glass tubes. Samples were filtered using 0.2 μm polyether sulfone (PES) filter and analysed by High Performance Liquid Chromatography (HPLC). Water samples (50 mL) were collected in triplicates, filtered using nylon filters (0.2 μm) and analysed by HPLC as described by (Temple et al., 2019). Quantification of NTO, DNAN and RDX was carried out using a calibration curve. The Limit of detection and limit of quantification are reported in Table S2 (Supplementary material 2).

2.5.2. Soil physical properties determination

Due to the nature of the sample, qualification but not quantification of changes of the physical properties was determined. Therefore, variations were evaluated on air-dried samples.

Bulk density (ρ_b) (kg/m^3) was calculated following the formula (1):

$$\rho_b = {}^{M_s}/_{V_s} \quad (1)$$

where M_s is the mass in mg of the soil sample, which was calculated by weighing the dry soil sample. V_s was calculated as the volume of the dry soil sample in m^3 (Han et al., 2016). Soil Particle density (mg/m^3) (SPD) was instead determined according to STM standards (D854 – 14) although, due to the nature of the samples, the soil was air dried instead of oven dried, therefore results have variability in SPD. Those calculations were necessary for the soil porosity variation (%) which was derived from the soil bulk density and the soil particle density. Soil porosity was calculated as follows:

$$f = \frac{1-\rho_b}{\rho_s} x\ 100 \quad (2)$$

with ρ_b as the bulk density in g/cm3 and ρ_s as the specific gravity of soil solids (or soil particle density) (Hazelton and Murphy, 2016).

2.5.3. Soil chemical properties determination

pH was recorded from soils (1:5) and leachate water using HI-98100 Checker Plus pH Tester. Electrical Conductivity (EC) was measured (mS/cm) in soil using HI-98331 Groline Direct Soil Conductivity & Temperature Tester. Soil nutrients were analysed using hatch test kits (Hanna Instruments - HI-3895) for qualitative evaluation of P, N and K variability. A colorimetric (P, N) and turbidimetric (K) evaluation was used to measure trace, low, medium, and high levels of nutrients concentration to determine their variability. Distilled water (30 mL) was added to soil samples (10 g) together with the additive powder from the kit, stirred and allowed to settle for 30 min. Potassium Availability (K^+) was measured in ppm following the procedure from (Motsara and Roy, 2008). Potassium Chloride (KCl) (1.907 g) was dissolved in Ammonium Acetate/Acetic Acid solution (50 mL) and standards were prepared ranging from 10 to 100 ppm. The acetate/Acetic Acid solution (50 mL) was added to soil (10 g), and. The samples shaken for 30 min. The solution was than filtered using a Whatman No.30 filter paper and the leachate analysed using a Flame Photometer.

2.5.4. Soil biological properties determination

The Viable Plate Count technique (Jett et al., 1997) was used to evaluate the number of bacteria present in the original soil samples to observe the variability at different IHE concentrations. Nutrient agar (23 g) was added to distilled water (1 L) and autoclaved at 121 °C for 1.30 h. The warm agar was added to sterile polystyrene plastic petri dishes (55 × 15 mm) and left to dry in a sterile environment. Soil (1 mg) was added to Phosphate Buffered Saline (PBS) (100 μL), the samples were diluted up to 9 times to enable visual counting of the bacteria colonies. The diluted solutions (up to 10^{-10}) were spread on the petri dishes which were then left in an incubator at 37 °C overnight. The standard deviation calculated for all results was found to be ≤9.7 % for colony forming units (CFU) estimation.

2.6. Environmental quality index (EQI)

Each of the soil indicators were scored following the procedure for the SQIs (Andrews et al., 2002; Chaves et al., 2017) although avoiding the PCA selection to ensure that all the values were considered. This is because SQIs between different sites are not directly comparable as the soil indicators of a particular site/area tend to be site- or area-specific. Therefore, weighted soil parameters might preclude a comparison between sites (Qi et al., 2009; Wienhold et al., 2004).

Based on previous research the most common set of indicators used to assess the impact of different contaminant on soil are listed in the indicators column in Table 2.

The average value from the six indicators used for each IHE concentration and day of collection was scored based on Amacher et al. (2007). This provided a list of scorings for SQIs based on the calculated experimental values (Supplementary Material 1). For the values where a score was not provided the "low is better" and "more is better" function was used (Lenka et al., 2022). This approach, by using the same set of indicators as of equal importance, ensures comparison between different sites.

Following the scoring from each physical, chemical, and biological indicator (Supplementary material 1), separately, the EQI of each parameter were summed:

$$EQI_{p/c/b} = S_1 + S_2 + S_3 + S_4 + S_5 \quad (3)$$

where S represents the score for each parameter. If one of the previous indicators from Table 4 was not analysed, 0 was the score given.

The EQI was calculated and normalised as follows:

$$EQI = \left({}^{EQI_p}/_{I_p}\right) + \left({}^{EQI_c}/_{I_c}\right) + \left({}^{EQI_b}/_{I_b}\right) \quad (4)$$

Table 2
Commonly used soil indicators to assess the effects of contamination and/or land use management on soil. The highlighted indicators were used for scoring the physical chemical and biological parameters in the EQI.

