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Determination of Chemical Explosive Remnants in Soils of Boko Haram's Affected Areas of Gombe State Nigeria

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Authors' contributions

This work was carried out in collaboration between both authors. Author ONM designed, supervised and reviewed all the drafts of the manuscript. Author BAM carried out the research and wrote the first draft of the manuscript. Both authors read and approved the final manuscript.

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ABSTRACT

Aim: This research was conducted to ascertain the level of pollution of our physical environment as a result of years of Boko Haram's activities in Gombe State, North-Eastern Nigeria. **Study Design and Place of Study:** Samples for this research were collected from Bajoga, Nafada

and Gombe metropolis and environs all in Funakaye, Nafada, and Gombe Local Government Areas respectively. Samples were taken only from places with recorded Boko Haram's explosion activities. A control sample for each study area was taken from an area that has no record of Boko Haram attacks. Gombe State is located in the North-Eastern part of Nigeria on latitude10⁰15'00''N and longitude 11⁰10'00''E bordering Yobe, Borno, Adamawa, Taraba and Bauchi states.

Methodology: The concentrations of explosive remnants (4-Nitrotoluene, 4-Propyl Benzaldehyde, 1,3-Dinitrotoluene, 2,4-Dinitrotoluene, 3,5- Dinitrotoluene, Trinitrotoluene, RDX and HMX) in the soil samples of the attacked areas were investigated using Gas Chromatography- Mass Spectrometer, GC-MS.

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Results: The result indicates 4-Nitrotoluene; 0.05-0.085µg/kg, 4-Propyl Benzaldehyde; 0.07-0.19µg/kg; 1,3-Dinitrotoluene; 0.005-0.060µg/kg, 2,4-DNT; 6.16-6.86µg/kg, 3,5-DNT; 0.40-0.90µg/kg, TNT; 0.29-0.66µg/kg and RDX and HMX were not detected. All the samples in the study area had 4-Nitrotolene and 4-Propyl Benzaldehyde concentrations above those in their respective controls. 1,3-DNT in Bajoga samples, 2,4-DNT in K Police station and K/mata, 3,5-DNT in Gombe metropolis, and TNT in NFD Police station were all found to be below the concentrations in their control

Conclusion: There is possible 4-Nitrotolene and 4-Propyl Benzaldehyde contaminations in all the studied sites and absence of pollution by 1,3-DNT in Bajoga, 2,4-DNT in K Police station and K/mata, 3,5-DNT in Gombe metropolis, and TNT in NFD Police station.

Keywords: Explosives; remnants; soil; boko haram.

1. INTRODUCTION

Boko Haram ('Western education is a sin') was founded around 2002 in Maiduguri, the capital of Borno state and largest city in Northeast Nigeria [1]. During Boko Haram's insurgency, bombs, explosions and landmines were mounted in various locations and many casualties were recorded. The devastation to the environment and civilian population caused by cluster bombs will be a lingering and insidious nightmare against the environment and people. The bombs detonated have chemical by-products. Chemicals supporting war activities, such as Herbicides or chemical weapons, have effects that are seen for generations [2]. Warfare today uses explosives and machinery to subdue enemies and territories. The intensity of environmental damage resultina from wars has been parallel remarkably to the technological 'advancement' in warfare. Use of more advanced arms and ammunition means more damage to the environment [2].

Energetic compounds, defined as the active chemical components of explosives and propellants, are necessary both for peaceful and military purposes. Commonly used military energetic compounds include the explosives 2.4.6-trinitrotoluene (TNT), hexahydro-1,3,5trinitro-1,3,5-triazine (RDX), and octahydro 1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) [3]. In missile, rocket, and gun propellants, nitroguanidine nitroglycerin (NG), (NQ), nitrocellulose (NC), 2,4-dinitro toluene (DNT), and different perchlorate formulations are used Many energetic compounds and their [4]. byproducts have contaminated the environment as a result of military actions, incorrect management, and disposal procedures, to the point that the health of people, animals, wildlife, and ecosystems is in danger.

