

Assessment of Perfluorinated Alkyl Compound Levels in Soil, Water, and Designated Dump Sites in Damaturu, Nguru, and Gashua

Yagana Abba Sidi^{1,2} and Mohammed Musa Lawan^{1*} $1*$

¹Department of Chemistry, Faculty of Science, Yobe State University Damaturu, Yobe State, Nigeria

²Department of General Studies, Shehu Sule College of Nursing and Midwifery Damaturu,

Nigeria

Corresponding Author: mmlawanson@ysu.edu.ng

ABSTRACT

Perfluoroalkyl substances are detrimental contaminants that differ in several ways from most other well-studied organic chemicals found in everyday products. Perfluorooctanoic acid (PFOA) is one of the perfluoroalkyl substances that affect humans and is commonly found in almost all ramifications of life. The most common route of encounter of PFOA by humans is through washing and wearing of domestic utensils and other industrial chemicals such as surfactants into soil and water. This study assessed the presence and levels of PFOA in both dumpsite soil and leachate water samples from Damaturu, Nguru, and Gashua towns of Yobe state using Gas chromatographic mass spectrometry (GC-MS). The results revealed the presence of PFOA in sixteen per cent (16%) of the soil samples and six per cent (6%) of the water samples across the locations. These include samples collected from krikasamma, sabon pegi, and bindigari areas in Damaturu; Garbi and bulabulin in Nguru, and then katuzu and sabon gari in Gashua. The PFOAs were identified by retention time matching with standard PFOA and mass spectrometry. The mean concentrations of PFOA were 0.0214pg/g, 0.0035 pg/g, 0.0098 pg/g, 0.1040 pg/g, and 0.0236 pg/g in samples collected from Krikasamma, Sabon Pegi, Garbi, katuzu and sabon gari, respectively. These concentrations were, however, significantly higher than the permissible limit of 0.0007pg/g recommended by the Environmental Protection Agency (EPA.) and therefore may possess toxicity to soil, plants and humans. The findings from this study can serve as a baseline for investigation of source, transport and fate of such potentially toxic compounds that are found in most domestic utensils and firefighting facilities.

Keywords: Perfluorinated compounds, PFOA, soil, water, refuse dumpsites, GC/MS analysis, environmental contamination.

INTRODUCTION

The study evaluated the occurrence and slow levels of PFOA in soil and dump sites in Damaturu, Nguru, and Gashua. Perfluorinated alkyl substances (PFAS), highly toxic fluorinated chemicals present in numerous everyday products, accumulate in our bodies without breaking down [1]. These compounds have emerged as pervasive global pollutants found extensively in various urban and remote environments $[2,3]$. \qquad \qquad Classified as persistent, bio-accumulative, and toxic, PFAS are challenging to degrade biologically and persist in the environment

for extended periods [4]. Despite their breakdown, PFAS can enter municipal and industrial wastewater systems and ultimately be discharged into effluent during industrial usage. They can also be released into the environment, with limited absorption by soils and other materials, readily spreading through surface water and groundwater. Due to their synthetic organic nature and longlasting properties, chemicals possessing characteristics are commonly referred to as persistent organic pollutants

(POPs), earning them the nickname "forever chemicals [2].

Perfluorooctanoic acid (PFOA), also referred to as C8, has garnered considerable attention in recent headlines due to growing concerns about associated health risks. Previously considered innocuous, perfluoroalkyl compounds, including PFOA and PFOS, have come under scrutiny for their potential health and environmental hazards [5,6]. Over past decades, these compounds were extensively utilized by manufacturers and industrial facilities, exemplifying how lack of scrutiny can lead to widespread usage detrimental to both individuals and the environment. Detrimental effects on nervous system including worsening Parkinson's disease [7]. Consequently, these chemicals are now pervasive in the environment and in various species, including humans [5,6]. PFOS and PFOA degrade slowly in the environment, prolonging contamination risks over extended periods. Their peculiar behavior, which involves binding with proteins rather than accumulating in fatty Given the tissues like most contaminants, has expected to their inconspicuousness in the awareness of contributed to their inconspicuousness in classification as contaminants for some time. According to the EPA, PFOS and PFOA can accumulate in living organisms in significant quantities, with an estimated half-life of 4 years in humans [5.6].