		Indicators	Reference
Physical properties	1	**Bulk density**	Williams et al. (2020)
	2	Water holding capacity	Williams et al. (2020)
	3	Porosity	Yu et al. (2020)
	4	Water infiltration rate	USDA, (2019)
	5	Aggregate stability	USDA, (2019)
Chemical properties	1	**pH**	Neina (2019)
	2	**Electrical conductivity**	Awale et al. (2017)
	3	**Cation exchange capacity**	Chaves et al. (2017)
	4	**Nutrient availability**	Cardoso et al. (2013)
	5	Soil organic carbon	Lehmann and Kleber (2015)
Biological properties	1	**Microbial biomass**	Niemeyer et al. (2012)
	2	Microbial biodiversity	Schloter et al. (2003)
	3	Microbial activity	Schloter et al. (2003)
	4	Soil respiration	Cardoso et al. (2013)
	5	Earthworms	Pervaiz et al. (2020)

where EQI is the Environmental Quality Index for the physical, chemical, and biological properties, I_p/c/b are the numbers of physical, chemical, and biological indicators considered. The EQI was calculated for the control, low, medium, and high concentrations for both soils for each day of collection (0-1-7-14-36).

2.7. Statistical analyses

Regression analysis were carried out on data related to the physical, chemical, and biological properties analysed using Excel (MS Office) to evaluate the relationship between each variable and their dependencies. Results are shown using draftsman plot (Fig. 1) to understand and display the distribution and patterns of the data. Moreover, using SPSS (IBM, USA), two-way ANOVA tests were carried out to analyse the dependency of each variable on the explosive concentration that was spiked on the soil mesocosms.

3. Results

3.1. Physical properties

Bulk density and soil particle density (SPD) were measured at day 1 and 36 for the control and the high contaminated samples as these parameters are least affected by the presence of IHE in a mesocosm study. For both soils, the SPD and the Bulk Density were higher at day 36 for the high contaminated samples compared to the controls, with a difference of 29 % for Soil A and 42 % for Soil B. All the calculated values, with the exception of the control sample for Soil B, were higher than 1.8 g/cm^3 which is a critical value for sandy loam soils as it suggests an extremely compact soil where root penetration would be restricted (Hazelton and Murphy, 2016).

3.2. Chemical properties

In Soil A (Fig. 1), on average, the control soil had a 62 % lower EC value compared to the high contaminated sample (1.83 mS/cm), indicating that the presence of IHE influenced the soil matrix. This was confirmed by the difference in EC detected in soil B (Fig. 1), in which the high contaminated samples had an EC 39 % higher compared to the controls. Overall, for both soils, EC characterised the matrices as non-saline. EC is also influenced by the amount of water in the soil, therefore after 15 days of non-registered rainfall the EC was 0 mS/cm for all samples.

Potassium ion concentration had a stronger correlation with the medium IHE soil contamination levels with r^2 of 0.81 and 0.57 for Soil A and B, respectively. In contrast, a poor correlation was found for the soils spiked with high IHE levels (r^2 = 0.005 and 0.0008 for Soil A and B) suggesting that when a higher nitrogen content is spiked into the soil K + is less likely to interact with the IHE molecule.

A clear correlation was observed between the different IHE concentrations and the soil pH in both soils (r^2 = 0.74 for Soil A at the high contaminated sample). This correlation was expected as it has been shown that pH influences IHE behaviour in soil (Mark et al., 2016; Temple et al., 2019; Wallace et al., 2011). The pH of the leachate from Soil A remained stable during the experiment. While the pH of the leachate from Soil B increased by 1 %, this increase was recorded for all samples including the controls and therefore cannot be attributed to the IHE content.

Qualitative assessment of nutrients (Table 3) showed a greater change in N and P for Soil B. Soil A had a consistent decrease across all samples in Nitrogen, compared to Soil B where the N behaviour was more variable. For the high and medium concentration samples Nitrogen decreased after day 1, although an increase of N across all samples resulted in a high N concentration at day 7. Soil A and B were also lacking in Phosphorus, which was mostly non detected across both soils.