In people, TNT is associated with abnormal liver function and anemia, and both TNT and RDX have been classified as potential human carcinogens [5]. TNT and its metabolites have been used in investigations on the mutagenicity of Salmonella strains and mammalian cell lines [6]. It was discovered that TNT and several of its metabolites were both mutagenic. Convulsions are a common feature of RDX's effects on mammals. After being exposed to RDX, factory workers in Europe and the US experienced convulsions, unconsciousness, vertigo, and vomiting [7].

There are a variety of ways energetic molecules can reach the soil environment, including the following: (i) Ammunition manufacturing facilities, such as wastewater lagoons and filtration pits; (ii) Packing or storage facilities; (iii) Facilities for waste disposal and destruction, such as open landfills, burn pits, and incinerators; (iv) Weapons firing ranges; and (v) Weapon impact zones [8].

Soil contamination by energetics at manufacturing sites, conflict areas, and military ranges is an international concern. Numerous military installations are reported as being contaminated by energetic chemicals in the US [9]. Bombing and other training exercises have an impact on roughly 50 million acres. There could be even more polluted locations throughout Europe and Asia [10].

Military agencies in the US, Canada, and many European and Asian countries have been forced to identify sites of energetic contamination and assess the effects of military activities on the quality of soil, groundwater, and surface water over the past 20 years due to an increase in environmental awareness.

2. MATERIALS AND METHODS

2.1 Study Area

This research was conducted on samples collected from Bajoga, Nafada and Gombe and environs all in Funakaye, Nafada, and Gombe Local Government Areas respectively. Samples were taken only from places with recorded Boko Haram's explosion activities. A control sample for each study area was taken from an area that has no record of Boko Haram attacks.

2.2 Sampling

Soil sampling was carried out according to the method described by Aluko et al. [11]. each sampling location, five replicate From samples were collected. These were thoroughly mixed to give a homogenous sample, out of which 500g was packaged in tagged polythene bags. Control samples were obtained 1km off the sampling sites. All collected samples were properly tagged and identified by their sampling locations. Furthermore, the coordinates were obtained. using а Global Positioning System (GPS) receiver. The collected soil samples were taken to the research laboratory of the Defence Industries Corporation of Nigeria (DICON) Kaduna for analysis.

2.3 Sample Preparation

The soil samples were dried and sieved through a metal sieve (4-mm opening size) before fortification. The blank sample was analyzed to determine the background level of the analytes. It showed no presence of the target chemicals. The soil samples (20 g) were fortified with a stock mixture of standard chemicals in 100-150 mL of methanol-acetonitrile and were thoroughly (1:1, v/v) mixed. After the solvent was evaporated with a rotary evaporator, the soil was allowed to sit in a fume hood for 24 hours at room temperature. The fortified soil was then stored at 2-4°C for later use. A portion of this spiked soil was set aside and aged at room temperature (25°C ± 2ºC) prior to extraction. The spiking level was 2.5 µg/g for each analyte. Also, the same level of the explosives was spiked directly on top of the Ottawa sand the extraction in cells as а control for the extraction processes. All the fortification samples were in triplicates.

2.4 Extraction Procedure

A Dionex ASE 200 extractor was used for all extractions. The bottom of each 22-mL extraction was fitted with two cellulose filters cell covered with 2-3 cm³ sand. Each sample was mixed with 1-5 g of sand as a dispersion agent and was loaded into the cell. The sand was used to fill up the cell. The cell was loaded onto the ASE 200 extractor. An extraction cycle began with the filling of the cell with a mixture of methanol-acetonitrile (1:1, v/v), then a 5-minutes preheating time, followed by a 5-minutes static extraction. The extract was flushed out of the system with a 60-s nitrogen purge into a glass collection vial. The sample extract (~30 mL) was concentrated with a rotary evaporator and brought to 4-5 mL for GC-MS.