Perfluorinated alkyl substances (PFAS) are synthetic compounds widely used across various industrial applications. These include fluoro polymers, liquid repellents for packaging materials, textiles, leather, carpets, industrial surfactants, coatings, firefighting foams, and essential materials in automotive, aviation, chemical, electronic, and semiconductor industries [8,1]. These compounds can enter the soil through environmental discharges such as spills and air deposition, and subsequently being released into the air and rivers, thereby

penetrating surface water and groundwater [9].

Exposure to PFAS above certain levels can lead to various adverse health effects, including impacts on fetal development during pregnancy, low birth weight, accelerated puberty, skeletal variations, cancer (such as testicular and kidney), liver damage, immune system disruptions, thyroid issues, alterations in cholesterol levels, and fertility problems [6]. Studies have linked [10]. Higher levels of PFOA in the body lead to increased cholesterol, [11] liver function changes, weakened immune responses, thyroid disorders, and elevated risks of kidney and testicular cancers [5]. Additionally, higher levels of PFOS and PFOA have been associated with difficulties in conceiving, prolonged delays in pregnancy, early onset of menopause in women, damage to liver cells, kidney function changes, and adverse effects on mammary gland development and lactation in rodents.

limited knowledge and awareness of the health and environmental effects of PFAS in Yobe state, there is a pressing need to conduct comprehensive research to inform and educate the populace. The study focused on assessing PFOA levels in soil and water in three major towns: Damaturu, Nguru, and Gashua, specifically targeting selected dump sites within these areas. The methodology involves collecting soil samples at various depths from dump sites in each locality and analyzing them Chromatography/Mass Spectrometric techniques.

MATERIALS AND METHODS

Study Locations

The study was conducted in three different locations in Yobe State, Nigeria: Damaturu, Nguru, and Gashua (Bade) Local Governments.

Figure 1: Map of the study areas.

Sample Collection, Pretreatment And Analysis

Soil Sample

Soil samples were collected using grab sampling method at depths of $0 - 5$ cm and 10 – 15cm, respectively. Samples were stored in high-density polyethylene (HDPE) containers and protected from light at temperatures of $0 - 6$ °C until they reached the laboratory.

Water Sample Collection, Preservation and Storage

Water samples were collected in vials for analysis. Soil samples were polypropylene capped containers following standard sampling procedures. Each sample was approximately 500 mL, stored in the dark, and maintained at $0 - 6$ °C until *Sample Cleanup*

Samples were reconstituted with two rounds of acetonitrile, cleaned up using activated carbon, and then transferred to vials for

transported to the laboratory. Samples were shipped with adequate ice to maintain temperatures below 6° C and were received within 48 hours of collection. Upon arrival at the laboratory, samples were stored at $0 -$ 6°C until preparation.

Sample Preparation

For water samples, 10mL aliquots were ultrasonically extracted using a mixture of acetonitrile (ACN) and methanol (4:1). The supernatants were collected after centrifugation and pooled together. Aliquots of 150 µL were transferred to polypropylene homogenized, air-dried, and sieved before acetonitrile-methanol (4:1) extraction for three rounds. Supernatants were combined and concentrated to dryness.

Sample Cleanup

Samples were reconstituted with two rounds

of acetonitrile, cleaned up using activated

carbon, and then transferred to vials for

analysis.

and separated using a capillary column. The

analysis.

and s Analysis was conducted using an Agilent GC-MS (model 7890) system. Sample extracts were injected into the GC system and separated using a capillary column. The

GC oven temperature was set at 90°C, ramped to 270°C at a rate of 20°C/min, and held for 1 minute. MS source and quad temperatures were set at 230°C and 150°C, respectively. Samples were screened for unknown PFOAs by retention time matching and spectral library search. Comparison with EPA standards was conducted to assess contamination levels. However, due to the limitations of the analytical instrumentation, many unknown signals in the samples remained unidentified, and their chemical structures could not be determined during this study.