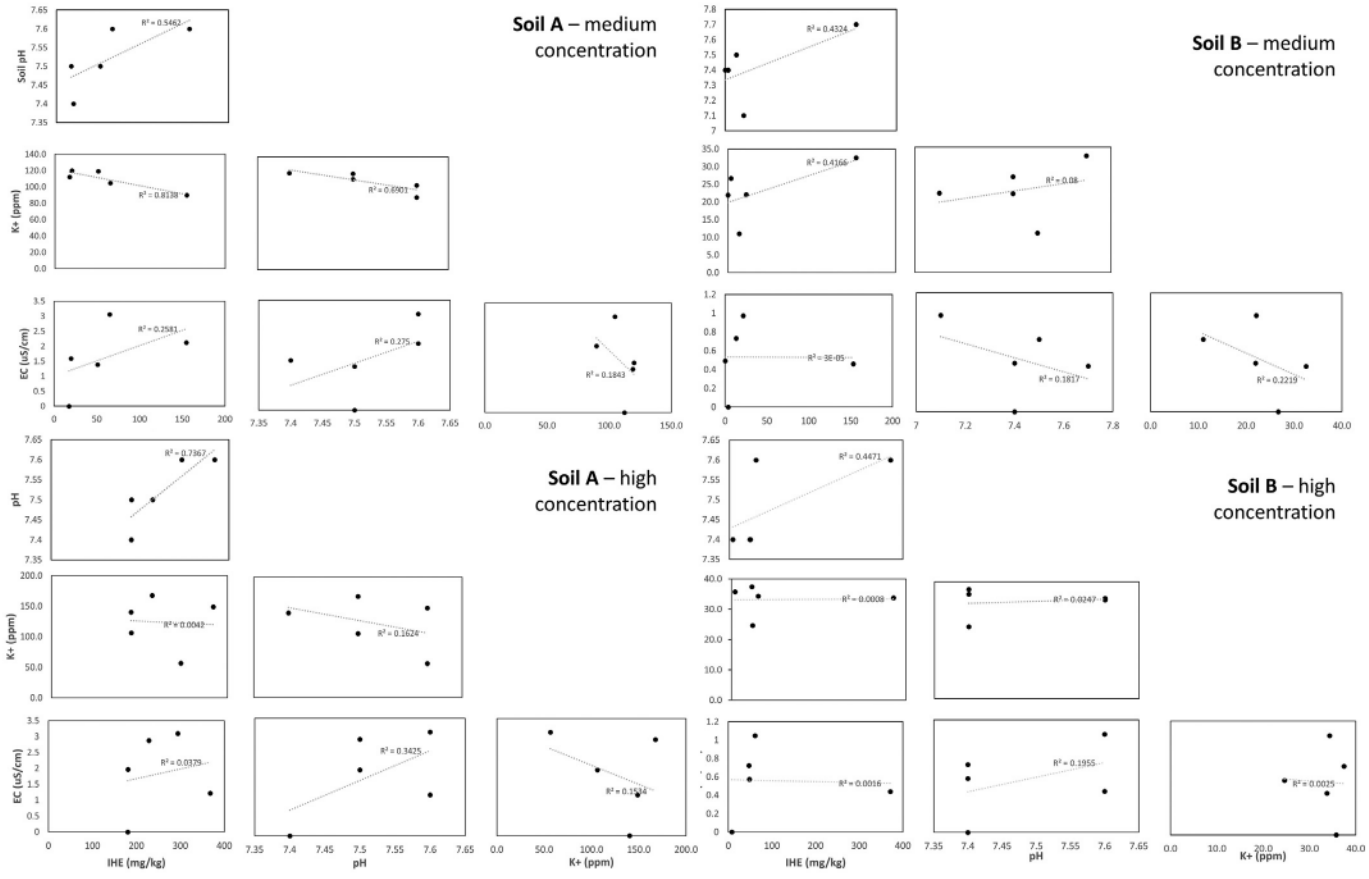


Fig. 1. Chemical properties and relationships between pH, EC, IHE and K^+ in Soil A and Soil B in presence of medium and high IHE concentrations (n = 56 per soil).

Table 3
Qualitative assessment of N, K and P from the control, low, medium, and high IHE contaminated soil samples.

	IHE contamination	Nutrient	Day 0	Day 1	Day 7	Day 14	Day 36
Soil A	Control	N	Low	Medium	Trace	Trace	Trace
		K	High	High	High	High	High
		P	Trace	ND	Low	ND	ND
	Low	N	Low	Trace	Low	Trace	Trace
		K	High	High	High	High	Medium
		P	Trace	ND	ND	Low	Low
	Medium	N	High	Low	Trace	Trace	Trace
		K	High	High	High	Medium	High
		P	Trace	ND	Low	ND	Trace
	High	N	Medium	Low	Medium	Low	Medium
		K	High	Medium	High	High	High
		P	Trace	ND	Low	ND	Trace
Soil B	Control	N	Low	Medium	High	Medium	Trace
		K	Trace	Medium	Trace	Trace	Trace
		P	ND	ND	ND	ND	ND
	Low	N	Low	Low	Medium	Trace	Trace
		K	Medium	High	Medium	Trace	Trace
		P	ND	Trace	ND	ND	ND
	Medium	N	High	ND	Medium	Trace	Trace
		K	Trace	Medium	Medium	Trace	Trace
		P	ND	ND	ND	ND	ND
	High	N	Low	ND	Medium	Trace	Medium
		K	Trace	Low	Trace	Trace	Trace
		P	ND	ND	ND	ND	ND

As confirmed from the K^+ availability experiments, total potassium was significantly higher in Soil A compared to Soil B. Total K was also more stable in Soil A (Table 3), differently from Soil B where an increase in K was determined after day 1 following a regular decrease in all analysed samples.

3.3. Biological properties

Soil microbial abundance based on CFU analysis indicated a significant relationship in Soil A between the IHE concentration over time and the CFU ($p < 0.05$). Soil A CFU increased after 7 days and a decrease from day 7 to 36 (Fig. 2). The decrease was more significant for low and high concentration samples. A two-way ANOVA also demonstrated that there was a stronger statistical significance between IHE concentration, soil pH and CFU ($p < 0.05$).

The same correlation has been found between the rainfall and the CFU in Soil B even though there is a difference in behaviour for the high contaminated sample. Conversely to Soil A, no significant relationship has been found between the IHE mixture concentration and the CFU counted. Instead, this relationship was found between the soil pH and the CFU (ANOVA $p < 0.05$) (Supplementary material 1).