2.5 GC-MS Analysis

All the extracts were analyzed with a Varian chromatograph 3800/4000 gas mass spectrometer equipped with an Agilent equipped with a splitter split/split less. A DB-1 capillary column (J&W Scientific, Folsom, CA) was used. with dimensions of 10-m length, 0.18-mm i.d., and 0.4-um film thickness. It is necessary to use a shorter column (10 m. rather than 30 m) with a thicker film (0.4 µm, compared with 0.25 µm) to minimize degradation of the very active analytes during separation processes. The injection port liner was the Silchrom type (Restek, Bellefonte, PA) and held at 210°C. The GC–MS transfer line and ion-trap temperatures were 200°C and 210°C, respectively. The GC oven temperature started at 80°C for 1 minute, was ramped at 20°C/min to 140°C, then at 6°C/min to 200°C. The final temperature (200°C) was held for 16 minutes. The helium flow was 1.4 mL/min. The MS was operated in electron ionization (EI) and full scan modes to monitor a 100- 650-amu range. The total analysis time from extraction to complete GC-MS run was approximately 40 minutes/sample.

Organic compounds in the sample were identified in Wiley's NIST 08 Mass Spectral Library if the obtained comparison scores were higher than 95%. Otherwise, fragmentation peaks of the compounds were evaluated, and the compounds were identified using the memory background for the identification of the compounds that appeared GC/MS in chromatograms. Contents of individual compounds in the extract were given in percent of the total compound in the sample. This was the standard way to quantify most organic compounds in the honey samples. The chromatograms obtained from the total ion count (TIC) were integrated without any correction for co-eluting peaks and the results were expressed as total abundance. All the peaks were identified based on mass spectral matching (\geq 90%) from both the NIST and Wiley libraries. Only compounds with 90% or greater spectral matching accuracy are reported. No response factors were calculated.

3. RESULTS AND DISCUSSION

The concentration of explosive remnants in the soil samples from Boko Haram's explosion sites in Gombe metropolis was estimated using GCMS and the results are presented in Table 1 while Table 2 shows the concentration of the remnants in the soil samples collected from Bajoga and Nafada.

4-Nitrotoluene with a molecular weight of 137g/mole was detected by GC-MS analysis in the soil of the study area at a retention time of 11.80 minutes with a percentage peak area of 4.20- 4.51. The concentration of 4-Nitrotoluene was found to range from 0.005-0.085µg/kg. Soil sample collected from Military Quarter guard recorded the highest concentration while the lowest concentrations were recorded by K/mata and Main market soil samples. The distribution of the 4-Nitrotoluene in the samples were observed to be in the order: Quarter guard> Dadin kowa Park> Gombe Division> K. Police station, Timber market> K/mata and main market as shown in Fig. 1.

All the soil samples in Gombe metropolis were found to have higher concentrations of the 4-Nitrotoluene than the control sample which recorded a concentration of 0.0008µg/kg. This indicates possible contamination with 4-Nitrotoluene in all the sampling sites within the metropolis.

Nafada Police Station and Nafada Mosques samples had 4-Nitrotoluene's concentrations of 0.006 and 0.008µg/kg. These concentrations are above that obtained in the Nafada Control sample (0.003µg/kg). All the soil samples collected from Bajoga had 4-Nitrotoluene concentrations greater than that obtained in Bajoga control samples. The concentrations in Bajoga samples were in the order: Ashaka Junction> GRA/Union Bank> Bajoga Police Station with concentrations of 0.007, 0.006, 0.006, and 0.005µg/kg respectively (Fig. 2). 4-Nitrotoluene was not listed on the Health Effect Assessment Summary Table (HEAST) cancer table and was not classifiable as to human carcinogenicity because of inadequate evidence in humans and animals, and limited evidence for genotoxicity in mammalian systems [12].

4-propyl Benzaldehyde was detected at a retention time of 14.00 minutes and a percentage peak area of 5.41 – 5.91%. The concentration of propyl benzaldehyde in the soil samples from Boko Haram's explosion sites in Gombe metropolis ranged from 0.12µg/kg in Timber and Main markets to 0.18µg/kg in Quarter guard soil sample. The concentrations in all the soil samples analyzed from the sampling sites in Gombe metropolis were found to be above that obtained in the control Sample with 0.011µg/kg. The concentrations were in the order: Quarter guard> D/kowa Park> Gombe Division> K/Mata> Kwadon Police Station> Timber market and Main market as shown in Fig. 3.