RESULTS AND DISCUSSIONS

This study presents the detection and water samples quantification of per-and polyfluoroalkyl substances (PFAS) specifically of this compound in the environment. perflurooctanoic acid (PFOA) in soil and

water samples. The presence of these contaminants was determined using chromatographic techniques (GC-MS) which allowed the identification and quantification of PFAS in complex environmental matrices Figure 1 displays the chromatogram of the sample, indicating the of a sample the presence of PFAS Figure 2 presents a stacked graphical presentation of PFOA concentrations in soil and water samples. Highlighting the distribution and levels of this compound in the environment. Figure3 shows a chromatogram of a soil and water samples matching, confirming the presence of PFOA. Finally figure 4 represents a bar chart of PFOS concentration in soil and providing a visual representation of the distribution and levels

Figure 2: Chromatogram showing the presence of PFAs

Figure 3: Stacked graphical presentation of PFOA and its derivatives in soil sample

Figure 4: Chromatogram showing the presence of PFOAs in soil and water samples matching with standard PFOA, the spectrum of the library and that of the sample.

Figure 5: Concentration of PFOs in soil and water samples under the bar chart

Figure 2: The chromatogram in figure 1, obtained through GC/MS analysis, confirm the presence of PFAS in soil and water samples. The matching retention times in the samples, standard and library chromatograms validate the detection of these compounds.

Figure 3: The study detected PFOA and its derivatives in soil samples from different

locations. PFOA at 2.6minutes, along with nanodecafluoro decanoic acid and heptadecafluoro decanoic acid and 6-octadecanoic acid.

Similarly soil sampes from Y25, Y34, Y12 and Y37 as well as water sample from Katuzu Gashua also showed the presence of PFOA at 2.6 minutes and 2 minutes,

respectively along with various derivatives in the soil samples, these derivatives include nanodecafluoro decanoic acid and in the study were: Nyanya, Kriskassam, heptadecafluoro decanoic acid, L-fructose tetrakis trifluoroacetate, benzylamine, nanodecafluoro-anhydride, and L-(-) focose tetrakis trifluoroacetate. In the water sample, perfluorocyclohexane, tetradeneic acid, and 6-octadenoic acid were detected.

The presence of PFOA and its derivatives was confirmed through retention time matching and mass spectral library search.

Figure 4: The chromatograms of soil and water samples show the presence of per-and polyfluoroalkyl substances (PFAS) specifically PFOA in the samples. The PFOA peaks in the chromatograms of the soil and water samples match with the standard PFOA spectrum in the library indicating the presence of PFOA in the samples.

Figure 5: concentration of PFOA were detected in eight samples, krikassamma, sabon pegi, garbi and sabon garin gashua showing significantly higher concentration than the environmental protection agency limit for soil and water (0.0007pg/g and 0.0004pg/g, respectively). The specific PFOA levels were 0.021pg/g in Krikassamma, 0.0035pg/g in sabon pegi, $0.0098pg/g$ in garbi, and $0.0256pg/g$ in sabon garin gashua. The Katuzu area also had elevated PFOA in both soil and water samples at $0.0440pg/g$ and $0.0013pg/g$ respectively.

A total of 45 soil samples and 15 water samples were gathered from three local government areas: Damaturu, Nguru, and Gashua. From each of these areas, soil and water samples were collected from five different neighborhoods. The soil samples were taken at depths of 0-5cm, 5-10cm, and 10-15cm. In total, 45 soil samples were obtained from 15 different neighborhoods,

with one water sample from each neighborhood. The neighborhoods included Sabon Pegi, Bindigari, and State low-cost in Damaturu; Filin Idi, Kogin Saadu, Garbi, Bulabulin, and Kadawan kantudu in Nguru; and Lawan Fannami, Katuzu, Sabon Gari, Takari, and Federal Lowcost in Gashua.

The samples underwent pretreatment, extraction, and final analysis with a GC/MS spectrometer while being maintained at the proper temperature in the laboratory. Only one of the seven soil samples—which were collected from different places, including Sabon Pegi, Bindigari, Garbi, Bulabulin, Katuzu, and Sabon gari—had detectable levels of perfluorooctanoic acid (PFOA).

