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Review

Nature-based approaches to reducing the environmental risk of organic contaminants resulting from military activities

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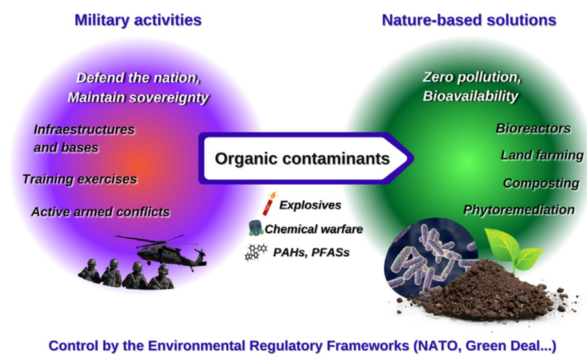
HIGHLIGHTS

- Organic contaminants of concern are emitted by military activities.
- Nature-based remediation technologies are considered eco-friendly and low-cost.
- Chemical warfare agents and military chemical compounds must be researched further.
- Incorporation of bioavailability-based assessments reduce risks from pollution.

GRAPHICAL ABSTRACT



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ABSTRACT

As is the case with many other industrial activities, the organic contaminants at military-impacted sites may pose significant hazards to the environment and human health. Given the expected increase in defense investments globally, there is a need to make society aware of the risks of emissions of organic contaminants generated by military activities and to advance risk minimization approaches. The most recent advances in environmental analytical chemistry, persistence, bioavailability and risk assessment of organic contaminants indicate that efficient risk reductions through biological means are possible. This review debates the organic contaminants of interest associated with military activities, the methodology used to extract and analyze these contaminants, and the nature-based remediation technologies available to recover these sites. In addition, we revise the military environmental regulatory frameworks designed to sustain such actions. Military activities that potentially release organic contaminants on land could be classified as infrastructure and base operations, training exercises and armed conflicts; additionally, chemicals may include potentially toxic compounds, energetic compounds, chemical warfare agents and military chemical compounds. Fuel components, PFASs, TNT, RDX and dyphenylcyanoarsine are examples of organic contaminants of environmental concern. Particularly in the case of potentially toxic and energetic compounds, bioremediation and phytoremediation are considered eco-friendly and low-cost technologies that can be used to remediate these contaminated sites. In addition, this article identifies implementing the bioavailability of organic contaminants as a justifiable approach to facilitate the application of these nature-based approaches and to reduce remediation costs. More realistic risk assessment in combination with new and economically feasible remediation methods that reduce risk by reducing bioavailability (instead of lowering the total contaminant concentration) will serve as an incentive for the military and regulators to accept nature-based approaches.

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Contents

1. Introduction

The power of states is traditionally analyzed in military, economic and geopolitical terms. The role of the environment and natural resources has defined the future of the power of nations and has slowly become a preponderant factor affecting national and international politics. Given that land power will likely be the main element of national power, it can be used to create strategic military effects (Johnsen, 2019). Chemical pollution is a threat to humanity, especially in relation to male fertility, cognitive health and food safety (Naidu et al., 2021). The protection and sustainability of the environment, specifically land conservation, is a critical concern in regard to defense and security. The political power and social importance of military activities affect decisions and their consequences in every area of society, from education to infrastructure. Considering the central role of military organizations, it is clear that no analysis of social, economic or political trends is complete without taking such organizations into account (Montgomery, 2020). Under the pressures caused by the expected increase in military activities at a global scale, sustainable solutions for military chemical pollution have become an urgent need.

In this review, we focus on minimizing the environmental risk caused on land by organic contaminants from military activities for two reasons: 1) to account for the most recent developments in environmental analytical chemistry, bioavailability and risk assessment (RA) of organic contaminants, which differ from those of more established, traditional approaches for inorganic (e.g., heavy metals) and radioactive pollution and 2) to account for the specificities of remediation methods for soils, sediments and adjacent waters contaminated by organic contaminants to allow for the development of nature-based approaches based on their biological removal, in line with recent interest in the application of bioeconomic concepts in sustainable land remediation (Francocci et al., 2020). We hope that readers find the approaches described in this review useful from the most recent findings connecting organic contaminants, biological treatments and military activities, which may differ from other reviews published over the last five years on other aspects of military pollution such as the bioremediation (Chatterjee et al., 2017) and phytoremediation (Via, 2020) of explosives, the remediation of inorganic and organic contaminants (Fayiga, 2019), and soil contamination (Broomandi et al., 2020). We also examine public information that has not been published in peer-reviewed scientific journals but has been included in different reports, mainly by military institutions. Finally, we approach this content from our own research, teaching experience and access to relevant documentation in the field from the Spanish Research Council (CSIC) and University Centre of Defense (CUD) over the last five years.

2. Environmental regulatory frameworks for military activities

The development of environmental mechanisms and policies as guidelines for military forces is internationally led by the North Atlantic Treaty Organization (NATO). NATO emphasizes the responsibility of citizens to participate in sustainable improvement, and the defense sector is not an exception (Goodman and Kertysova, 2022). Likewise, this organization recognizes that military activities must comply with environmental policies, except in extreme circumstances in which the sovereignty of its nation members is at risk. NATO and its member countries, such as the UK, the USA, Denmark, Greece, Holland, Canada, and the Czech Republic, as well as non-NATO countries, such as Australia and Sweden, have specific environmental sections within their armed forces that positively anticipate the results of environmental management in their operations (Oglanis and Loizidou, 2017). The USA Department of Defense was the first to form, as early as 1970, an organization to carry out such supervision in the form of an environmental management system (EMS). Later, other countries began to create military EMSs (Ferro, 2012). For example, the Spanish Ministry of Defense aims to reduce the degradation and contamination of the soil of military lands. The policy followed by the department for the decontamination and remediation of soils can be summarized by the establishment of the most appropriate prevention and management measures to reduce the potential risks of soil contamination. Likewise, a plan for the prevention and recovery of contaminated soils of military installations has been published (Ministerio de Defensa, 2021).