3.4. Weathering and IHE concentration effect on soil

The experiments were conducted during the summer season in the UK with an average temperature expected to be around 15.6 °C, based on the 1981–2010 average. The recorded average temperature was 16.6 °C with soil temperature recorded, within the first 5 cm, to be 15.3 °C. Unexpectedly, during the experiments, rainfall was not registered for 19 days, of which 15 were consecutive (from day 21 to 36). The highest rainfall was registered on day 19 (17.7 mm) with an average rainfall throughout the experiment of 1.6 mm (Fig. S1).

Rainfall influenced the transport of chemicals within the matrix, with a rise in leachate when the rainfall was at high levels which could increase the transport of the chemicals from soils to groundwater systems (Zhang et al., 2019). As the rainfall was higher in the first part of the experiment (from day 0 to day 20), a higher dissolution and transport of the IHE mixture was expected during this time. Due to the differences in solubility of the three IHE constituents NTO: 17200 mg/L; DNAN: 216 mg/L, and RDX: 59.7 mg/L (Lent, 2019), it was expected that a higher there would be higher concentrations of NTO and DNAN in the leachate during rainy periods compared to RDX.

Moreover, the amount of rainfall was also associated with a decrease or increase in microbial activity which sees a decline in microbial communities when rainfall is lower (Wu et al., 2020). The CFU calculated in this experiment highlighted the dependencies of the microbes on rainfall, with a strong relationship determined in Soil A ($r^2 = 0.8$). Consequently, due to the rainfall, which was not registered in the second part of the experiments, a decrease is CFU was detected in all samples.

From day 0 to 36, the IHE composition was extracted and NTO, DNAN and RDX independently assessed (Fig. 3). In the controls (non-contaminated soil), no IHE concentration were found for both Soil A and B. The concentration at day 0 was the day on which the experiments were set up and therefore used as the baseline for comparison. Due to the three-week preparation time to enable evaporation of excess water, the concentration of IHE detected was lower at Day 0 than the initial spiked concentration. Therefore, in all the samples contaminated with the lower concentration of IHE (15.34 mg/kg) only 38 % and 6 % of the concentration, respectively for Soil A and B, was recovered after 1 day of experiment with the concentration falling to 0 mg/kg at day 7.

In Soil A, at the medium concentration (Fig. 3), 23 % of the IHE mixture was recovered on day 0 of experiment, with a decrease and then a stabilisation of the IHE mixture in the soil which was recovered with an

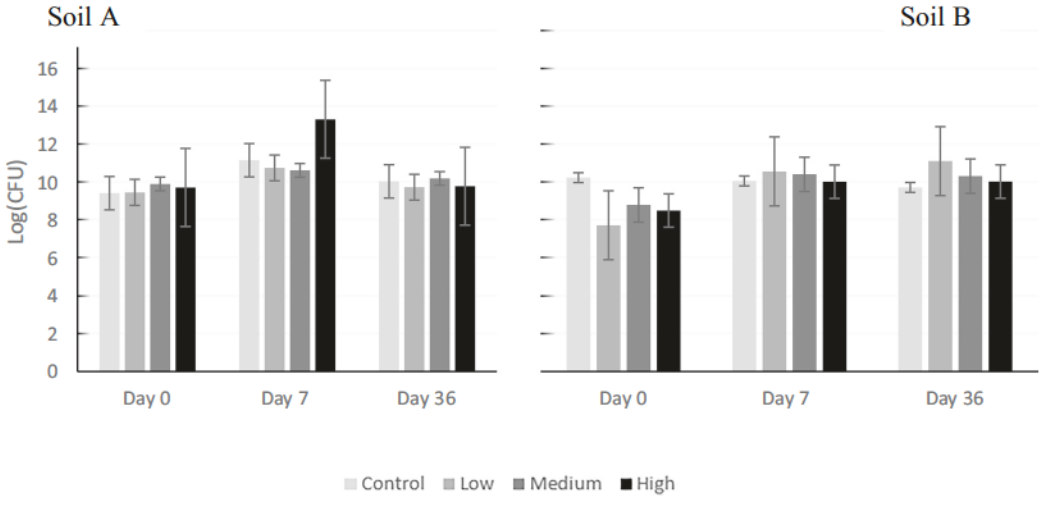


Fig. 2. CFU/mL at day 0–7–36 presented in a $\log_{10}$(CFU) scale ($n = 54$ per soil).

