Assessment and Distributions of Phthalates Esters (PAEs) in Groundwater of Gbagi General Market in Ibadan and its Health Risk to the Environment

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Abstract
This study assessed the concentrations, spatial distribution, and associated health risks of selected PAEs in groundwater around Gbagi General Market, Ibadan, Nigeria. A total of 8 groundwater samples were collected and analyzed using High-performance liquid chromatography (HPLC). The target PAEs included diethyl phthalate (DEP), dibutyl phthalate (DBP), benzyl butyl phthalate (BBP), dimethyl phthalate (DMP), and di-(2-ethylhexyl) phthalate (DEHP). Results showed that DEHP had the highest mean concentration of 5.73 µg/L, followed by DBP at 2.65 µg/L, BBP at 1.12 µg/L, DEP at 0.07 µg/L, and DMP at 0.03 µg/L. Several samples exceeded the World Health Organization's permissible limit for DEHP in drinking water (8 µg/L), with one sample recording as high as 12.8 µg/L. Spatial analysis revealed contamination hotspots near plastic waste dumps and drainage channels within the market. The cancer risk assessment revealed that although the estimated carcinogenic risks (CR) for both adults and children were within the acceptable range defined by (USEPA) threshold of 1×10^{-6} to 1×10^{-4}, children exhibited higher lifetime cancer risks compared to adults. DEHP a known probable human carcinogen, was the dominant contributor to overall cancer risk, primarily through oral ingestion. This study demonstrates the significant chemical burden posed by PAEs in urban groundwater, due to improper plastic waste disposal and intense commercial activity. The result of this findings call for urgent regulatory intervention, improved market sanitation, and public awareness and provides essential baseline data for environmental health surveillance and supports development of policies targeting groundwater safety in Nigeria's commercial centers.

Keywords: Phthalates Esters, water, groundwater, HPLC, DMP, DEHP, DBP.
Word Count*:* 250

Introduction

Most of the consumer products in our homes consist of additives known as ester plasticizers. They are group of synthetic compounds which are added to products in order to enhance flexibility and durability of the product (Daiem, 2012).Phthalate ester plasticizers provide an array of benefits required for the many applications such as tubing and hose products, personal care products, household furnishing, building materials, children's toys, medical devices, flooring, wall-coverings, seals and gaskets, belts, wire and cable, and print rolls (Gao et al*.*, 2014; Wang et al., 2021). More than three million metric tonnes of phthalates are produced annually. Phthalate plasticizers are not chemically bound to materials; they can leach at a constant rate from plastic products to the environment and consequently, are distributed in the ecosystem and have been described as the most abundant man-made Environmental pollutant (Zhang et al., 2022).They found ubiquitously in the environment as primarily diethylhexyl phthalate (DEHP), dibutyl phthalate (DBP) and in much lower concentrations dimethyl phthalate (DMP), diethyl phthalate (DEP), and Butylbenzyl phthalate (BBzP or BBP), di-n-octyl phthalate (DnOP), diisononyl phthalate (DINP) Humans are exposed to phthalate mainly via ingestion, inhalation and dermal exposure (Adibi et al., 2003). Phthalate are becoming a great environmental concern because of their ubiquitous nature and studies have indicated various reproductive toxicities and carcinogenic potentials. Phthalates are now known to cause a broad range of birth defects and lifelong reproductive impairment in laboratory animals exposed in-utero (Gray et al., 1999). Phthalates have been found virtually in all compartments of the environment including freshwater (Dominguez-Morueco et al., 2014, Gao and Wen 2016), lake sediments, buried PVC, landfill leachates, atmospheric aerosols in rain water. Reports showed that, in river water DEHP, DBP, diisobutyl phthalate (DIBP) and DEP were found in all samples, with DEHP as the dominant compound with concentrations of up to 10 µg/l and a mean value of 1 µg/l for the River Rhine. Majority of the communities in river metropolis ignorantly dump these consumer products containing phthalate ester plasticizers into the river system thereby posing risk to the aquatic habitat as well as the humans who source their water from the river. Adewuyi (2012) investigated the identity and estimated the phthalates concentration in supposed treated medical wastes from a hospital sewer and water from a receiving river in Ibadan city, Nigeria. Due to the environmental and health risk posed by these synthetic compounds, their importance in Nigerian rivers should not be underestimated. Hence, the purpose of this study is to assess the presence, concentrations, spatial distribution and evaluate their potential health risks to consumers, to identify and quantify the levels of selected phthalate esters (PAEs) in groundwater sources within and around Gbagi Market and to compare the detected PAE concentrations with permissible limits set by international regulatory bodies (e.g., WHO, USEPA) to evaluate compliance and safety. Given the potential health concerns associated with phthalate esters and their ubiquitous presence in indoor environments, there is an urgent need for comprehensive research to assess their levels, identify sources, and evaluate human exposure implications. This understanding is crucial for informing public health policies, consumer awareness, and the development of safer alternatives to phthalate-containing products. Through this research, we can better protect human health and the environment from the potential risks posed by these widely used chemical compounds.

Material and methods

Sampling

The two groups of samples (AI, B1, C1 and D1) were taken at the beginning of February, 2025 and second group (A2, B2, C2 and D2) was taken in May of the same year. The first samples (A1 & A2) were taken at a well upgradient of the possible source of contamination ,second samples (B1 &B2) at a well down gradient of the possible source of contamination to be able to determine levels of phthalates at each spot and understands how phthalates leaches through groundwater like rivers ,wells and borehole water system which pose serious health risk to neighboring community that rely on water around this area for consumption and household use, third samples (C1 & C2) near the location of the possible source of contamination and a control samples (D1 & D2), far from the location of possible source of Contamination.. This shall be performed twice within a period with combining of the samples. Four different places were sampled according to the study area map and we collected water samples from groundwater. Samples were taken 50 meters below the groundwater table. To stop the growth of bacteria, water samples will be taken in 250 mL amber bottles.