In Europe, military activities must also adhere to the European Green Deal (European Commission, 2021), Europe's new growth strategy. Three specific actions have been arranged by the European Commission that will serve as a great source for the new agreement. Europe will become the first climate-neutral continent in the world by 2050. The benefits involve zero pollution, reasonable and secure energy, clever transport, and high-quality food. The Green Deal describes actions associated with a toxic-free environment including contamination prevention and procedures to clean and remedy contamination, the restoration of biodiversity, the usual roles of groundwater and surface water, and the generation of a sustainable chemical policy. In addition, the European Defense Agency (EDA) (European Defence Agency, 2020) supports its 26 Member States in developing their defense resources through European collaboration. Working as an enabler and implementer for Ministries of Defense that agree to participate in collaborations and projects, the Agency has become the 'axis' for European defense cooperation with knowledge and networks permitting it to involve the whole spectrum. Furthermore, the EU's "Natura 2000" natural habitats network incorporated a considerable number of abandoned military sites: examples are found in Belgium (70 %), the Netherlands (50 %), and Denmark (45 %) (Tobias et al., 2018).

As a consequence of climate change, the scarcity of natural resources and the increasing global population, new questions about environmental quality are predicted at the world level. The human force over the environment certainly dominates the development of international trade, the economic growth of countries, and population change, e.g., an increased demand for housing and migration. One of a series of policy shifts prompted by current world political instability (including for example, the recent invasion of Ukraine by Russia) is that many countries would sharply increase their spending on defense to >2 % of their economic output. This new evidence is a current critical issue of environmental concern. Hence, there is an urgent need to make the scientific community and society, and in particular the military society, aware of the risks posed by the emission of organic contaminants as a result of military activities and to advance risk minimization approaches. Therefore, new strategies to prevent negative effects on the environment and human health will have to be presented by international policies such as those related to NATO, European Green Deal, and the EDA.

3. Military activities causing chemical contamination

Military activities have an environmental impact on terrestrial ecosystems via physical or chemical intrusions. Considering organic contaminants, these effects can be categorized into three groups of activities: (i) the establishment of infrastructures and military bases, which include the construction areas of service buildings and the permanent facilities necessary for support, deployment and operation; (ii) regular procedures of military training exercises that involve the control of organizations and selected military actions to carry out their exercises in peacetime in specific places; and (iii) active armed conflicts that include a combination of active military activities that may include air strikes, strategies of naval vessels or land forces, and the use of chemical weapons. We will focus primarily on land pollution, covering maritime or air military activities when they result in the release of organic chemicals into soils, sediments and adjacent waters.

The military infrastructures include the construction areas of service buildings, and a base operation is a facility that is directly owned and operated by or for the military or one of its branches and in which supply facilities are organized. During operation, large amounts of harmful waste are generated, such as corrosives, solvents, paints, fuels and oils. Some pesticides and biocides are also used by specialized units of the armed forces to kill organisms that cause disease and endanger public health and control pests that destroy homes and structures vital to public safety. Another use of pesticides by militaries involves the use of military materials, such as camouflage netting and blast wall geotextile material, to reduce the incidence of mosquito bites in several environments. Even the aerial application of herbicides, such as Agent Orange, was widespread during the Vietnam War (Ginevan et al., 2009). All these actions could contribute to pollution by organic contaminants (Britch et al., 2020; Aldridge et al., 2020). Another environmental impact could be due to the construction of ammunition sites where the production and processing of explosives was carried out. In these places, explosive production mainly played a significant role during World War II, when for example, 2,4,6-trinitrotoluene (2,4,6-TNT) was manufactured (Eisentraeger et al., 2007). At the end of the war, unfired weapons stayed in several ammunition dump sites. Other similar constructions of ammunition sites after the Cold War, World War I and World War II have already been identified in Europe (Gorecki et al., 2017).

Training exercises involve the frequent use of live fire training ranges, indicating reliable site-specific contamination and degradation (Goldsmith, 2010). Training exercises involve the use of energetic materials in military ranges. Energetic materials include explosives, pyrotechnics and propellants. The use of these materials is of course magnified during armed conflicts. Military pyrotechnics are used for the illumination, signaling, and simulation of battle noises and effects and include items such as flares and signals. The quick and effective firing of pyrotechnic countermeasure decoy flares is vitally important to the safety of expensive military platforms such as aircraft (Woodley et al., 2017). Another example of military training activity is target shooting. In the USA alone, there are approximately 3000 military shooting ranges (Wan et al., 2013). Such targets were historically composed of clay or limestone and a hydrocarbon-based binder (Rodriguez-Seijo et al., 2020; Reigosa-Alonso et al., 2021), which is an important source of polycyclic aromatic hydrocarbons (PAHs). At the ranges, the fragmentation of targets generated significant soil pollution via PAHs. Other organic contaminants, such as the explosive hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), are intensively utilized in military training ranges (Lorenz et al., 2013) As a result, groundwaters and soils in the USA have been contaminated (Kalderis et al., 2011; Cary et al., 2021). *Per*- and polyfluoroalkyl substances (PFASs) are other organic contaminants that are present in historical fire-training exercises where military firefighters repeatedly apply aqueous film-forming foams to extinguish hydrocarbon-fuel fires (Backe et al., 2013; Hu et al., 2016; Kostarelos et al., 2021, Naidu et al., 2020). The US Department of Defense has identified 687 military installations with suspected pollution by these organic contaminants (United States Government Accountability Office, 2021).

