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Research article

Controlled experiments on dissolution and remediation of 2,4,6-trinitrotoluene in distilled water and seawater

Joseph Sadler [a], Matar Movshovitz [a], Federica Persico [a], Natalie Mai [a], Carmen Fernandez Lopez [b], Nilgun Sen [c], Lisa Humphreys [a], Evie Kadansky [a], Sally Webb [a], Bhumika Sharma [a], Josh Wardrop [a], Frederic Coulon [d], Melissa Ladyman [a], Tracey Temple [a,*]

[a] Cranfield University, Centre for Defence Engineering and Physical Science, Defence Academy of the United Kingdom, Shrivenham, SN6 8LA, UK
[b] Centro Universitario de la Defensa, Universidad Politécnica de Cartagena, C/ Coronel López Peña S/N, Santiago de La Ribera, 30720, Murcia, Spain
[c] Turkish National Police Academy, Institute of Forensic Sciences, Ankara, Turkey
[d] Cranfield University, Faculty of Engineering and Applied Sciences, Cranfield, MK43 0AL, UK

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ABSTRACT

Prior to the ratification of the London Convention on the Prevention of Marine Pollution by Dumping of Wastes and Other Matter in 1972, dumping of military munitions at sea was considered a safe and secure method of disposal. There is increasing desire to remove the now corroding and unstable munitions from this prime ocean real estate to develop offshore wind and solar farms. However, after 50–100 years of exposure to the marine environment, corroded munitions may be leaching toxic explosives to the environment, and remediation methods involving detonation are also likely to leave toxic residue. Therefore, to investigate the potential for explosive leaching at dump sites, the dissolution of the common high explosive 2,4,6-trinitrotoluene (TNT) in seawater was investigated. Then, to determine the potential for remediation, the efficacy of commercial activated carbon and waste derived biochar for adsorption of TNT from seawater was investigated. It was observed that TNT dissolves slower in seawater compared to distilled water, which suggests that explosives in underwater ordnance may remain in bulk mass for longer periods of time than expected and be easier to remove. Furthermore, the small-scale laboratory test demonstrated that both activated carbon and biochar reduced the concentration of TNT in distilled and seawater by up to 90 % after only 2 h. This study provided insight into alternative sustainable remediation options for TNT-contaminated water using commercially available activated carbon and biochar generated from waste products.

1. Introduction

It is estimated that millions of tons of unused explosive ordnance was intentionally 'disposed of' at sea recorded prior to enforcement of the London Convention on the Prevention of Marine Pollution by Dumping of Wastes and Other Matter in 1972 [1,2]. Driven by political, safety, security, and financial considerations [1], a significant volume of unused World War I and II ordnance

* Corresponding author.
E-mail address: t.temple@cranfield.ac.uk (T. Temple).

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stockpiles were disposed of at sea [3]. Add to this, World War shipwrecks that remain stranded laden with explosive ordnance on the ocean floor [4]. Approximately 1.6 Mt of ordnance are believed to be scattered across German coastal waters, and around 200,000 t in Norway coastal waters [5]. Switzerland's freshwater lakes are also affected by dumped ordnance, turning these areas of natural beauty into hazardous spaces [6]. However, the full extent of the problem remains unclear as the location of dumpsites were rarely recorded and limited information regarding the nature (high explosive, chemical, small arms etc) and volume of the ordnance is available. In some locations, ammunition back to the 19th century has been found [7,8].

In the past, leaving dumped munitions undisturbed in inaccessible areas of the seabed was considered the optimal solution [9,10]. However, the race for green energy is redefining what was previously considered inaccessible and unusable seabed to develop offshore wind and solar farms making it economically feasible to destroy or remove dumped explosive ordnance. The most common method for clearing dumped explosives is to detonate them in-situ to minimise the risk of accidental detonation by moving them; however achieving a clean underwater detonation is challenging especially with large stockpiles where there may be sympathetic detonations, or where expected detonations fail due to degradation of the explosive fill. This can result in significant deposition of explosive and degraded explosive residue in the environment [3,11–13].

This is in addition to existing local contamination arising from leaching of the high explosive content from shells compromised by corrosion [5,14–17]. Submerged ordnance is exposed to various water currents, driven by wind forces, tidal forces, internal waves, and thermohaline differences, which can reach velocities up to 3 m/s [18]. These physical conditions significantly alter the rate of corrosion of the shells and subsequently the rate of chemical dissolution and physical erosion of exposed explosive fills. This makes it difficult to predict how significant the impact of explosive contamination will be [18,18,19].

The presence of legacy explosives in conventional munitions found underwater varies, with 2,4,6-trinitrotoluene (TNT) being a prominent concern due to its extensive use and potential for degradation into toxic by-products such as amino-dinitrotoluene (ADNT), diamino-nitrotoluene (DANT), and 1,3,5-trinitrobenzene (TNB) when exposed to light and at depth in sediment [17,20–22]. As early as the 1970's, studies showed the toxic properties of TNT degradation products on algae and fathead minnow [23], with further studies highlighting how TNT can bioaccumulate in several marine organisms e.g. *Salmon Alevins* [24], *Hyalella azteca* [21], and lead to changes in the environment e.g. red water [24,25]. Research has also shown that these dissolved explosives can be up taken by marine organisms, such as blue mussels (*Mytilus Edulis*), algae (*Fucus Vesiculosus)* and fish thereby posing risks to both marine environments and human health through bioaccumulation in the food chain [26–31].