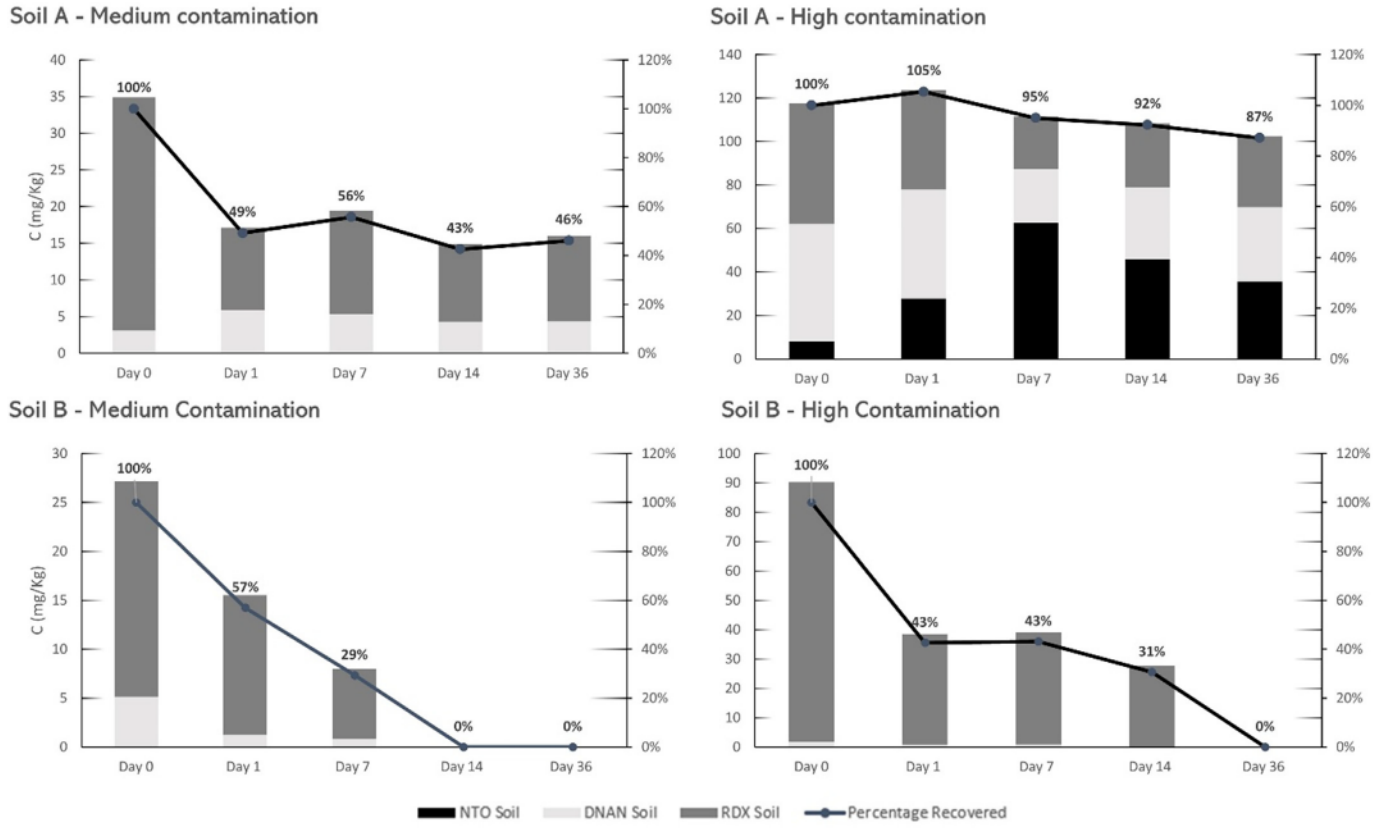


Fig. 3. The concentrations in mg/kg of NTO, DNAN and RDX recovered soil for Soil A and B at the medium and high concentration. The percentages on the graph represent the total % of the IHE formulation recovered from day 0 to 36.

average of 48 ± 5 % from day 1 to day 36 compared to day 0. NTO, being the most soluble of the three compounds, was not recovered in soil, although it was found in its original form in the leachate at day 1 (44.7 mg/L) and day 14 (33.5 mg/L). DNAN was recovered in all samples with an average concentration detected of 4.6 ± 1.1 mg/kg in soil A. As expected, RDX was the most stable with a concentration that varied from 31.8 mg/kg at day 0 to 11.7 mg/kg at day 36. RDX was also recovered in water at each day of experiment ranging from 2.6 to 0.5 mg/L.

At high concentration in Soil A, a higher percentage of the explosive mixture was recovered at day 0 (117.6 mg/kg) with no degradation during the three weeks of preparation of the RDX compound and low degradation of NTO and DNAN, which was recovered in the leachate at day 1 (Table 4). The total degradation in soil from day 0 to day 1 was negligible with a recovery of 5 % higher than at the beginning of the experiment. This recovery highlights the difficulty in analysing IHE compounds, with NTO being the most difficult to detect, therefore an error was expected as highlighted in previous work (Temple et al., 2019) due to the heterogeneous nature of the soil matrix. NTO was recovered in high quantities in the water leachate ranging from 163.7 mg/L at day 1 to 73.2 mg/L at day 36, emphasizing its rapid transport through soil due to the diminishing leachate concentration over time. DNAN was not significantly detected in water (0.6 mg/L at day 1) with only 37 % decrease in recovery from day 0 to day 36. 55 % and 68 % of the total RDX was recovered respectively at the medium and high concentration after 36 days (12.8 mg/kg; 36.7 mg/kg).

In Soil B at the medium concentration only 18 % of the IHE mixture was recovered at Day 0 with a further decrease in soil of 42 % from the start of the experiment to day 1 (15 mg/kg) and 71 % at day 7 (8 mg/kg). NTO, DNAN and RDX were also detected in the collected leachate for the medium concentration samples. NTO was not recovered in soil but was detected in water at day 1 (3.1 mg/L) and day 7 (3.0 mg/L) demonstrating it low adsorption to Soil. DNAN decreased in concentration from day 0 to day 1 by 77 %. DNAN was not detected in water leachate until day 36 when a small amount (0.3 mg/L) was recovered. RDX decreased by 35 % on day 1 and 67 % on day 7, compared to the concentration detected at day 0 (22 mg/kg). From day 14 to day 36 RDX was not recovered from soil, although 3.3 mg/L was detected in water leachate at day 36. The increased rate of transport of RDX in soil B compared to Soil A can be attributed to the decreased organic content in the matrix.