War zones are usually extremely dangerous for scientists to visit and collect data. Experimental methodology is generally not viable due to controversies that happen without the knowledge of scientists. Armed conflicts usually happen by chance, remain confidential, or occur in zones that are not easy for scientists to enter (e.g., through drone or aerial attacks). During war activities, any pre-war and post-war efforts to conserve animal populations and ecological integrity are often ignored. When armed conflicts occur between several nations and on large spatial scales, there is a lack of international capacity to monitor the dangers caused to ecosystems. However, in recent years, with the progress of novel and advanced methodologies to document and monitor environmental pollution and damage, studies about the ecological effects of armed conflicts have been more easily conducted (Casana and Laugier, 2017; Zwijnenburg et al., 2020). Modern weapons settled quickly after the development of gunpowder, and when industrial development was enhanced in the 19th century, the opportunities for armed forces increased. For instance, munitions and wrecks introduced PAHs into surrounding benthic sediments due to the HMS Royal Oak shipwreck in World War II (Thomas et al., 2021). The massive use of Agent Orange in the Vietnam War (Dang et al., 2017) generated environmental pollution. Even in deserts, wars produced seasonal variation in sand and dust storms (Broomandi et al., 2020) and the environmental threat faced by oil spill-contaminated sediments during the 1991 Gulf War (Alshemmari, 2021). In the recent conflict between Russia and Ukraine,new threats to the environment emerged when Russia launched a full-scale invasion of Ukraine on 24 February 2022. Ukraine is a country with several tunnels and shafts that have been inundated. These mines, which have been confirmed to be radioactive, affect a city's water supply due to the spread of chemical contaminants. Scientists have informed that the hazards to the cities could be “more deep and dangerous than Chernobyl”. Ukraine is now experiencing an environmental crisis as well, involving not only mines but also contaminated releases from industrial services and organic pollution produced by munitions and shelling (Russia-Ukraine War, 2022).

4. Organic contaminants of interest

The organic contaminants introduced into soil by military activities are usually grouped as potentially toxic compounds (PTCs), energetic compounds (ECs), chemical warfare agents (CWAs) and military chemical compounds (MCCs). Their concentrations in soil in military areas may be unacceptably high and, along with their high toxicity and persistence, this may give rise to environmental risks (Broomandi et al., 2020; Funkhouser and Glueck, 2015). Specific examples of contaminants associated with military activities and the analytical methodology for each group of contaminants are shown in Table 1. Contamination by PTCs is principally caused by military infrastructures and base operations and by chemical storage areas (fuel, oils, lubricants, paints, solvent and corrosives) to be used, for example, in the maintenance of military vehicles, producing waste in large quantities. This poses an environmental risk that should be considered if we also take into account the environmental persistence of these compounds. The analytical methods used to study PTCs are commonly known; for example, the nonhalogenated solvent and diesel range organics present in water samples can be analyzed by a gas chromatography/flame ionization detector (GC/FID, EPA method 8015) (United States Environmental Protection Agency (USEPA), 2003), and semi-volatile organic compounds present in air, water, soil, sediment and waste can be analyzed using a gas chromatography/mass spectrometry detector (GC/MS, EPA method 8270) (United States Environmental

Table 1
Organic contaminants of interest, military activities associated and methods of analysis.

Group	Examples		Military activities	Method of analysis
Potencially toxic compounds	Hazardous waste	Petroleum, oils and lubricants, paints, solvents, corrosives	Storage areas or accidental discharges in bases (Broomandi et al., 2020; Funkhouser and Glueck, 2015)	Gas chromatography/flame ionization detector. EPA method 8015 (United States Environmental Protection Agency (USEPA), 2003) or gas chromatography/mass spectrometry detector. EPA method 8270 (United States Environmental Protection Agency (USEPA), 2018; Barshick et al., 1996; Law et al., 2018)
	Pesticides	Spatial repellent in wall geotextile or camouflage netting	Pest control at military bases and security of military staff (Britch et al., 2020; Aldridge et al., 2020)	GC/flame ionization detection (Regulation (EU) No 528/2012 of the European Parliament and of the Council of 22 May, 2012) and high sensitivity proton-transfer-reaction mass spectrometer (HS-PTR-MS) (Vesin et al., 2013)
	Per- and polyfluoroalkyl substances (PFASs)	PFHxS, PFOS, PFOA, PFNA, PFHpA and PFBS	Fire training (Backe et al., 2013; Hu et al., 2016; Kostarelos et al., 2021)	Liquid–liquid extraction and analysis by HPLC–MS/MS or GC–MS with ionic detection (Backe et al., 2013; Kostarelos et al., 2021; John et al., 2022)
Energetic compounds	Explosives	Nitroaromatics: TNT	Was used during World War I and currently is a common military explosive used in training exercises (Pichtel, 2012)	Solid-phase extraction (for aqueous samples) ultrasonic extraction (for solid samples) or modified method EPA8330B (Temple et al., 2019). The analysis used a gas chromatograph with an electron capture detector (EPA method 8095) (United States Environmental Protection Agency (USEPA), 2002)
		Nitramines: RDX and HMX	RDX is a common military explosive. HMX is used exclusively in military applications, including as a detonator in nuclear weapons, in the form of agglutinated powder explosive, and as a rocket propellant (Pichtel, 2012)	
	Propellants	Nitroglycerin Nitroguanidine Nitrocellulose Dinitrotoluenes (DNT)	Are used as components of gun and artillery and to produce dynamite used in live-fire military training ranges (Pichtel, 2012)	
Chemical warfare agents	Nerve agents	Tabun (GA) Sarin (GB) Soman (GD) Ciclohexilsarin (GF) O-ethyl-d-2(diisoproylamino) ethyl-methylphosphonothiolate (VX)	Can be dispersed from missiles, rockets, bombs, projectiles and ammunition (Chauhan et al., 2008; Lastumaki et al., 2020)	Extraction methods include: liquid–liquid extraction, solid-phase extraction, liquid-phase microextraction, solid-phase microextraction and gas phase methods. Analysis methods include GC/MS and LC/MS (Pardasani et al., 2011; Singh et al., 2015; Singh et al., 2016; Smith et al., 2004)
	Blister agent (vesicants)	Sulfur Mustard (SM) Nitro mustard (NM) Lewisite (L)	Produced during World Wars I and II. Although they do not cause death, they can incapacitate the enemy (Chauhan et al., 2008; Lillie et al., 2017)	Colorimetric, ionic mobility spectrometry, flame photometric and photoionization (Fatah et al., 2005)
Military chemical compounds	Tear-producing agents	2- Chloroacetophenone (CN) O-chlorobenzylidene Malononitrile (CS) Dibenz-(b,f)-1,4-oxazepine (CR)	Are used as riot control agents, in training, in the control of civil disturbances and in counterguerilla operations (Chauhan et al., 2008; Lillie et al., 2017)	For CN, CS and CR, gas chromatography with mass spectrometry (Ferslew et al., 1986)
	Vomiting agents	Diphenylchloroarsine (DA) Dyphenylcyanoarsine (DC) Adamsite (DM)		For DA and DC, gas chromatography with electron capture detector (Haas and Krippendorf, 1997) HPLC is used for adamsite (Haas et al., 1998)