Given the likelihood of residual explosives remaining at sea from disposal operations or abandoned munitions it is essential to understand the potential for impact on the marine environment through leaching of explosives fills. Marine conditions vary considerably, but there have been few studies into how dissolution of TNT differs in saltwater environments. There have been studies to simulate leaching rates of TNT undertaken by Refs. [32,33] to investigate the influence of pH, temperature, and stirring speed showing that dissolution rates of explosives are influenced by variations in temperature and salinity, while pH changes have minimal impact. However, these studies employed water stirring methods that may not accurately reflect natural underwater currents as water is less likely to be in direct contact with the solid surface.

Knowing that there is significant potential for contamination of the marine environment from discarded ordnance, research is focussing on developing environmentally safe and cost-effective techniques to remediate TNT residues from water environments. While there is limited research on remediation of explosives-contaminated seawater [34], there have been investigations into remediation methods for processing wastewater. In industry, remediation by Activated Carbon (ACs) is the preferred approach due to its convenience and cost-effectiveness [35–38]. Rajagopal & Kapoor, (2001) examined AC adsorption capacity for TNT, 2,4-Dinitrotoluene (DNT) and Nitrobenzene (NB) contaminated water using static and dynamic experiments. They showed that ACs adsorption capacity was dependent on the surface area and other factors such as hydraulic loading rates, feed concentrations and bed heights. As most ACs used are derived from bituminous coal, recent investigations have also begun exploring biochar derived from organic carbonised waste materials as a more sustainable alternative [39,40]. For example [40], utilised rice husk biochar to successfully adsorb TNT and 1,3,5-Trinitroperhydro-1,3,5-triazine (RDX) contaminated water, indicating that sustainable, environmentally friendly, operator-safe, and cost-effective remediation methods can be employed. Although, the cost-effectiveness of large-scale implementation can be challenging due to the environmental concerns related with the use of carbon and the high costs of handling waste materials rendering necessary research into more sustainable options [36].

The aim of this study was to provide insight into the environmental impact of UXO in marine environments, focussing on controlled laboratory methods to explore the dissolution behaviour of TNT in distilled and seawater and methods for remediation using ACs and biochar.

2. Materials and methodology

2.1. Chemical and consumables

TNT was acquired by a Defence partner and was used at Cranfield University following in house health and safety procedures that align to UK policy. Acetonitrile (ACN) 99.9 % HPLC grade was purchased from Thermofisher and used for the extraction of the TNT from water and carbon. Seawater was prepared in house using "Sea mix" purchased from Peacock Salts. Norit® 830 was purchased from Cabot Corportion, 2011 and CENTAUR ® 12 × 40 was purchased from Calgon Carbon. The rice husk and wheat straw biochar were purchased from the UK Biochar Research Centre at Edinburgh University.

2.2. Dissolution of TNT in distilled water and saline water

2.2.1. Melt-casting of TNT

For the dissolution experiments, 3 g of TNT was melt-cast at 110 °C into PTFE cylindrical moulds of 5 ± 1 mm diameter. The fragments were cooled for 4 h at room temperature until they reached 70 °C to avoid accidental ignition. When the cylindrical fragments were completely cooled, they were weighed, measured with a calliper and then the surface area was calculated using the following equation (1):

$$Surface\ Area = 2{*}side_{SA} + center_{SA} = 2{*}\frac{\pi d^2}{4} + \frac{M}{\rho}{*}\frac{4}{\pi d^2}{*}\pi d = \frac{\pi d^2}{2} + \frac{4M}{d\rho}\ \left[cm^2\right] \quad (1)$$

where M is the mass of the fragment, d the diameter, ρ the density and SA is the Surface Area (1.6 cm^3/g).

In total, 13 fragments were produced with masses that ranged from 129 to 181.2 mg and surface areas from 0.98 cm^2 to 1.25 cm^2.

2.2.2. Dissolution of TNT in saltwater when exposed to different flow rates

To mimic water currents flowing over the TNT fragments, a Filatronic aquarium pump (200 L/h) was placed at the bottom of a 600 mL glass beaker filled with 500 mL of either distilled water or synthetic seawater. The beaker was entirely covered with aluminium foil to prevent photodegradation of the TNT. The synthetic seawater was made by mixing 41.5 g of 'Seamix' from Peacock salt with a mixture of 66.1 % Sodium Chloride (NaCl), 16.3 % Magnesium Sulphate ($MgSO_4$) 12.7 % Magnesium Chloride ($MgCl_2$), 3.30 % Calcium Chloride ($CaCl_2$), 1.60 % Potassium Chloride (KCl) with 1 L of distilled water for 24 h as suggested by manufacturing guidelines to reproduce representative deep seawater, this is to ensure that the final solution not only matches the salinity but also replicates the ionic composition of seawater. The typical water salinity is 35 %, although the higher percentage used is to account for the other salts present in natural seawater, ensuring an accurate simulation of its chemical balance. The pump was connected to a flowmeter which recorded flow rates over the time of the experiment. The TNT fragments were placed on top of the flowmeter using a Delrin connector (Fig. 1). Different velocities were tested and were grouped in one of four categories: very low (0–1 cm/s), low (10–20 cm/s), medium (30–40 cm/s) and high (50–60 cm/s), with an error of ±1.5 cm/s. The velocity ranges tested were based primarily on equipment availability and the opportunity to trial controlled wave actions and was not intended to fully represent natural systems. All experiments were conducted at water temperature of 23–25 °C, which was monitored using temperature sensors throughout the study.

Two controls for both distilled and seawater were conducted where one fragment was placed in non-flowing water and the second was placed outside the set up to ensure the waterflow was the contributing factor. There were 19 experiments conducted in total at different velocities (Table 1). Because of the challenges in achieving a steady flowrate some velocities were tested more than once. For efficiency, some TNT fragments were reused, however all fragments were air dried to constant weight and reweighed before and after each test.