Nafada and Bajoga samples. the In concentrations of 4-propyl benzaldehyde ranged from 0.07µg/kg to 0.19µg/kg as shown in Table The highest concentrations of propyl 2. Benzaldehyde in Nafada and Bajoga were observed in samples from NFD-Police Bajoga police station respectively while the lowest concentrations were observed in NFD-Mosque and Ashaka Junction samples respectively (Fig. 4).

All the samples recorded higher concentrations than their respective controls. This indicates possible propyl benzaldehyde contamination in the soils of the study area.

The results for the concentration of 1.3dinitrotoluene in the soil of Boko Haram's explosion sites in Gombe metropolis are shown in Table 1. This had a Molecular weight of 182g/mole and was detected by the GC-MS at a retention time of 21.48 minutes and peak area of 2.09 – 4.09%. The highest concentration was 0.014µg/kg in Timber market soil followed by 0.013µg/kg in D/kowa Park soil, 0.008µg/kg in main market soil, 0.007µg/kg in Gombe Division soil sample. Quarter guard, Kwadon Police station and K/Mata soil samples recorded the lowest concentrations of 0.005µg/kg. When compared with the control sample, The soil samples have 1,3-DNT concentrations a little above that in the control as indicated in Fig. 5.

Compound	Sampling locations									
	GDS	QGS	KPS	DPS	K/MATA	TMS	MMS	GMC		
4-NT	0.012	0.085	0.007	0.019	0.005	0.007	0.005	0.0008		
4-PB	0.15	0.18	0.13	0.011	0.14	0.12	0.12	0.011		
2,4-DNT	0.009	0.003	0.006	0.005	0.005	0.007	0.007	0.004		
1.,3-DNT	0.007	0.005	0.005	0.013	0.005	0.014	0.008	0.003		
2,4,6-TNT	0.1	0.17	0.14	0.18	0.11	0.15	0.009	0.009		
2,4-DNT	6.21	6.37	6.66	6.42	6.66	6.33	6.16	6.44		
3,5-DNT	0.53	0.59	0.51	0.6	0.55	0.4	0.5	0.62		
TNT	0.32	0.45	0.3	0.29	0.29	0.45	0.3	0.19		
RDX	ND	ND	ND	ND	ND	ND	ND	ND		
HMX	ND	ND	ND	ND	ND	ND	ND	ND		

Table 1. The Concentrations of explosive remnants (µg/kg) in soil samples from Boko Haram's explosion sites in Gombe metropolis

Table 2. The Concentrations of explosive remnants (µg/kg) in soil samples from Boko Haram's explosion sites in Bajoga and Nafada in Gombe state

Compound	Sampling locations										
	NPS	NMS	NSC	BPS	AJS	GRA	UBS	BJC			
4-NT	0.006	0.008	0.003	0.005	0.007	0.006	0.006	0.001			
4-PB	0.19	0.15	0.07	0.19	0.12	0.16	0.16	0.02			
2,4-DNT	0.008	0.006	0.008	0.006	0.012	0.008	0.007	0.005			
1,3-DNT	0.006	0.014	0.005	0.012	0.005	0.007	0.008	0.017			
2,4,6-TNT	0.16	0.13	0.15	0.64	0.15	0.11	0.11	0.18			
2,4-DNT	6.86	6.33	6.13	5.88	6.14	6.13	6.14	6.11			
3,5-DNT	0.9	0.58	0.6	0.71	0.58	0.51	0.80	0.6			
TNT	0.66	0.41	0.44	0.52	0.4	0.39	0.42	0.31			
RDX	ND	ND	ND	ND	ND	ND	ND	ND			
HMX	ND	ND	ND	ND	ND	ND	ND	ND			



Fig. 1. Distribution of 4- nitrotoluene in soils from Gombe metropolis





Fig. 2. Distribution of 4- Nitrotoluene in soils from Bajoga and Nafada



Fig. 3. Distribution of 4- propyl benzaldehyde in soils from Gombe metropolis



Fig. 4. Distribution of 4- propyl benzene in soils from Bajoga and Nafada

The concentrations of 1,3-DNT in the Nafada soil Samples are: $0.06 \mu g/kg$ and $0.014 \mu g/kg$ in NFD-Police station and NFD-Mosque respectively.