Protection Agency (USEPA), 2018; Barshick et al., 1996; Law et al., 2018). The extraction method depends on the type of analyte and on matrix types (EPA method 3500).

Pesticides are often applied as a spatial repellent in different types of military materials, such as wall geotextile or camouflage netting, to protect soldiers from insect bites during field operations (Aldridge et al., 2020). One compound of this PTC group is transfluthrin, an organohalogen compound, whose effectiveness has been recently investigated (Britch et al., 2020). This semivolatile organic compound, with a relatively low vapour pressure level, is mostly in the gaseous phase and in very low proportions in the particulate phase of air. The measurement of this compound in the gas phase can be done with high sensitivity proton-transfer-reaction mass spectrometry (Vesin et al., 2013). However, the most common analytical method for pesticides are GC/ECD for soil samples and GC/MS for water/air samples (Regulation (EU) No 528/2012 of the European Parliament and of the Council of 22 May, 2012). A relevant group of PTCs found in military fire training areas are PFASs. They can have long chains, including perfluorohexanesulfonic acid (PFHxS), perfluorooctanesulfonic acid (PFOS), PFOA and perfluorononanoic acid (PFNA), or short chains, including perfluor-obutanesulfonic acid (PFBS) and perfluoroheptanoic acid (PFHpA) (Hu et al., 2016). They constitute a group of synthetic chemicals that are chemically stable and persistent in the environment, bioaccumulate and are toxic at low concentrations, being recently considered of environmental concern. Their relatively high water solubility and low octanol/water partitioning coefficient (log K_{ow}) cause them to be easily transferred into groundwater, reaching the subsoil and being able to be taken up by plant roots (John et al., 2022). Analytical methods of PFASs detection in different environmental compartments involve the use of a variety of sensors with dissimilar selectivity, sensitivity, and configurations, including those based on nanotechnology, electrochemistry and fluorescence, as well as the determination of total fluorine content through HPLC and GC (John et al., 2022). Besides, the standard analytical method already used to analyze PFASs at military-affected sites includes liquid–liquid extraction and HPLC–MS/MS or GC–MS with ionic detection analysis (EPA method 8327) (Backe et al., 2013; Kostarelos et al., 2021).

Military activities such as manufacturing operations, training exercises, demolition and disposal and the active use of weapons are sources of common soil contamination by ECs, what includes explosives and propellants. There are several types of military explosives, but when considering only those of organic contaminants, we can classify them as nitroaromatics (such as TNT) and nitramines (such as RDX and HMX), which are the secondary explosives (i.e., they are detonated by primary explosives) most used in military activities (Pichtel, 2012). 2,4, 6-TNT can be mixed with other compounds to produce different explosives. DNT (2,4-dinitrotoluene), which may appear as an impurity during the manufacturing of TNT, is also considered a priority contaminant by the US EPA, has a low aqueous solubility and is detected in the soil of live-fire military training ranges. The royal demolition explosive (RDX, hexahydro-1,3,5-trinitro-1,3,5-triazine) is the basis for some other common military explosives and is used as the main compound of many of the polymer bonded explosives used in nuclear weapons. Finally, the high melting explosive (HMX, octahydro-1,3,5-tetranitro-1,3,5,7-terazocine) can be prepared by nitrolysis of RDX (is a by-product of this compound) and can be mixed with TNT. All these ECs are considered recalcitrant and cause environmental risks and are the subject of intensive research on soil remediation. The other group of ECs is composed of propellants, which are chemicals used in the production of energy or pressurized gas that is subsequently used to create fluid motion or to generate projectile propulsion (United States Environmental Protection Agency (USEPA), 2002). They are formed by one or more explosives mixed with different additives, where the main component is nitrocellulose. Other solid propellants used for gun and artillery are nitroglycerin, nitroguanidine and dinitrotoluenes. In contrast to TNT, RDX and HMX, nitroglycerin is rarely found in soils and studies on this compound in soils are scarce. All the ECs mentioned above do not sorb strongly to soil nor volatilize, which leads to mobilization in the biosphere, thus causing environmental concern (Pichtel, 2012; Pennington and Brannon, 2002; Juhasz and Naidu, 2007; Clausen et al., 2011). The analytical methods for the majority of ECs are standardized EPA methods (Table 1). Nitroaromatic and nitramine compounds can be analyzed in water, soil and sediments with solid-phase extraction techniques (for aqueous samples) or ultrasonic extraction techniques (for solid samples) using acetonitrile as the extraction solvent and CG/ECD (EPA Method 8095). This method can also be used to analyze propellants (United States Environmental Protection Agency (USEPA), 2002). For example, to extract and analyze RDX from soil samples, an adapted EPA method (8330B) was proposed in a very recent study where the effect of the type of soil in this extraction was also studied (Temple et al., 2019).