To analyse dissolution, aliquots of water (1.2 mL) were extracted periodically over 3 days using a glass pipette. The first aliquot was collected at 40 min followed by sample collection every hour up to 3 h, then at approximately 21, 24, and 27 h depending on observed dissolution as per HPLC analysis. Each sample was analysed by HPLC. At the end of each test, TNT fragments were collected and air dried for one week to determine final mass.

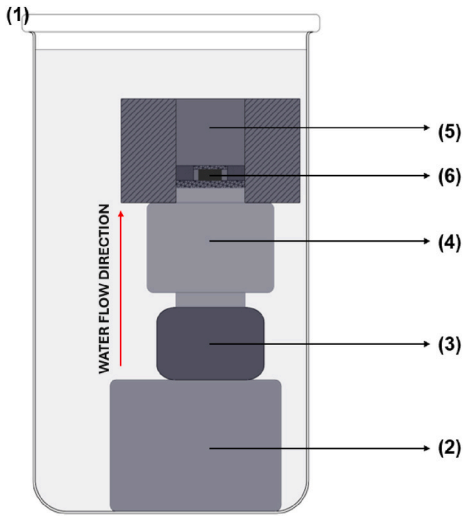

Fig. 1. Experimental set up to examine the dissolution rate of TNT in water showing a 600 mL glass beaker filled (1) with 500 mL distilled water or seawater. Inside the beaker the aquarium pump (2) connected via a Delrin connector (3) to a flowmeter (4) with a PVC holder on top (5). TNT fragment (6) was placed in the PVC holder (5).

Table 1
– Type of experiment, water velocities studied and the mass of all TNT samples for each set-up.

Type of experiment	Velocity (cm/s)	Replicates	Mass of TNT fragments (mg)
Distilled and Seawater			
Control 1	No flow	2	168.8
Control 2	No direct flow	2	181.2
Distilled Water			
Very-low velocity	1	1	157.6
Low velocity	12–16	5	139.2–169
Medium velocity	28–33	3	122.4–158.1
High velocity	51–58	4	117.8–171.4
Seawater			
Very low velocity	0–1	2	160.2–167.9
Low velocity	7–11	2	147–162.6
Medium velocity	28–38	2	129–148.6

2.3. Comparison of commercial carbon and biochar for TNT adsorption

TNT (100 mg) was dissolved in 2 L of distilled and seawater at room temperature using a Heidolph MR3002 stirring plate with a magnetic bar stirrer. During the dissolution process, which took approximately 2 days, both solutions were completely covered with aluminium foil to prevent photodegradation of the explosive material. Various carbons were selected for testing (Table 2). Three commercial ACs including Cabot Norit 8 × 30 mesh (N830), Cabot Norit 12 × 40 mesh (N1240) and Calgon Centaur (Generic activated carbon), were selected following previous research from Fawcett-Hirst et al., 2020. These commercial ACs were compared to rice husk and wheat straw biochar purchased from the UK Biochar Research Centre at Edinburgh University, as there has been evidence that these materials adsorb explosives such as TNT, RDX [41] and Toluene [42]. Moreover, as the surface area has shown to play a major role in the adsorption of materials, the biochar, rice and wheat husk were pulverised using a grinder and compared to their non-pulverised equivalents to determine the differences of adsorption capacity due to the increased surface areas.

To compare the carbon efficiency differences in contaminated seawater and distilled water, 50 mL of the TNT stock solution was added to the ACs and Biochar (5 g). To ensure reliability of results each carbon was prepared in triplicate for a total of 46 samples which included two controls: 1) TNT spiked water in the absence of carbon to control for TNT concentration and 2) carbon in water not exposed to TNT as a negative control. The amber vials were placed on an orbital shaker (Stuart™) and the TNT-contaminated water was collected at 15, 45, 60 and 120 min using a glass Pasteur pipette, filtered using a 0.2 μm nylon filter and stored at 4 °C until analysis. The time lapse was to determine adsorption rates overtime.

To determine the mass of TNT adsorbed to carbon of the TNT the carbon was collected after analysis, weighed, and left to air dry for 7 days in an enclosed space to avoid photodegradation of the TNT component. Samples were extracted with 5 mL pure Acetonitrile (ACN) (99.9 %) per gram of carbon and placed on an orbital shaker at 145 rpm for 18 h. Samples were filtered using a 0.2 μm Nylon filter and stored at 4 °C until HPLC analysis [43].

2.4. Adsorption efficiency in a contained environment

Based on the results from the experiment described in 2.1, two of the most efficient adsorptive materials, NORIT 1240 and ground rice husk, were taken forward for further testing. To avoid the adsorptive material dispersing throughout the water, it was contained in a 100 % BPA-Free plastic pod (29 mm height × 37 mm top diameter × 23.7 mm bottom diameter) topped with a stainless-steel mesh size of 0.14 mm. The pods, in triplicates, were filled with either 5 g of NORIT 1240 or rice husk and placed in 500 mL amber jar vials filled with 150 mL of 50 ppm TNT stock solution in seawater. The amber vials were then placed on an orbital shaker and the TNT-contaminated water was collected at 30, 75 and 135 min then at 24 and 48 h. The final collections were reported to ensure that equilibrium was achieved. Finally, the samples were analysed using HPLC.

Table 2
First selection of Activated Carbons with materials and distinctive characteristics.