No NTO was recovered from the high concentration samples of Soil B, although some explosive was recovered in water at day 1 (7.2 mg/L) and day 7 (2.3 mg/L). This suggested that NTO underwent chemical changes, as it was not found in either soil and only 6 water samples in its original form. DNAN recovery was lower at day 1 and similar at day 7, compared to the medium contaminated samples of Soil B. It is likely that degradation was more rapid in Soil B compared to soil A as DNAN was recovered in much lower quantities in its original form. A higher recovery of RDX from Soil B compared to the medium concentration was detected, which remained stable until day 14, with 31 % of the explosive recovered compared

Table 4
IHE recovered from the collected leachate for the medium and high concentration in mg/L.

	Soil A			Soil B		
	NTO	DNAN	RDX	NTO	DNAN	RDX
Medium concentration						
Day 1	44.7 ± 3.3	N.D.	2.6 ± 0.2	3.1 ± 0.2	N.D.	3.14 ± 0.66
Day 7	N.D.	N.D.	0.5 ± 0.07	3.0 ± 0.8	N.D.	2.2 ± 1.46
Day 14	33.5 ± 0.3	N.D.	2.7 ± 0.07	N.D.	N.D.	N.D.
Day 36	N.D.	N.D.	1.2 ± 0.02	N.D.	0.3 ± 0.3	3.3 ± 0.09
High concentration						
Day 1	54.7 ± 94.4	0.4 ± 0.1	2.1 ± 3.3	7.1 ± 1.9	N.D.	2.7 ± 1.3
Day 7	75.5 ± 20.8	N.D.	3.7 ± 0.6	2.3 ± 3.5	N.D.	20 ± 13.9
Day 14	70.4 ± 8.5	N.D.	2.1 ± 0.02	N.D.	N.D.	19.2 ± 0.7
Day 36	73.2 ± 15.9	0.7 ± 0.2	4.9 ± 0.2	N.D.	N.D.	8 ± 0.5

to day 0. Although, no RDX was detected at day 36 in soil. RDX was regularly recovered in the water leachate with an average of 12.5 mg/L.

It is worth noticing that even though IHE has was not detected in some of the samples, the concentration may have been lower than the limit of detection (<0.004).

3.5. Preliminary environmental quality index (EQI) calculation

Each parameter was scored following Amacher et al. (2007) to provide a list of SQIs based on the calculated experimental values and theoretical values taken from Lenka et al., 2022 when scores could not be derived (Table 5).

Nutrient values obtained were then averaged. SOC values were scored as "0" (Table 5) as soils could not be oven dried due to the explosive nature of the samples. DNAN degrades at approximately 200C therefore the procedure could have impacted the nature of samples giving false results., Therefore SOC values were not fit-for-purpose for the EQI although more research is needed for application of SOC calculation methodologies to ensure that quantitative analyses are carried out.

The biological, chemical, and physical scores were summed according to formula (2) (Supplementary material 2 - Table S3). Following the scoring, data were added up and normalised based on the number of indicators that were analysed experimentally. The normalised scores were summed up following formula (3) and an EQI was obtained for each concentration and day of experiment (Supplementary material 1).

Due to the limitations of analysis of the explosive contaminated samples the chemical indicators were the most influential parameters in the EQI as chemical parameters were mostly analysed for the preliminary EQI, respectively 60 % and 76 % on average for Soil A and Soil B, followed by the biological section (23 % for Soil A and 20 % for Soil B) and the physical indicators (1 % for both Soil A and B). Therefore, because further studies are needed to understand how to overcome the explosive contaminants issues for some analyses, the EQI obtained are preliminary values. Although, these values will provide insight on possible influences of explosives on soil determining a baseline until further analyses can be done to have more comprehensive values.

In both soils (Fig. 4), the EQIs had similar variation, ranging from 0.63 to 1.87 for Soil A and 0.60 to 1.80 for Soil B. The EQI calculated for the non-contaminated samples was, as expected, lower for Soil B, compared to Soil A, as Soil B is a land degraded matrix. Although, the EQIs for Soil A and B did not decrease overtime having only a 1 % difference between the first to the last day of collection. As expected, as the contamination in the samples increased so did the differences in EQI between the soils. At the low, medium, and high concentration Soil B average values were 6 %, 20 % and 24 % lower respectively compared to Soil A at the same concentrations. In fact, the difference between the highest affected sample in Soil A (with the lowest EQI on average – low contaminated samples) and the control, differed by only 1 % compared to Soil B where this difference was 24 %. Overall, the EQI increased for all the samples at Day 7, again highlighting the importance of the weather conditions as a main influential factor for the analysed properties.

Chemical properties, which were the most studied in this work, significantly affected the outcome of the EQI and further work is needed to increase the amount of data available for the physical and biological values. However, these preliminary results have shown how Soil B was increasingly affected by the IHE contamination, subsequently increasing the rate of degradation. In Soil A, this difference could not be determined, meaning that the explosive components are not currently affecting the soil properties at the studied concentrations.