The last two groups of organic chemicals, CWAs and MCCs, are usually associated with active armed conflicts. The difference between these groups is that CWAs are very toxic compounds used to kill, seriously injure or incapacitate people, while MCCs are less toxic and commonly used as riot control agents and for training. The main CWAs are nerve agents (transmit many nerve impulses in different parts of the body) and blister agents (cause general destruction of tissues forming blisters in the skin). Within the group of nerve agents, there are two subgroups: the G-agents (organophosphate ester derivatives of phosphoric) and the V-agents (whose chemical composition is the same as the G-agents but also contain sulfur). The difference between these two subgroups is the volatility, which is relevant for the toxicity of these compounds. The V-agents have a low volatility, they spread more slowly and thus are more persistent in the environment than the G-agent (Chauhan et al., 2008). The hydrolysis process is often considered to be the main pathway involved in the environmental fate of CWAs. These chemicals are usually not very persistent in the environment but intermediate hydrolysis products can be more persistent and more toxic (Bartelt-Hunt et al., 2006; Kingery and Allen, 1995; Wagner and MacIver, 1998; Munro et al., 1999; Small, 1984).

The main individual CWAs used in military activities are listed in Table 1. The persistence of CWAs depends on several factors: the form in which they are dispersed (aerosol or liquid), their volatility and the meteorological conditions (temperature, wind speed and precipitation). Nonpersistent CWAs have high volatility and are rapidly dispersed, and therefore, there is little probability of resulting soil contamination (Fatah et al., 2005). Currently, these compounds are prohibited, but they can still be used by terrorists, and due to their high toxicity, it is very important to be able to detect them in the environment. For this reason, there have been some recent studies about the sample preparation and analysis of these compounds. Regarding the detection of CWAs, the majority of publications are related to nerve agents because they have a highly lethal effect (Kim and Huh, 2014; Pardasani et al., 2011; Singh et al., 2015; Singh et al., 2016; Montauban et al., 2004; Smith et al., 2004). In addition to these traditional methods, recent studies propose the use of gas sensors and different nanomaterials for the detection of nerve agents and their degradation products (Kim and Huh, 2014). When the compounds are present in the gas phase, sampling, detection and analysis can be more complicated and in these cases, methods can be adapted for field analysis with the use of portable GC–MS (Smith et al., 2004). Regarding sample preparation before extraction, preconditioning is very important, such as for nerve agent VX, for which better results are obtained when a sample is pretreated with a buffer than after direct extraction by organic solvent (Montauban et al., 2004). These compounds are usually analyzed through GC/MS, LC/MS or nuclear magnetic resonance (NMR) after extraction using a method depending on the type of environmental matrix (Table 1). In the military field, detecting and identifying CWAs to protect areas and people before they are affected and to defend themselves against attacks is very important, and for this reason, there are many technologies that can carry out air sampling quickly and alert if there is a hazard posed by the contamination of these toxic compounds. These detectors can be controlled in situ or remotely, and their detection systems include colorimetry, ionic mobility spectrometry, flame ionization, flame photometry infrared spectroscopy, electrochemistry, surface acoustic waves, thermal and electrical conductivity, and polymer composite detection material photoionization (Fatah et al., 2005).

MCCs have neutralizing effects, producing irritation mainly in the eyes and respiratory system, as tear-producing agents and vomiting compounds (Table 1). These compounds are mostly not authorized for military use but potentially exist in military actions. With regard to the extraction and analysis of MCCs, in the case of vomiting agents, the diphenylarsenic compound can be extracted from soil samples by extraction with acetone in an ultrasonic bath (Haas and Krippendorf, 1997), and the analysis method involves gas chromatography with an electron capture detector. In addition, derivatization with 1-ethane thiole or 1-propane thiole is necessary before injecting the sample into the chromatograph to achieve a more selective analysis. The adamsite can be extracted in the same way but needs to be analyzed by HPLC using reversed-phase chromatographic columns (Haas et al., 1998). For tear-producing agents, spectral data (ultraviolet, fluorescence, proton nuclear magnetic resonance, and infrared) and gas chromatography with mass spectrometry are used (Ferslew et al., 1986). When MCCs are present in water samples, solid-phase microextraction and a GC-flame ionization detector can be used.

5. Nature-based technologies applicable to the remediation of contaminated military sites

The methods used for the remediation of military sites depend on both the type of pollutant and the military activity carried out. The available techniques for soil remediation can be in-situ or ex-situ, and may involve a variety of biological, physico-chemical and thermal processes. Currently, incineration is the most effective and widely used remediation alternative, but this method is expensive because of the costs involved in the total removal and replacement with soil from another location and energy for incineration. Other remediation options include chemical extraction and termal desorption, also requiring ex-situ treatment and landfill sealing, with subsequent costs and impacts. Advantages and disadvantages of non-biological and biological technologies used in soil remediation are described in detail elsewhere (Pavel and Gavrilescu, 2008). In this review, we emphasize nature-based technologies because they may have lower costs than other treatment techniques, which could facilitate their use to eliminate organic contaminants at military contaminated sites. Also, the soil retains many of its key functions, which may allow for land use after treatment, thus providing further economic and social value (Francocci et al., 2020). However, some limitations should also be pointed out, such as their usually slow performance, the need to condition pre-treatments to facilitate biological activity and the often unpredictable endpoints due to limited bioavailability (Alexander, 1999). Clearly, the applicability of one or another approach to treat a given site needs to be analyzed individually. Nevertheless, successful examples of nature-based technologies for military activity contamination are summarized in Table 2. Many other published examples of the treatment of civilian contamination could be given for each specific case, but this would extend beyond the focus of our analysis. Technologies approaching risk reductions for inorganic contaminants (e.g., metals) eventually co-occurring with organic contaminants are also not considered here, and the interested reader is referred to other works mentioned in the introduction.