Materials	Main composition	Comments
NORIT 830	Bituminous coal	Designed for cleaning contaminated water
NORIT 1240	Bituminous coal	Designed for cleaning potable water
Generic AC	Bituminous coal	Cheaper alternative to NORIT series
Rice Husk	Rice	Cheaper, sustainable alternative. Waste product formed from the hard outer layer of grains of rice which is removed during the milling process
Wheat husk	Wheat	Waste product formed from the hard outer layer of wheat which is removed during the milling of wheat

2.5. Analysis by High Performance Liquid Chromatography

Each sample was analysed using High Performance Liquid Chromatography (HPLC) using a 150 × 4.6 mm Agilent Zorbax Eclipse Plus C18 column in a Waters Alliance 2695 with a Waters 996 photodiode array detector. The mobile phase was 60:40 ACN and deionized water with 0.1 % formic acid at a flow rate of 1.5 mL/min. The column temperature was 30 °C. The injection volume was 10 μL. Peak identification was performed by comparing the retention time and UV profile of the compounds to TNT standards. For the standards TNT (25 mg) in water (0.5 L) stock was diluted to six concentrations between 50 mg/L and 0.5 mg/L. The output signals were monitored at a 245 nm wavelength. The HPLC run time was 4 min and TNT peaks appeared after 2.5 min (LOD: 0.196 mg/L; LOQ: 0.594 mg/L).

2.6. Fitting to adsorption isotherms

To understand the interaction between the TNT and the adsorptive materials different isotherm models were used using the data from the experiments in section 2.3. The Langmuir isotherm model was calculated following equation (2) [44]:

$$\frac{1}{q_e}=\frac{1}{Q_0}+\frac{1}{Q_0 K_L C_e} \quad (2)$$

Where *Ce* is the concentration of contaminant in solution at equilibrium in mg/l; *qe* is the mass of contaminant adsorbed per unit of adsorbent material in mg/g, *Qo* is the maximum capacity of the resulting monolayer in mg/g and *KL* is the resultant Langmuir isotherm constant in l/mg [44].

The Freundlich isotherm was calculated using Equation (3) [37,45,46] (Equation (3)).

$$\log q_e = \log K_F + \frac{1}{n}\log C_e \quad (3)$$

Where K_F $\frac{\frac{1}{L^n} * (mg)^{1-\frac{1}{n}}}{g}$ represents the Freundlich adsorption capacity constant and n is the Freundlich constant representing the adsorption intensity.

And the Temkin isotherm model, which assumes that the heat of adsorption of all molecules in the layer decreases linearly with coverage due to adsorbate-adsorbate interactions, was calculated using Equations (4) and (5) [44].

$$q_e = \mathrm{B}\ln A_r = \mathrm{B}\ln C_e \quad (4)$$

$$B=\frac{RT}{b_r} \quad (5)$$

Where B is derived from the universal gas constant (R) and the temperature in kelvin (T) using their usual notation divided by the Temkin isotherm. The constant b_r represents the binding constant in L/g.

Carbon was also visually analysed using a Hitachi SU5000 Scanning Electron Microscope (SEM) at 10 kV, spot size of 6.4 mm and a magnification of 120 × to determine surface changes (e.g. micro-holes; cracks) to the biochar materials when in contact with seawater and distilled water.

Table 3
Dissolution rate of TNT fragments in seawater and distilled water normalised to 25 °C dissolution value using the equation from Ref. [32] and divided by the surface area and of each fragment, calculated according to equation (1).

Water type	Velocity [cm/s]	Fragment mass before experiment [mg]	Fragment mass after experiment [mg]	Fragment calculated surface area [cm^2]	Dissolution rate * e^2 [mg/min]	Dissolution rate factor *e^2 [mg/cm^2/min]
Distilled water	13	167	117.8	1.23	7.51	6.12
	15	169	110.7	1.24	7.72	6.24
	30	158.1	87.6	1.18	6.86	5.80
	55	146.9	97.8	1.13	12.2	10.83
	56	171.4	118.2	1.25	10.3	8.24
	13	159.4	150.8	1.19	5.31	4.46
	58	117.8	105.6	0.98	7.44	7.58
Seawater	0–1	160.2	158.1	1.19	1.05	0.88
	0–1	167.9	161.7	1.23	3.06	2.48
	7	147	141.1	1.13	2.59	2.30
	11	162.6	156.4	1.21	3.28	2.72
	28	129	123.3	1.04	2.53	2.44
	38	148.6	140.7	1.14	3.24	2.85

3. Results and discussion

3.1. Dissolution of TNT in water

Different flow velocities were used to evaluate if the dissolution rate of TNT in saltwater was affected by differences in water currents, in this case flow rate passing over solid TNT. The results indicated that TNT dissolution commenced immediately upon contact with water flow and the results showed a direct relationship between the water velocity and the TNT dissolution rate, with faster dissolution observed at higher flow rates (Table 3).

Previous studies [32] showed that TNT dissolution rate in deionized water ranged from 20 to 50 μg/cm^2/min [32] which concurs with the results of this study where dissolution rate was observed to be between 35 and 74 mg/cm^2/min for the velocities in the range of 13–58 cm/s. The small observed difference can be attributed to the method of water contact: stirring in Ref. [32] *versus* direct flow over the TNT fragment in this study. In a stirred system water flow is largely uniform and tangential leading to more homogeneous exposure around the fragment, but potentially less direct contact with specific surfaces. In contrast, direct flow (as used in our experiments) creates a continuous flow over specific areas of the TNT surface, enhancing dissolution rates by maintaining a steep concentration gradient at the solid-liquid interface. The control, where the explosive was placed in the water without any flow, dissolved at a rate of 1.06 μg/min over the first 27 h. This low rate was expected as still water limits dispersion of the explosive materials, therefore limiting the dissolution rate. In contrast, the second control, where water flow was present but not directed on the TNT fragment, had a dissolution rate of 11.2 μg/min over the first 4 h. This rate was higher compared to the first control, indicating that water movement facilitated a higher dissolution rate. However, it was also noted that the dissolution was 68 % lower compared to the experiment with direct flow at the lowest velocity indicating that direct flow over the fragment increases the dissolution rate.