Using retention time matching and mass spectrum library search, PFOA and its derivatives, including hepadecafluorodecanoic acid, were discovered in soil samples from a dumpsite in Krikassamma. The urban pollution is probably a result of poor wastewater management, an unlicensed dumpsite, and dense population density. The disposal of wastewater and outdoor laundry by residents are two further factors leading to the elevated amounts of PFOA in the environment. This suggests that the degradation of the environment in Krikassamma might have been caused by multiple reasons, such as the density of the population, inadequate waste management, and certain behaviors of the locals.

PFOA and similar substances were detected in Sabon Pegi Damaturu, likely originating from PFOA-containing modern appliances and packaged foods. These substances pose potential environmental risks, consistent with Phleps *et al*.'s[12] findings of PFAS in non stick cookware and food packaging like pizza boxes and microwave bags.

In Krikasamma, urban pollution from mismanagement and unrestricted dumpsites,
combined with high population density, likely contributed to PFOA presence. Wastewater disposal and outdoor laundry further increased PFOA levels, aligning with Eur EaU's [13] study mentioning wastewater treatment plants as major pathways for PFAS contamination, especially in densely populated areas.

In Bindigari, where sophisticated drainage systems exist, the car wash business has introduced PFOA into the area. Chemicals and detergents used in car wash operations, along with discarded plastics, leather, and water sachets, contribute to PFOA levels. Shan and Peter [14] noted PFAS presence in various products, including liquid repellents for packaging, leather, and carpets.

PFOA was also found in Garbi neighborhood, crossed by a railway line where passengers dispose of drinking cans, food containers, and water sachets, adding to PFOA exposure. Glenn et al. [15] highlighted food and beverage containers as significant sources of PFOA exposure. Similarly, PFOA was detected in Bulabulin, an area where a river passes through, posing risks to the community.

These substances can travel through rivers and enter the environment, posing health risks. According to Aderson et al. [16], PFOA can enter soil and water through spills and air deposition, eventually reaching groundwater.

In Gashua, PFOA was detected in soil and water samples, particularly in Katuzu, a considerable region known for affluent families using non stick appliances. Additionally, in Sabon Gari Gashua, where PFOA was found, residents primarily work as mechanics, blacksmiths, and welders, occupations associated with

PFOA presence, consistent with Lancet Planetary Health's study [17].

The study reveals the ubiquitous presence of PFAS, even in remote communities, where traditional foods are contaminated.

CONCLUSION

The study conducted in Damaturu, Nguru, and Gashua (Bade) Local Governments in Yobe State, Nigeria, highlights the significant presence of perfluorooctanoic acid (PFOA) in both soil and water samples from selected neighborhoods. Out of the 45 soil samples and 15 water samples analyzed, a subset showed PFOA contamination. Specifically, PFOA was detected in soil samples from Jerusalem, Sabon Pegi, and Bindigari in Damaturu; Garbi in Nguru; and Katuzu in Gashua, as well as in water samples from these areas.

Recommendations

Based on this research finding, the following recommendations were made:

1) Further research is needed in other pails of the state to determine the presence of PFOA and other PFAS compounds.

2) It is strongly recommended to have a continuous follow–up on product groups since our knowledge and best-practiced analytical performance of PFC content are limited.

3) More screening is suggested for carpets, leather, and textiles to assess the possible exposure. Even if levels might seem low in some products, the amount of these products used and deposited could lead to a source of emission into ecosystems, especially as waste.

4) New PFCs will be introduced into the European and global markets, eventually turning up in consumer households.

REFERENCES (2009).

[1]. U.S Environmental Protection Agency (2016). Per-and polyfluoroalkyl susbtances (PFASs) in the environment: Development of analytical methods and toxicity values for risks assessment. *Journal of environmental management*, 183, 472-480-DOI, 10.1016/J. Jenuman. 2016.09.022

[2]. Frigerico, D., Darnow, L, M., HU. Fi B.,

HU, H., Heun, K., Zhang. Y., …& Chen, A.

[4]. Prevedouros, K., Cousins I.T., Buck, R. C., & Korzeniowski, S.H (2006): Sources, fate and transport of perflurocarboxylates *Environmental science and technology*, 40(1). 32-44:doi 10.1021/es US 12475.