To our knowledge, there are no nature-based remediation techniques for all organic contaminants released into soil by military activities, and their applicability depends on the situation involved. For example, PFASs present strong C—F bonds in their structures such that they resist biological degradation technologies (John et al., 2022), but they can be taken up by plants during phytoremediation (Bolan et al., 2021; Gobelius et al., 2017). Additionally, propellants, nerve agents, and blister and tear-producing agents are soil contaminants, for which nature-based remediation methodologies have been rarely investigated, although there are some studies about the environmental fate and biodegradation of some of these organic contaminants, indicating the potential for nature-based approaches. For example, organophosphate-degrading enzymes have been studied extensively due to their ability to degrade nerve agents, such as Tabun (Pereira et al., 2019) and Sarin.(de Castro et al., 2019; Selleck et al., 2017). In accordance with recent scientific evidence, the direct biodegradation of sulfur mustard by soil microorganisms is considered increasingly feasible, although further developments are required to enhance the solubilization of the aged forms of this agent in contaminated soils to facilitate its microbial transformation into innocuous products (Ashmore and Nathanail, 2008). Recent research further suggests that haloalkane dehalogenase DhaA on the surface of *Bacillus subtilis* spores could degrade sulfur mustard (Wang et al., 2019). The possible participation of microorganisms in the liberation of soluble arsenical compounds from organoarsenic agents (e.g., DC, Table 1) has also been studied (Kohler et al., 2001). Lorenz et al., 2013 researched how the root-colonizing bacterium *Pseudomonas fluorescens*, designed to express XplA, is able to degrade RDX in the rhizosphere (Lorenz et al., 2013). With this knowledge, it is not surprising that the application of nature-based technologies on soils is feasible with these chemicals with additional research and development. However, we focus on commercially available technologies, which have already been used and validated, at least at the pilot scale, to treat soils contaminated by military activities, mainly by PTCs and ECs.

5.1. Case studies of bioremediation

Bioremediation relies on the spontaneous degrading activity of microorganisms to clean groundwater and polluted soil (Ortega-Calvo et al., 2013; Ortega-Calvo et al., 2020). Active microorganisms may already be present at a site and be stimulated by the addition of appropriate nutrients and the adjustment of ecological conditions, which is called biostimulation. In some other situations, the addition of specific active microorganisms to the site through bioaugmentation is considered necessary. A variety of soil amendments, such as organic waste materials, may be incorporated to favor microbial activity. Composting can also be used during the bioremediation of sites contaminated by military activities. In a composting system, organic material (manure or vegetable waste) is used to produce aerobic and anaerobic processes that generate heat. The use of this technique is limited by the formation of toxic metabolites, a risk that must be controlled during the process (Kalderis et al., 2011). Further resources on bioremediation can be found in a reference work (Alexander, 1999).

Some examples of bioremediation technologies applied to military sites impacted by PTCs are presented in Table 2. In the former Soviet air bases in Poland, biostimulation methods of polluted soils were performed to eliminate aviation fuel and heavy fractions of diesel oil (Kołwzan et al., 2008). In perhaps one of the largest remedial actions in Central and Eastern Europe, in situ and ex situ biostimulation was employed at the former military airport of Zatec, Czech Republic to treat soil contaminated by hydrocarbons (Raschman and Vanek, 2008). Some other studies showed, for instance, at an Alpine former military site, where biostimulation was compared vs. natural attenuation. The results reveal significantly higher total petroleum hydrocarbon removal rates than contaminant losses attributed to natural attenuation (Siles and Margesin, 2018).

The degradation of ECs in military-contaminated soil by bioremediation processes has also been investigated to determine the appropriate conditions for biodegradation to occur (Jugnia et al., 2017; Jugnia et al., 2018; Anand and Celin, 2017). For instance, Jugnia et al. (2018) demonstrated that increased anaerobic activity was strongly connected to the disappearance of RDX from soils with the application of amendments in a former military demolition range area. Kalderis et al. 2011 found that where different bioremediation technologies for explosives appear, a sulfate-reducing consortium can be used to remove TNT from a soil (Kalderis et al., 2011). In addition, in this publication, aerobic and anaerobic bacterial species that degrade TNT, RDX, HMX, and PETN were shown. In another study, RDX degradation rates were determined after bioaugmentation with *Gordonia* sp. strain KTR9 to assess under biostimulation conditions in an RDX-polluted aquifer in a former military installation (Michalsen et al., 2016).

5.2. Case studies of phytoremediation

Even though there have been no successful full scale applications for phytoremediation methodologies (Via, 2020), the ability of several types of plants to remove organic contaminants at levels comparable to those found in military contaminated sites is well documented, mainly for ECs (Table 2). Plants can accumulate or directly metabolize chemicals, by themselves or in combination with microorganisms in both soil and groundwater

Table 2
Examples of nature-based technologies for military activity contamination.