TNT in seawater dissolved over time at a similar rate when different velocities were applied however, dissolution of TNT was lower compared to distilled water with a dissolution ranging from 30 to 32 μg/cm^2/min (Table 1). The experiments demonstrated that in saline water TNT dissolves more slowly with concentrations 30 % lowered compared to distilled water over the same time.

The observed difference in dissolution rates between distilled water and seawater can be attributed to the presence of salts in seawater. The high salinity and solids content reduce the availability of water molecules to interact with and dissolve non-polar substances [47]. After 200 min in saltwater, it was noted that the concentration of TNT ranged from 6 to 7 mg/L regardless of the water flow rate; a concentration 50 % lower compared to the same velocities in distilled water. This differs with [33], who reported that TNT dissolution rates are similar in saltwater and freshwater conditions.

The TNT fragments exhibited varying mass changes during the experiments (Table 3), with distinct differences observed between seawater and distilled water. In seawater, the fragments showed a lower average mass decrease ranging from 2.1 to 7.9 mg, whereas in distilled water, the mass decrease ranged from 3.9 to 53.2 mg. A notable instance occurred at a flow velocity of 38 cm/s in seawater,

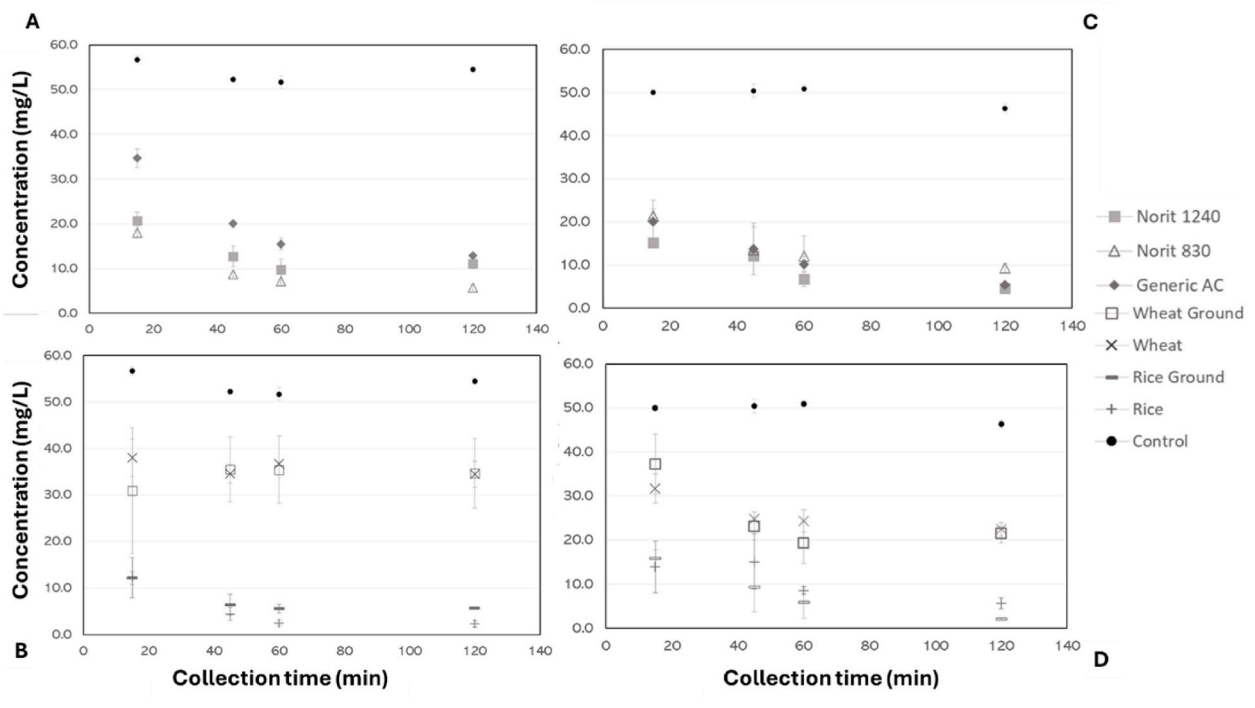

Fig. 2. – TNT concentration in mg/l in distilled water (A–B) and seawater (C–D) at different collection times (min). Results for ACs on the left (A–C) and biochar on the right (B–D) with standard deviation.

where the TNT mass decreased by 7.9 mg. In contrast, at a similar velocity (30 cm/s), the mass loss in distilled water averaged 70.4 mg, approximately ten times greater than that in seawater. These findings demonstrate the reduced capacity of seawater to dissolve TNT, resulting in slower dissolution rates that affect the quantity of dissolved explosive suggesting that it would take significantly longer for toxic levels of contamination to arise in saltwater environments. This would be further reduced by high flow rates diluting dissolved explosives over a wide area.