4. Discussion

The increased interest in understanding soil changes due to IHE exposure has been led by a rise in usage of these new explosive mixtures and further discoveries of soil surface and groundwater contaminations (Jenkins et al., 2006; Morley et al., 2006; Temple et al., 2018). The fate and transport of the IHE compounds is guided by the soil composition (Monteil-Rivera et al., 2021; Taylor et al., 2017; Temple et al., 2018) is influenced by the soil properties. Sandy Loam and Loamy Sand, which have been named Soil A and B in this research, have many physical similarities but were characterised by more significant chemical differences. Soil A had on average the highest K+ content, pH, and EC. Moreover, because Soil B was collected from a training range, degradation of the matrix led to a different relationship with the chemical compounds (Tetteh, 2015) which were spiked for the evaluations.

Because of the dependent relationship between K and N components in soils (Johnston and Milford, 2012), which increases N adsorption as K availability rise, Soil A retained NTO, DNAN and RDX more compared to Soil B, due to an increase in adsorption processes which has been also consistently reported for DNAN in the literature (Linker et al., 2015). This retention was strengthened by the high pH (8.7) which increases the interaction between the NTO and the soil matrix (Mark et al., 2016). This interaction can also lead to changes in the NTO molecule, increasing degradation due to polar interactions. This study did not analyse for degradation products, although, because of the high organic content in Soil A it was expected to not find NTO in its original form, because of the increased likelihood of NTO degradation products. Conversely a high concentration of NTO was recovered in the leachate from Soil B suggesting that when soil is more prone to degradation, it is less likely for the explosive to accumulate in the soil system. This suggests that there is a higher likelihood of the chemical contaminating groundwater systems. RDX, was recovered in both soils, with highest retention in Soil A due to the affinity between the molecule and the organic content, which was higher compared to Soil B (Lent, 2019). RDX is known to be the most stable compound in the IHE (Lent, 2019; Temple et al., 2019), and therefore as expected was generally recovered at high concentration in both soils in the medium and high contaminated samples. Moreover, compared to NTO, RDX was highly resistant to microbial degradation (Lent, 2019), confirmed by the low degradation rate reported for Soil B, which had the highest microbial content. Therefore, the IHE are less likely to transport to sub-soil and groundwater systems in Soil A, although there is an increased likelihood of degradation products and subsequently an increased contact with the soil matrix. It may be expected that in soils with increased contact time with IHE, there would be an increased likelihood of negative impacts, however in this work the opposite was observed with Soil B being more effected by the IHE.

Table 5
Example of chemical properties scored for the calculation of the EQI. A value of "0" was selected when missing data were present.

Soil A – medium contamination chemical properties		Value obtained from experimental analyses					Scored values					Reference/Technique used
		Day 0	Day 1	Day 7	Day 14	Day 36	Day 0	Day 1	Day 7	Day 14	Day 36	
pH		7.6	7.6	7.4	7.5	7.5	1	1	1	1	1	Amacher et al. (2007)
EC		2.1	3.01	1.6	1.4	0	3	3	3	3	0	Amacher et al. (2007)
CEC		0	0	0	0	0	0	0	0	0	0	Missing analyses
K+	Nutrients	90	105	120	119	112	0	1	1	1	1	Amacher et al. (2007)
N		4	2	1	1	1	4	2	1	1	1	"More is better"
P		1	0	2	0	1	1	0	2	0	1	"More is better"
SOC		0	0	0	0	0	0	0	0	0	0	Missing analyses

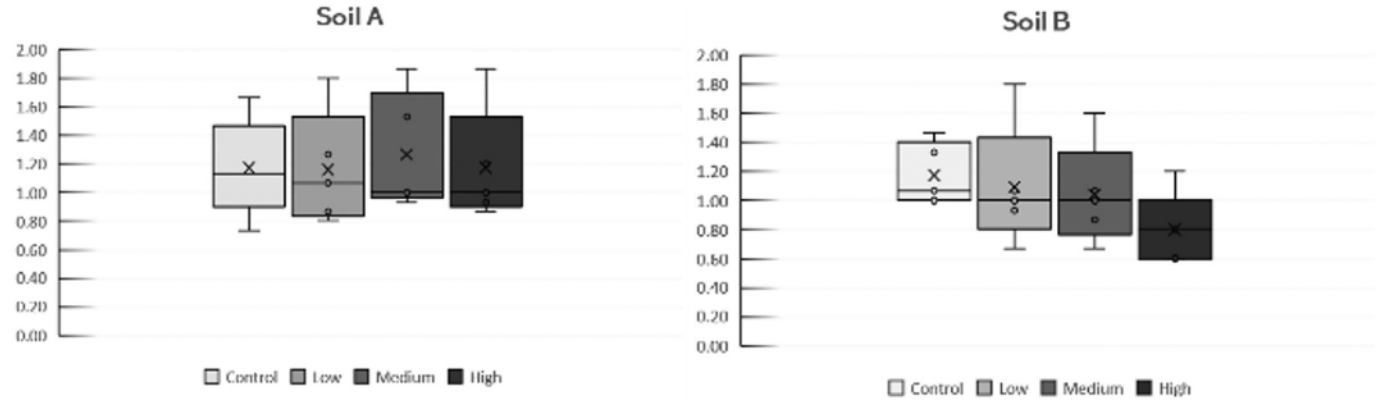


Fig. 4. Environmental Quality Index (EQI) calculated across all the samples for the control, low, medium, and high concentration.