Nature-based technology	Description	Organic contamination site	Example
Natural attenuation	Spontaneous pollutant removal, continuous monitoring	Hydrocarbon-contaminated soils	Siles and Margesin, 2018
Land farming	In situ periodical fertilization with inorganic nitrogen phosphorous and potassium (NPK)	Hydrocarbon-contaminated and explosive-contaminated soils	Siles and Margesin, 2018; Clark and Boopathy, 2007; Raschman and Vanek, 2008
Composting	Amendment with biodegradable organic materials, fertilization, and pile maintenance under controlled humidity and aeration	Explosive-contaminated soils	Kalderis et al., 2011; Payne et al., 2013
Composting + bioaugmentation	In situ amendment with biodegradable organic materials following bioaugmentation	Explosives-contaminated soils, sediments, and groundwater	Michalsen et al., 2016; Jugnia et al., 2017; Jugnia et al., 2018
Prepared-bed bioreactor	Ex situ treatment with recirculation of irrigated water and nutrients	Hydrocarbon-contaminated soils	Kołwzan et al., 2008
Soil slurry reactor	Mechanical mixing with liquid phase, controlled aeration	Explosives-contaminated soils	Clark and Boopathy, 2007
Phytoremediation	Use of plants to mobilize the contaminant into plant biomass	Explosives and PFAS-contaminated soils and groundwater	Lee et al., 2007; Rylott et al., 2011; Hannink et al., 2001; Hannink et al., 2002
Phytoremediation and bioremediation	Use of plants and soil bacterial diversity	Explosive and vomiting agents-contaminated soils and waters	Cary et al., 2021; Lamichhane et al., 2012; Thijs et al., 2018; Teng et al., 2017

(Hannink et al., 2002). Phytodegradation is a more favorable technique for the elimination of hydrophilic organic compounds than hydrophobic organic chemicals (Fernandez-Lopez et al., 2021). Several plants have been used for the phytoremediation of explosives, such as *Echinochloa crusgalli, Helianthus annuus, Abutilon avicennae* (Lee et al., 2007), *Vetiveria zizanioides* (Das et al., 2010) and *Phragmites australis* for TNT (Kalderis et al., 2011) and *Panicum maximum* for RDX and HMX (Lamichhane et al., 2012; Payne et al., 2013). Transgenic plants can also be used to extract and detoxify TNT. This type of plant expresses nitroreductase and manifests a remarkable increase in the capacity to allow, take up, and detoxify TNT (Hannink et al., 2001). Rylott et al. (2011) developed engineered TNT-resistant *Arabidopsis* plants for RDX biodegradation. Data also suggest that switchgrass (*Panicum virgatumhas*) may be employed to eliminate RDX in live-fire training ranges, munitions dumps and minefields (Cary et al., 2021). The sycamore maple tree (*Acer pseudoplatanus*) is another plant that has been assessed for phytoremediation on military-contaminated sites (Thijs et al., 2018).

A pot experiment was conducted to study the efficiency of *Pteris vittata* to clean up diphenylarsinic acid (DPAA), a hydrolytic or oxidative organic arsenical product of chemical vomiting agents that were largely produced during the World Wars. Soil was collected from a forest in Northeast China, where DPAA is often detected. The results showed an enhanced removal of DPAA and the recovery of soil ecological function using *P. vittata* associated with *Phyllobacterium myrsinacearum*, a plant growth-promoting bacterial isolate (Teng et al., 2017). In a field study on a fire training site at Stockholm Arlanda airport (Sweden), different realistic phytoremediation approaches were tested to assess the PFASs phytoremediation potential of a range of plant species from the local vegetation. The best scenario, resulting in the highest levels of PFASs uptake by plants, involved the use of a shelter wood of mixed silver birch and Norway spruce stands (Bolan et al., 2021; Gobelius et al., 2017).

6. Integrating bioavailability science into the nature-based remediation of military sites

Bioavailability of organic contaminants is important in research on nature-based remediation, but very little discussed in connection with military activities. Bioavailability is a measure of how much a substance is able to gain access to an organism for uptake or adsorption across its cellular membrane. This idea is essential for RA and monitoring remediation (Naidu et al., 2015), as it considers what amount of a pollutant present in a polluted area is accessible for uptake by organisms and that can thus, in theory, cause harm (Hodson et al., 2011). Despite the positive experiences from the implementation of bioavailability in the RA of soils and sediments contaminated by metals, no integrated approach for application exists for organic chemicals (Ortega-Calvo et al., 2015; ISO Technical Committee, 2020). Usually, only total concentrations are considered in toxicity evaluation and RA, which may lead to high remediation costs to remove organic chemicals, resulting in more passive or "dig and dump" activities. More realistic RA in combination with new and economically feasible remediation methods that reduce risk by reducing bioavailability (instead of lowering the total concentration of the pollutant) will be an incentive for the military and regulators to accept nature-based approaches. Moreover, in many situations, the established treatment methods mainly remove the bioavailable pollutant fractions (Ortega-Calvo et al., 2013). Additionally, military-polluted lands may be of non-sensitive use, such as training fields (as opposed to residential or agronomic uses), which would justify less stringent pollutant threshold values (Peijnenburg, 2020). Therefore, bioavailability could be used by the military sector as a tool to create site specific environmental requirements and reduce remediation costs.

In practice, soil organic contaminants can be divided into four compartments well defined for simplicity: non-extractable (or sequestered), slowly desorbing, rapidly desorbing, and dissolved in water (Fig. 1), as defined by Ortega- Calvo et al. (2015) (Ortega-Calvo et al., 2015), and this classification was later adopted by the International Organization for Standardization (ISO) to quantify bioavailability (ISO Technical Committee, 2020). As a first step, a physicochemical approach is generally used to determine bioavailability (Fig. 1), building upon the recent standard ISO 16751 method, which uses desorption extraction to determine the bioavailability of nonpolar organic compounds (with an aqueous solubility <100 mg/L or a (Log) octanol-water partitioning coefficient > 3). To be useful with military chemicals, the chemical window for application of this methodology should be extended to more polar target pollutants (TNT, DNT, etc.), for which the regulatory implementation of modern bioavailability concepts is still nonexistent. The methods can also include in vivo assessments of bioavailability through oral and dermal exposure, which has recently been employed in the RA of a military former Foster Air Force Base skeet shooting range location polluted by PAHs (Forsberg et al., 2021; Meyer, 2022). At this site, lead was identified as another contaminant of potential concern, although the focus was on PAHs to reduce uncertainty in human health RA associated with direct contact with contaminated soil and to develop a site-specific cleanup goal for PAHs at the site. Remediation cost savings as a result of these bioavailability results, implying new (higher) protective concentration levels for benzo