Based on the findings from this study, it was estimated that to dissolve a fragment of 150 mg in seawater it can take up to 50 h. This simplified estimate can potentially suggest that for underwater munitions, which can contain up to 10 kg of explosives, it will take a considerable amount of time to dissolve the explosive in the water thus not representing an acute short-term problem as suggested previously [30,48]. However, some ordnance stockpiles would have been dumped over 100 years ago, enough time for metal shells to corrode and expose the explosive fill which may have already had tens of years to leach and contaminate the environment. Contamination issues are more likely to arise from clearance activities where low order detonations deposit fragmented explosives residues in the marine environment. Although a slower dissolution rate of TNT in seawater is expected compared to distilled water, the possibility of degradation products may be reduced in seawater due to factors such as limited light, lower microbial activity, and lower oxygen levels, which typically stabilise TNT in these conditions [18,32,49]. Further research is needed to incorporate all the appropriate variables in the estimate.

3.2. TNT adsorption by activated carbon in saltwater

While accumulation of contamination from dumped munitions may take considerable time, short term low concentration contamination may arise due to clearance operations. It will be necessary to be able to rapidly deploy remediation solutions to these areas to minimise pollution and maintain the marine environment. Therefore, an initial investigation into the efficacy of carbon adsorption of TNT from seawater was conducted. As commercial AC is a commonly used industrial solution, Cabot Norit 8 × 30 mesh (N830), Cabot Norit 12 × 40 mesh (N1240) and Calgon Centaur (Generic activated carbon) were compared to biochar derived from rice husk and wheat straw. Investigation into the use of biochar derived from waste products as adsorbents has been steadily increasing as a low-cost alternative to commercial carbons with the additional benefit of being able to produce large quantities of biochar from local waste products. While obtained in granular form, samples were mechanically ground to increase available surface area and the two sizes compared (Fig. 2).

In both seawater and distilled water, the wheat biochar demonstrated lower performance compared to the other carbons and biochar, adsorbing only between 40 % and 58 % of the TNT present (Fig. 2b – d). Interestingly, the ground form of wheat biochar showed slightly better performance than its original form, indicating that increased surface area indeed enhances the biochar's adsorptive capabilities [40]. Rice husk showing increased adsorptive performance when ground, particularly in seawater. Overall, rice husk performed as the best material in seawater, adsorbing more than 60 % of the TNT within just 15 min. By the end of the experiment, concentrations of TNT remaining in distilled water and seawater were 2.36 ± 0.64 mg/L and 5.63 ± 1.24 mg/L, respectively, for unground rice husk, whereas ground rice husk showed concentrations of 5.72 ± 0.07 mg/L in distilled water and 2.11 ± 0.23 mg/L in seawater. This demonstrated that the increased surface availability of the ground rice husk was particularly beneficial for adsorption of TNT in seawater.

Similar variability was observed with activated carbons (Fig. 2a and 2c). Norit 830 performed comparably to ground rice husk in seawater, while NORIT 1240 was outperformed by ground rice husk. Although the high iodine number of NORIT 830 suggests it has a greater potential for adsorption, its enhanced performance is more likely due to its pore structure and surface area [36], which enhance interaction with dissolved explosives. Moreover, AC has proven effective in removing heavy metals from seawater [50], suggesting its potential as a versatile solution for purifying seawater from various contaminants arising from dumped ordnance. The adsorption processes involved two mechanisms: film diffusion and intra-particle diffusion. Film diffusion occurs as explosives migrate from water to the activated carbon surface, forming a monolayer that temporarily inhibits further adsorption until the contaminants penetrate the internal pore structure via intra-particle diffusion. However, the efficiency of AC in real seawater may differ depending on the specific composition as the salinity was artificially produced in this study to be generically representative. Further analyses of this interaction are discussed in Section 3.3 focussing on Norit 1240 and ground rice husk biochar. These were selected for further analysis in closed

Table 4
– Correlation Factor (R^2) calculated from isotherm correlation for the Langmuir, Freundlich and Temkin model for each Activated Carbon and Biochar in both distilled water and seawater.

Materials	Distilled Water			Seawater		
	R^2					
	Langmuir	Freundlich	Temkin	Langmuir	Freundlich	Temkin
Wheat (ground)	0.338	1	1	1	1	0.999
Wheat	0.998	0.972	0.999	0.999	0.999	0.997
Rice (ground)	0.532	0.985	0.987	0.999	0.014	0.966
Rice Husk	0.019	0.931	0.937	1	1	0.973
Norit 1240	0.997	0.975	0.984	0.999	0.778	0.926
Norit 830	0.764	0.990	0.991	0.999	0.995	0.999
Generic AC	0.364	0.991	0.993	0.998	0.816	1

systems as it is unlikely that granular carbon would be added to open water systems and saturated carbon must be collected for disposal.

3.3. Maximum adsorption capacity via analysis of isotherms

Adsorption occurs due to the interaction of intermolecular Van Der Waals forces between molecules, leading to the attraction and binding of contaminants onto the surface of an adsorptive matrix. Therefore, the capacity for adsorption primarily depends on the surface area, which in turn is influenced by the preparation method of AC which can alter surface groups, along with the inherent material structure derived from the primary source material [51]. Isotherm correlation factors can be used to infer the behaviour of TNT in both distilled water and seawater when the contaminant was in contact with the different adsorptive materials (Table 4).