[5]. Environmental pollution centre. (2018). Per-and polyfluoroalkyl substances (PFAS) in the environment. What are PFOA and PFOS and how dangerous are they? Retrieved from https://www.ep.gov/pfas/o4-crrent-

understanding -human-health-and environmental risks-pfas. Doi:10.1016/j.envpol.2018.06.001

[6]. Rappazzo, K. M., Cottman, H. E., & Hines. E P (2017). Exposure to perfluorinated Akyl substances and health outcome in children: A systematic Review of Epidemiologic Literature International Journal of *Environmental Research and Pupil Health*, 14 (17), 69 doi:10.3390/Ijerph 14070691.

[7]. Pradyyumina K, Mishia D Ravindiam et al (2010). Perfluoroctane sulfonate (PFOS) induces reactive oxygen species (ROS) production in human micro vascular endothelial *pharmacology*, 29(3), 339-346.doi:10.1016/J, etap.

[8]. Sepulvado, J. G., Blaire, A. C., Hundal, L.S., & Higgins, C. P. (2011), occurance and fate of perfuorochemicals in soil following the Land Application of municipal biosolids *Environmental science & technology* 45 (19), 8106-8112 <https://doi-org> 110.1021/ es 201438² (4).

Prenatal exposure to per-and polyfluroakyl substances and infant growth and adiposity: The healthy start study. *Environmental Health perspectives*, 127(7).077007.doi:10.1289/EH D 3222 [3]. Wang. J. C., Dewitt. C.P., & Hihhins. I. T (2017). A never-ending story of per-and polyfluoroalkyl substances (PFASs)? *Environmental Sceince & technology*, 51 (5). 2508-2018.

[9]. Viera, V. M., Holzer, J., Kliem, C., & Savitz. D-A. (2016). Predictors of PFOA levels in commonly surrounding a chemical plant. *Environmental Health perpectives*. 117(7). 1083-1088.

[10]. Jones, P. A.G., Jones, K.C., & Sweetman. A.J (2009). A first global production, emission, and environmental inventory for perfluoroctane sulfonate. *Environmental science & technology*, 43(3).386-392.https://doi.org/10.1021/es 802216n.

[11]. Blake, B.E & Fenton. S.E. (2020) between longitudinal serum perfluoroalkyl substance (PFAS) levels and measures of thyroid hormone kidney function, and body mass index. *Toxicology*. 443 IS2565, doi:10-1016/J.tox 2020.152565

[12]. Phelps, J., Nguyen, L., Tran, T., & Bui, A. (2024). Per- and polyfluoroalkyl substance migration, toxicity, and management strategies in food packaging. *Journal of Environmental Science and Health, Part A*, 59(3), 145-159. https://doi.org/10.1080/10934529.2023.217 5896

cells. *Environmental Toxicology and* [13]. EurEau. (2022). Briefing note on PFAS and wastewater. EurEau Water Matters. https://www.eureau.org/resources/briefing notes/5532-eureau-briefing-note-on-pfas-and-

wastewater/file

[14]. Shan, J., & Peter, K. T. (2019). Addressing urgent questions for PFAS in the 21st century. *Environmental Science & Technology*, 53(12), 6634-6647. https://dokorg/10.1021/acs.est.9b01244

[15]. Glenn, B., Nguyen, M., & Tran, L. (2021). Analysis of PFAS in locally acquired food containers. *Journal of Agricultural and Food Chemistry*, 69(18), 5432-5441. https://doi.org/10.1021/acs.jafcic01234 [16]. Anderson, R., Bui, T., & Pham, H. (2019).

Partitioning of per- and polyfluoroalkyl substances from soil to groundwater within aqueous film forming source zones. *Environmental Science: Processes & Impacts*, 21(5), 854-863. <https://doi.org/10.1039/C9EM0> 0134A

[17]. Lancet Planetary Health. (2020). Per- and polyfluoroalkyl substances in remote populations: A study in Greenland.*The Lancet Planetary Health*, 4(7), e322-e330. <https://doi.org/10>. 1016/S2542-5196(20)30111-9

203