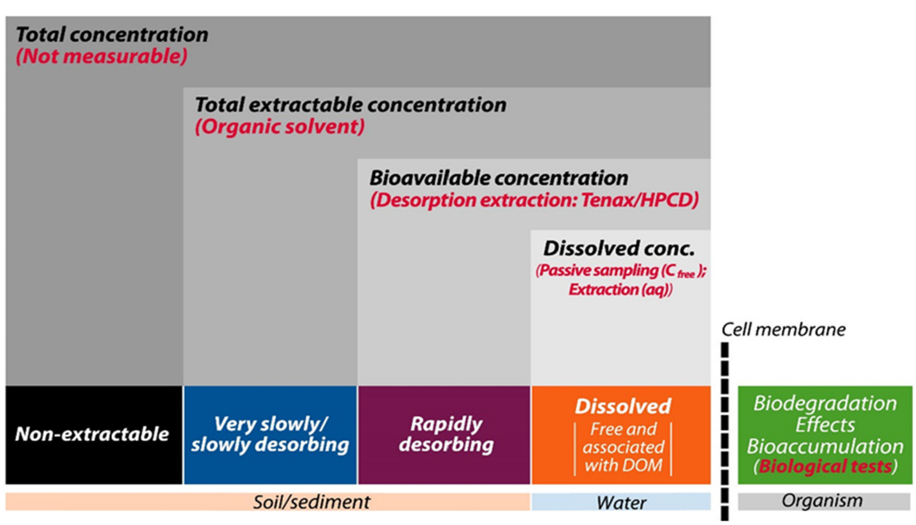

Fig. 1. Scheme of the four compartments of organic contaminants in soils. Methods to determine the concentration of contaminants in each compartment are given in parentheses (Ortega-Calvo et al., 2015).

(*a*)pyrene, may total over 6.5 USA dollars for this site. In spite of considerable interest, the authors indicate in the study that the development of a consensus method for estimating PAH bioavailability lags behind that of inorganic contaminants such as lead and arsenic, for which bioavailability is already included in the RA of other sites in USA (Forsberg et al., 2021; Meyer, 2022).

In addition, in assessing the bioavailability of the target chemicals, their phase exchange during bioremediation itself could be dissected to provide support to evaluate the effectiveness of the treatments. In this way, the assessment could address how nature-based remediation influences the distribution of chemicals into different bioavailability pools, i.e., non-extractable residues (NERs), slowly and rapidly desorbing fractions, and freely dissolved concentrations, with an emphasis on the rapidly desorbing fraction. The recent standardization efforts could be used similarly to previous bioavailability assessments of PAHs in contaminated soils treated by nature-based technologies (Ben Said et al., 2021; Posada-Baquero et al., 2019; Posada-Baquero et al., 2020; Posada Baquero et al., 2021). In a large pilot scale, bioremediation methods were applied to treat high total petroleum hydrocarbon contents. The best results were obtained through combined biostimulation and bioaugmentation (Ben Said et al., 2021). In another study (Posada-Baquero et al., 2019), bioavailable concentration assessment in combination with total extractable concentrations showed advantages in assessing the bioremediation of PAH-contaminated soils, including a military soil with a long history of industrial exploitation from Fidenza (Italy) that was bombarded during the Second World War. The results indicate that using bioaugmentation or stimulation with biosurfactants as the sole bioremediation method could permanently lead to improved performance. Research on the changes in bioavailability was also used as a useful tool during an experiment conducted in a greenhouse researching the effect of a biosurfactant application on slowly desorbing PAHs (Posada-Baquero et al., 2020).

Risks increases due to the co-metabolism of organic contaminants are an important aspect to consider in nature-based remediation, as additional detoxification approaches may be needed due to increased toxicity after partial biological processing (Fernandez-Lopez et al., 2021). For example, co-metabolism governs the microbial degradation of TNT, and the immobilization of TNT metabolites in soil needs to be examined, since they can be released into the environment via, for example, dissolved organic matter, thus creating additional risks (Stenuit and Agathos, 2010). However, recent bioavailability research applying state-of-the-art physicochemical approaches to the ecotoxicity of NERs of TNT in soil indicate that biodegradation leads to effective risk reductions (Harmsen et al., 2019). These authors determine that for TNT, toxicity was eliminated from soil by removing the bioavailable fraction through desorption extraction (Fig. 1), and therefore the toxicity was caused by this fraction and not by NER. This is a great implication for the nature-based remediation of military sites contaminated by explosives.

7. Conclusion

Many investigations have shown the applicability of nature-based remediation technologies to military-contaminated sites, especially with potentially toxic compounds and energetic compounds. Some organic compounds, such as chemical warfare agents (nerve and blister agents) or military chemical compounds (tear-producing and vomiting agents), show potential for use with these technologies but they must be investigated further. To this end, we propose the use of nature-based methodologies to reduce the risks of chemical pollution with the integration of modern, bioavailability-based assessments of process performance and endpoints. These methodologies will ensure that the site-specific target values for risk reduction and the sustainability and cost-effectiveness of the treatment can be achieved. The knowledge examined in this paper constitutes an opportunity to facilitate the inclusion of these sustainable solutions under the pressure caused by the expected global increase in military activities.

CRediT authorship contribution statement

Carmen Fernandez-Lopez: Conceptualization, Methodology, Writing, **Rosa Posada-Baquero:** Conceptualization, Methodology, Writing, and José-Julio **Ortega-Calvo:** Writing- Reviewing and Editing, Funding acquisition.

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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