The correlation factor of the Langmuir model for rice husk in seawater indicates that a monolayer forms rapidly, suggesting that intra-particle diffusion becomes the limiting step of adsorption before any significant diffusion occurs. This process completely differs from the rice husk in distilled water where a lower correlation factor (0.019) suggested that TNT is less likely to form a monolayer limiting further adsorption on the surface. This was confirmed by a greater correlation with the Freundlich model. This observation

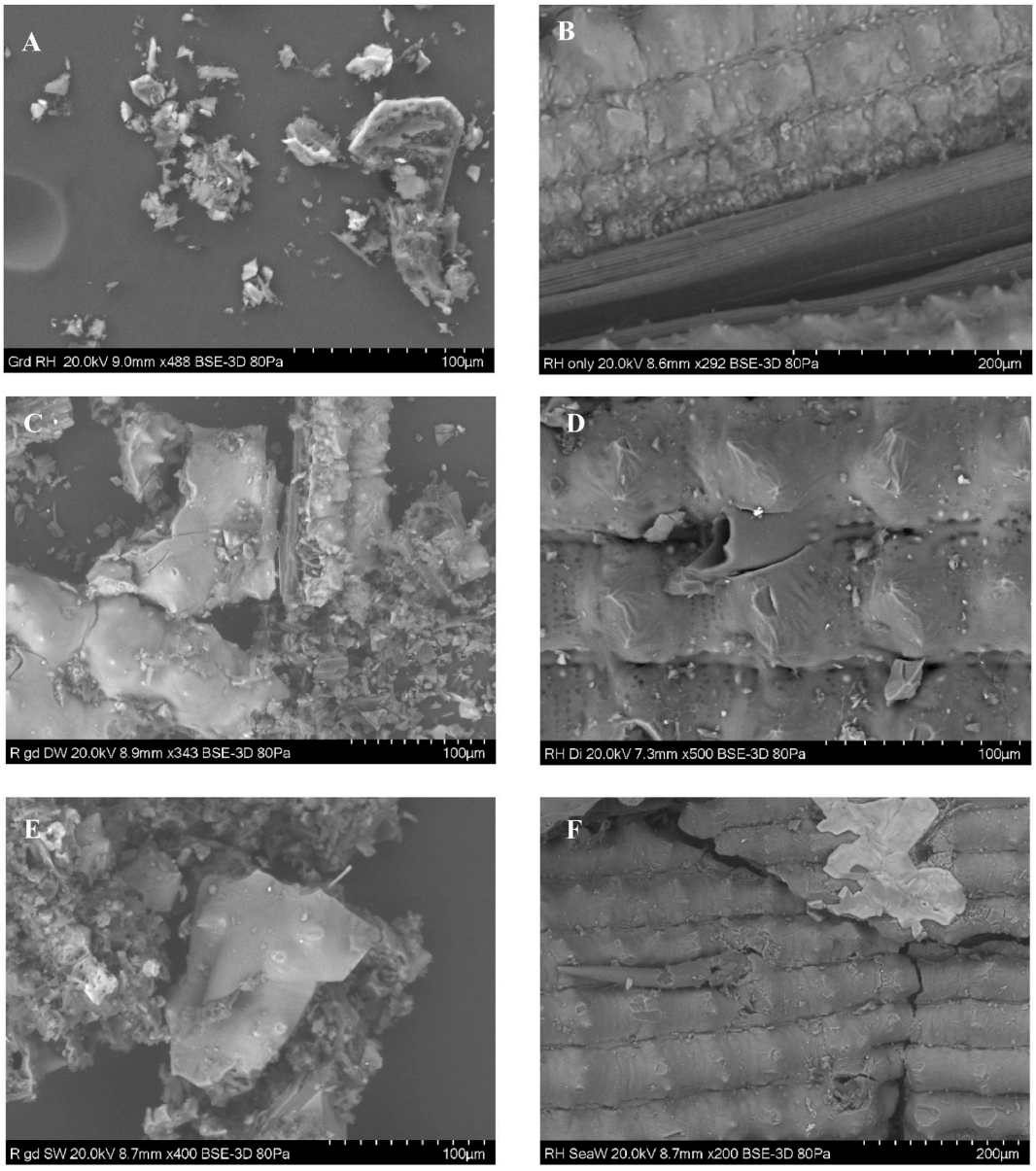

Fig. 3. – SEM images for A- Ground rice husk; B- rice husk; C – Ground rice husk in Distilled water; D – Rice husk in distilled water; E – Ground rice husk in seawater; F- Rice husk in seawater. Fig. 3D has been further magnified to render the surface area and the structure more visible.

contrasts with previous studies, where biochar materials typically favour the Freundlich model [52,53], indicating heterogeneous surface interactions. The ground rice husk experienced an opposite behaviour to the not ground equivalent, allowing rice husk to better adsorb TNT. This tendency to adsorb TNT over salts could explain why rice husk outperform commercial ACs in seawater, as their structure may allow for more efficient TNT adsorption. Further investigation into the biochar's surface and pore structure is needed to confirm this hypothesis.

Moreover, the inter-adsorbate interactions indicated by the Temkin isotherm model had a greater effect in seawater in general compared to distilled water, probably associated with the interaction between the salts, the adsorptive material and the TNT. Following the Temkin analysis, a negative B value was experienced across all the ACs and biochar in both distilled and seawater, indicating that the interactions that are occurring do not result in an exothermic reaction.

This relationship can be explained by the SEM analysis (Fig. 3) where the surface of the rice husk specifically, as the most efficient sustainable solution, was compared when in contact with both distilled water and seawater.