Bajoga Soil samples have concentrations all below that of the Bajoga control Sample. Higher concentration of 1,3-DNT in Bajoga control sample indicates an absence of 1,3-DNT pollution in Bajoga soils from Boko Haram attacked areas.

2,4-Dinitrotoluene was detected at the retention time of 24.95 minutes with a peak area of 18.0 -18.71%. The concentration of 2,4-DNT in soil samples in Gombe metropolis ranged from $6.16\mu g/kg - 6.66\mu g/kg$.

K. Police Station and K/mata soils have concentrations of 6.66 μ g/kg which was greater than the concentration in the Gombe control soil sample (6.44 μ g/kg) as in Fig. 7. Since the control sample had concentration above all the soil samples from Gombe metropolis except K. Police Station and K/mata soil samples indicating possible pollution only in these locations.

NFD-Police Station and NFD-Mosques have concentrations of 6.86µg/kg and 6.33µg/kg respectively which are both above the concentration in the NFD-Control (6.13µg/kg). This indicates pollution of the Sampling location by 2,4-DNT in Nafada Samples (Fig. 8).

All the Samples from Bajoga have almost the same 2,4-DNT concentrations including the Control Sample. This shows that there is no evidence of 2,4-DNT pollution in Bajoga Soil Samples.

The concentration of 2,4-DNT obtained in this study was lower than that reported by Pennington *et al.*, [4] at the cold lake Air-weapon range (0.20mg/kg). A higher concentration of 237mg/kg was also reported in the surface soil of Fort Lewis (WA) [13]. A 2,4-DNT concentration of <1mg/kg was found at the Donnelly Training Area (AK) [14]. The Concentration of 2,4-DNT in US and Canadian Artillery ranges was reported to decrease from 9.6mg/kg in the top 0-3cm to 0.56mg/kg at 10-20cm [4].

The retention time of 3,5-Dinitrotoluene was 25.26 minutes with a peak area of 9.33- 10.11%. The concentration of 3,5-Dinitrotoluene was found to be evenly distributed in the soils of Boko Haram's explosion sites in Gombe metropolis

ranging from $0.40\mu g/kg - 0.60\mu g/kg$. The distribution of this remnant was in the order: D/Kowa Park> Quarter guard> K/Mata> Gombe division> K. police Station> Main Market> Timber Market. It was observed that all the concentrations in the affected areas were less than the concentration in the control sample which recorded $0.62\mu g/kg$ (Fig. 9). it can therefore be suggested that no evidence of 3,5-DNT pollution from Boko Haram's activity within the study area.

In Nafada soil samples, NFD-Police station has 3,5-DNT concentration of 0.90µg/kg which was greater than 0.60µg/kg recorded by the NFD-control sample while NFD-Mosques sample recorded 0.58µg/kg 3,5-DNT control which was closely above the NFD Control. These indicate possible 3,5-DNT pollution from Boko Haram's attack on NFD-Police Station.

The concentration of 3,5-DNT in soil samples from BJG-Police Station, Ashaka Junction, GRA and Union Bank were; 0.71, 0.58, 0.51 and 0.80 μ g/kg respectively. The concentration in the control was 0.60 μ g/kg which was higher than that in Ashaka Junction and GRA but less than that in BJG-Police station and GRA (Fig. 10).

DNTs are widely used in manufacturing explosives and propellants as a gelatinizing, plasticizing and waterproofing agents in industries such as the munitions and mining industry. It is also used as a modifier for smokeless gunpowder in the munitions industry. DNT mixtures are predominantly used in the production of polyurethane polymers. These mixtures are also used as an intermediate in the production of dyes, plastics, herbicides and automobile airbags [15,16].

DNT is commonly deposited through live-fire and blow-in-place detonations at military ranges and found in waste streams and soil near munitions manufacturing and processing facilities [17].

Trinitrotoluene, TNT with a molecular weight of 227g/mole was detected at a retention time of 30.02minutes with a peak area of 6.12 - 6.82%. The concentration in the soil samples from affected areas of Gombe Metropolis were found to be; 0.32, 0.45, 0.30, 0.29, 0.29, 0.45 and 0.36µg/kg in Gombe division, Quarter guard, K. Police station, D/Kowa Park, K/mata, Timber Market and Main Market respectively (Fig. 11). The concentration of the TNT in the control Sample, 0.19µg/kg was found to be lower than

the concentration in all the samples in the study sites indicating no possible remnant contamination in the study sites.