Similar physical changes were observed in the soil matrix for both the control samples and the contaminated samples. These changes cannot to be attributed to the presence of IHE, so must be attributed to the method in which the mesocosm were set up and the weather conditions.

There greater variation in the chemical properties in Soil B compared to Soil A. In general, the EC was most significantly influenced in both soils, which was expected as increase in EC has been previously used as an indicator of pollution (Edwin-Wosu and Nkang, 2019; Gevao et al., 2000). Contaminants can increase the concentration of ions in the soil, which increases the soil's ability to conduct electricity, therefore it can be a cause for concern as certain contaminants can be harmful to plants and animals. The controls had a 60 % and 40 % lower EC for Soil A and B respectively compared to the contaminated samples of the same soil. This change is likely due to the increase in nutrient content (Othaman et al., 2020) provided by the nitrogen rich IHE. As expected, the difference was more significant (60 %) in Soil A due to the greater retention of IHE constituents. In Soil A, this was confirmed by the increase in nitrogen content compared to the control. pH slightly decreased overtime in all samples, including the controls, therefore those changes were not attributed to the presence of IHE, although, as highlighted above, pH can alter the retention of IHE in soil and therefore is a key parameter to observe.

Biological changes were evaluated by microbial abundance which was, at the beginning of the experiment, 85 % higher in Soil B compared to Soil A. Although, the microbial abundance was mostly subject to the weather conditions with an increase in microbial abundance during the first 7 days when the highest level of rainfall was recorded. Further studies are needed to investigate the relationship between potential degradation product formation and the CFU in the soil. For the same reason, a decrease in CFU was recorded all samples at day 36 due to the unexpected 20 day dry spell prior to completion of the experiments. It is worth noticing that at day 36 the microbial abundance was highest in the high concentration sample of Soil B, in contrast to Soil A where the highest microbial abundance was found in the control. More studies are needed to analyse if this difference is due to the high explosive content. Due to the low IHE concentration in soil, the biological parameters were not affected by the IHE components at the concentrations investigated, which was expected due to the concentrations being were lower compared to the latest ecotoxicological data (Dodard et al., 2013; Monteil-Rivera et al., 2021).

The EQI value was calculated based on the scoring of different soil parameters. Although, because of the challenges faced when explosive contaminated samples further studies are needed to create a more comprehensive visualization of the environmental challenges. Therefore, based on the current analyses a preliminary EQI was calculated and values determined for both soils. For Soil A, the EQI value suggests that at the studied concentrations the IHE are not negatively impacting the soil health. However, this cannot be said for Soil B where a significant decrease in EQI has was recorded between the low and high concentration samples. A reasonable number of live rounds fired annually is up to 10,000 (Galante et al., 2017), however this work has shown that with contamination levels from as few as 100 detonations soil quality can deteriorate. For large live-fire ranges 100 detonations can occur within two weeks, which would correlate to a major decrease in soil quality over 1 year for Soil B. This highlights the importance of continuing research into the threat that IHE pose to some soils, such as Soil B, a naturally degraded soil likely to be found on training ranges, as the investigated contamination levels are all below reported LD_{50} values suggesting that soil health begins to deteriorate long before accepted toxic concentrations are reached (Dodard et al., 2013; Monteil-Rivera et al., 2021).

5. Conclusions

In this work, the impact of the IHE mixture has been assessed on two different soils, a pristine sandy loam and a degraded soil collected from a military training range. It has been found that IHE behaviour in the environment is dependent on the soil matrix, such as whether the soil is initially degraded or non-degraded. The pristine soil (Soil A) was found to have more chemical interactions with the IHE constituents, which appeared to reduce the consequences on the soil matrix. The degraded soil (Soil B) was more sensitive to the presence of IHE, with significant changes to nutrients and EC observed, suggesting that potentially IHE has a great impact on degraded soil. Therefore future research is needed to investigate the cumulative impact of IHE on degraded soils as it is more likely to be affected by the explosive components. Although, it is worth highlighting that soil composition and external weather conditions play a key role on the transport and fate of explosive residues. Therefore, further studies are needed to quantify the consequences after an increased number of detonations (>10,000), as 100 detonations, the equivalent in this study, is not representative of actual training activities. Currently, SQIs are not comparable between different sites, this is because the SQI has a different value depending on the chosen indicators and the different areas. The EQI, developed here is based on the same concept but enables comparison and therefore should start generating comparable data between sites enabling a standardise soil quality scale. It is concluded that compared to traditional explosives IHE compositions are unlikely to have the same major impact on the environment at the studied concentrations for pristine soil, although further studies are needed to determine at what point the IHE concentration can cause a major stress on the environment and potentially have an impact on the biota and people's health especially on degraded soils.

CRediT authorship contribution statement

Federica Persico: conceptualization, methodology, writing - original draft, investigation, visualization. Frederic Coulon: conceptualization, methodology, writing - review & editing, visualization, supervision. Melissa Ladyman: conceptualization, methodology, writing - review & editing. Carmen Fernández López: experimental analyses, writing - review

& editing, investigation. Tracey Temple: conceptualization, methodology, writing - review & editing, visualization, supervision.

Data availability

Research data can be found in the Cranfield University data repository (CORD). Data supporting this study are openly available from CORD at https://doi.org/10.17862/cranfield.rd.21501063.v1.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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Evaluating the effect of insensitive high explosive residues on soil using an environmental quality index (EQI) approach

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