The scale of the captured images ranged from 100 μm to 200 μm, aiming to assess particle sizes and surface changes when rice husk interacted with distilled water and seawater. The rice husk was tested in both ground and non-ground forms, increasing the surface area exposed to TNT-contaminated water. In its original form, the rice husk grain sizes varied between 62 ± 1 and 58 ± 1 μm (Fig. 3b), whereas the ground rice husk ranged from 1 ± 0.5 to 300 ± 0.5 μm (Fig. 3a). The larger range of particle sizes for the ground rice husk reflects the increased diversity in particle sizes after grinding, which may have altered the overall surface area and adsorption characteristics. In distilled water, non-ground rice husk outperformed ground rice husk due to its smoother surface structure (Fig. 3d), which facilitates intra-particle adsorption and enhances TNT adsorption within the internal pores. A smoother surface can in fact facilitate intraparticle adsorption as it may allow for a more uniform distribution of the TNT within the pores or on the adsorbent surface. Conversely, in seawater (Fig. 3f), ground rice husk demonstrated better performance than non-ground rice husk. This difference can be attributed to the physical surface damage caused by seawater, where impurities, such as salts and other dissolved components, attach to the rice husk surface leading to alteration of the surface, reducing opportunities for TNT-Carbon interaction. Compared to ground rice husk (Fig. 3e), surface damage is evident on non-ground rice husk, with cracks visible across the entire grain.

3.4. *Efficiency of ground rice husk and Norit 1240 in a contained environment*

Adsorption of TNT was in general slower in the contained pod (Fig. 4B) experiments compared to the loose carbon, which was expected as it will take longer for contaminated water to passively saturate the carbon. Even so, TNT concentration fell below the limit of detection within 135 min of initial exposure with Norit 1240 (Fig. 4A). The ground rice husk did not perform as well with concentration after 135 min at 27.3 ± 0.79 mg/L. After 24 h (1440 min), the ground rice husk had adsorbed 95 % of the TNT that was present in the water leaving a concentration of 2.61 ± 2.36 mg/L. This performance was similar to the adsorption for granulated ground rice husk loose in solution as used in the previous experiment, showing that even in an enclosed space the ground rice husk has a comparable performance. Even though the efficiency of the ground rice husk was slightly lower compared to the Norit 1240, the use of waste materials presents a sustainable advantage in selecting rice husk over Norit.

4. Conclusions

This study demonstrates that TNT dissolves slower in the presence of salts compared to in distilled water system. The slower dissolution rate observed in seawater suggests that TNT may persist longer in marine environments. Furthermore, the investigation into ACs and biochar for adsorption-based remediation revealed that ground rice husk performs as well as commercially available NORIT 1240, capable of reducing TNT concentrations by more than 90 % in both distilled and seawater scenarios. Ground rice husk, in particular, emerges as a sustainable alternative to conventional ACs, highlighting its potential for future applications in marine environmental cleanup efforts. These findings not only contribute to our understanding of how TNT interacts with different water matrices but also offer practical insights into effective remediation strategies, although future research will focus on exploring the

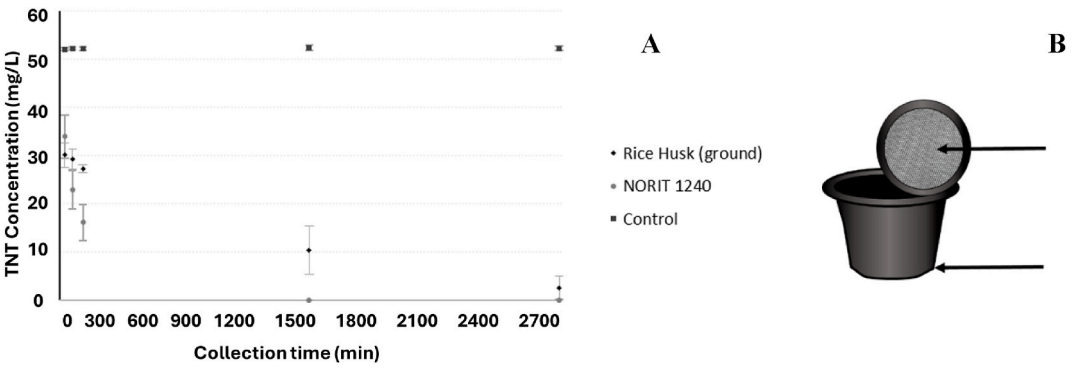

Fig. 4. – A - TNT concentration in mg/l after being in contact with ground rice husk and Norit 1240, placed in a contained environment, collection time (min). B – Enclosed environment with mesh at the top and bottom of the pod. All results are presented with standard deviation.

specific adsorption mechanisms to ensure a more thorough comparison that goes beyond the efficiency of these materials.

This study offered insight into the issues described above by conducting small-scale controlled experiments in a laboratory by using widely available equipment, while still producing valuable data to progress this area of research. Moving forward, more advanced research is needed into the behaviour of TNT in complex marine environments and the development of sustainable remediation technologies at larger scale by undertaking fully representative and reproducible studies as evaluating underwater munitions in seawater is extremely challenging with numerous variables.

CRediT authorship contribution statement

Joseph Sadler: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Matar Movshovitz:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. **Federica Persico:** Writing – original draft, Supervision, Investigation, Formal analysis, Data curation. **Natalie Mai:** Writing – review & editing, Methodology, Data curation. **Carmen Fernandez Lopez:** Writing – review & editing, Investigation. **Nilgun Sen:** Writing – review & editing, Investigation. **Lisa Humphreys:** Writing – review & editing, Investigation. **Evie Kadansky:** Writing – review & editing, Investigation. **Sally Webb:** Writing – review & editing, Investigation. **Bhumika Sharma:** Writing – review & editing, Investigation. **Josh Wardrop:** Writing – review & editing, Investigation. **Frederic Coulon:** Writing – review & editing, Supervision, Investigation. **Melissa Ladyman:** Writing – review & editing, Investigation. **Tracey Temple:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Data availability statement

Data supporting this study are openly available from https://doi.org/10.57996/cran.ceres-2632.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Professor Tracey Temple at the time of submission was an Associate Editor for Helyion. Professor Frederic Coulon is a Section Editor for Helyion. If there are other authors, they 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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