The concentration in Nafada soil samples as shown in Table 2 were 0.66, 0.41 and 0.44 μ g/kg in NFD-Police Station, NFD-Mosque and NFD-Control respectively while that of Bajoga soil samples were; 0.52, 0.40, 0.39, 0.42 and 0.31 μ g/kg in BJG-Police Station, Ashaka Junction, GRA, Union Bank and BJG-control respectively. This suggests presence of remnant pollution in all sampling sites from Bajoga and NFD-Police station (Fig. 12).

TNT makes up around 99% of military grade TNT in most cases and additional components

including 2,4-DNT, 2,6-DNT, 1,3-DinitroBenzene and 1,3,5 TNB can also exist [18].

TNT concentration of 358mg/kg was reported in soil taken from a nearby M72 rocket explosion at the depth of 0 - 0.5cm and this concentration decrease to 1.7mg/kg at the depth of 6 - 10cm [4]. Residues of TNT at a concentration of <1-314mg/kg were also found at the Donnelly training area, AK [14]. At the cold Lake Airweapon range, Pennington *et al.*, [4] reported a TNT concentration of 3-408mg/kg. 2,4,6-Trinitrotoluene has been detected in surface soil samples at an average concentration of 13,000 mg/kg at the U.S. Department of Energy's Weldon Spring site in St. Charles County, Missouri [19].



Fig. 5. Distribution of 1,3-dinitrotoluene in soils from Gombe metropolis



Fig. 6. Distribution of 1,3-dinitrotoluene in soils from Bajoga and Nafada

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Fig.7. Distribution of 2,4- dinitrotoluene in soils from Gombe metropolis



Fig. 8. Distribution of 2,4- dinitrotoluene in soils from Bajoga and Nafada



Fig. 9. Distribution of 3,5- dinitrotoluene in soils from Gombe metropolis





Fig. 10. Distribution of 3,5- dinitrotoluene in soils from Bajoga and Nafada



Fig. 11. Distribution of trinitrotoluene in soils from Gombe metropolis



Fig. 12. Distribution of trinitrotoluene in soils from Bajoga and Nafada

At the West Virginia Ordnance Works located in Mason County, West Virginia, 2,4,6trinitrotoluene and other nitroaromatics have been detected in surface soils at burning sites in concentrations of up to 4% (40,000 mg/kg). Nitroaromatics, principally 2,4,6-trinitrotoluene, were detected at up to 20,000 mg/kg within 5-10 meters of the foundations of processing and refining facilities [19].

These literatures reported far greater concentrations than those reported in the soils of this study.

HMX and RDX were not detected in all the samples investigated in this study.

Many literatures reported HMX and RDX in varying concentrations in soils. An RDX concentration in the range of <1mg/kg – 50mg/kg and HMX of <1mg/kg – 9.1mg/kg were reported at the 23 Military installation in US and Canada [4]. At the grenade impact zones of Fort Lewis (WA) and Fort Richardson, RDX concentration of 51.2mg/kg and <0.1-0.5mg/kg respectively were reported by Jenkins et al. [20]. HMX concentration of up to 10,400mg/kg was obtained in the soil from a nearby M72 rocket explosion at 0-5cm [4].

4. CONCLUSION

The concentrations of explosive remnants in the soil of the study area (4-Nitrotoluene, 4-Propyl Benzaldehyde, 1,3-Dinitrotoluene, 2,4-DNT, 3,5-DNT, RDX and HMX were investigated. All the samples in the study area had 4-Nitrotolene and 4-Propyl Benzaldehyde concentrations above those in their respective controls. This indicates possible remnant contaminations in these locations. 1,3-DNT in Bajoga samples, 2,4-DNT in K Police station and K/mata. 3.5-DNT in Gombe metropolis, and TNT in NFD Police station were all found to be below the concentrations in their controls indicating absence of pollutions from the remnants in these locations.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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