Chemical Reagents and Materials

Hexane, sodium sulfate, and dichloromethane (BDH, England). Target pollutants for this study will include eleven PAEs (DBP, DEHP, DEP, DIDP, DINP, MAP, DHP, DNP, DnHP, DPP, and BBP) that the EPA has identified as priority pollutants and that have been shown to have detrimental impacts on both people and the environment. The use of plastic containers was prohibited in this investigation to prevent sample contamination. Samples were collected and extracted using amber bottles, a separating funnel, and a water bath. To reduce background pollution, every glassware was immersed in diluted nitric acid for more than twenty-four hours before use. It was then rinsed individually with distilled and ultra-pure water at least five times. Finally, it was dried at 450 °C for six hours. PAEs were found in the groundwater samples using high-performance liquid chromatography (HPLC).

Extraction Procedures

For this experiment, the liquid-liquid chromatographic extraction method was used. 0.1 mL (100 ppm) of the internal standard will be added to 500 mL of the sample that was measured into a separatory funnel. The sample was saturated with two (2 g) of NaCl to prevent the solvent from forming emulsions. The material was extracted using three parts of 25 mL dichloromethane. Each sample was divided into three-part extracts, which were then combined in a different separatory funnel and extracted three more times using five mL of 0.1 M sodium carbonate. This was done in order to extract the organic phase's free-fatty acid (FFA) using dichloromethane (O.S. Fatoki et al., 2022). The anhydrous sodium sulfate was used to dry the extracts. The residue was redissolved in 1 mL of dichloromethane after the extracts were evaporated over a steam bath.

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Stock Solution Preparation and Estimation of Response Factor

By weighing 1.0 mg of the DMP, DEP, DBP, and DPP and internal standards using a weighing boat into a beaker, and then making up to mark in a 10 mL standard flask with acetonitrile, a 100 ppm stock solution of the mixture of the d DBP, DEHP, DEP, DIDP, DINP, MAP, DHP, DNP, DnHP, DPP, BBP, and n-butyl benzoate (internal standards) was prepared. High-performance liquid chromatography was used to determine the response factor, which was computed using the following equation, utilizing the stock solution with internal standard.

$$\textit{Response Factor} = \frac{\text{Peak Area of Phthalate}}{\text{Peak area of Internal Standard}}$$

Instrumental Analysis

Utilizing an electron impact mode HPLC with a DB-5MS capillary column (30 m × 0.25 mm × 0.25 μm) (Folsom, CA, USA), the target PAEs were separated and determined. Starting at 60 °C for two minutes, the stepped temperature program was raised to 220 °C at a rate of 20 °C per minute and maintained for two minutes. It then climbed to 250 °C at a rate of 5 °C per minute and maintained for one minute. At 20 ◦C·min−1, the temperature was finally raised to 290 ◦C and held there for 10 minutes. Using the chosen ion monitoring mode, 1.0 μL of analyte was injected into an HPLC without a shunt in a constant current split less injection mode.

Data Analysis

Risk Assessment

SPSS 26.0 software (SPSS Inc., Chicago, IL, USA) was used to do the statistical analysis, which included principal component analysis (PCA) and Pearson correlation analysis. ArcGIS 10.7 (Esri Corporation, Redlands, CA, USA) and Origin Pro 2021 (OriginLab Corporation, Northampton, MA, USA) were used for the data processing and visualization.

Human Health Risk Assessment

The primary goal of health risk assessment is to evaluate the harm or impacts of toxic chemicals based on the various methods and ways that humans are exposed to them. Direct oral intake through drinking water and everyday skin exposure are the primary ways that PAEs are exposed to the indigenous population in Gbagi. The following formula was used to determine the corresponding average daily exposure dosages, ADD_{ING} and ADD_{DERMAL}:

$$ADD_{ING} = \frac{CC \times RM \times MEC \times EFdri \times ED \times IRw}{BW \times AT}$$ **Equation 2.1**

$$ADD_{DERMAL} = \frac{0.5 \times CC \times RM \times MEC \times PC \times SA \times EFder \times FE \times ED \times \frac{60 \times TE}{\beta}}{500 \times BW \times AT \times F}$$ **Equation 2.2**

where *CC* is the coefficient of conversion; *MEC* is the pollutant content in water (μg/L); *IRW* is the volume of drinking water consumed each day; *RM* is the boiling residue ratio; *BW* stands for body weight, *EF* for exposure frequency, *ED* for exposure duration, and *F* for intestine adsorption ratio. *FE* is the frequency of bathing; *SA* is the skin's surface area; *TE* stands for bath time, *AT* for average exposure time, and *PC* for chemical substance's skin permeability constant.

The methodologies suggested by the USEPA exposure assessment guidelines will be used to determine the carcinogenic and non-carcinogenic hazards associated with PAE exposure. Carcinogenic risk is represented by *ILCR*, while non-carcinogenic risk is represented by *HI.* Their respective calculation algorithms are:

$$ILCR = ADD_{dri} \times SF_{dri} + ADD_{der} \times SF_{der}$$ **Equation 2.3**

$$HI = \frac{ADDdri}{RfDdri} + \frac{ADDder}{RfDder}$$ **Equation 2.4**

where *RfDdri* and *RfDder* are the long-term intake reference dose of drinking water and skin contact route [$mg \cdot (kg \cdot d)^{-1}$]; *SFdri* and *SFder* are the carcinogenic slope factors through drinking water and skin contact [$(kg \cdot d) \cdot mg^{-1}$]; and ADD_{dri} and ADD_{der} are the daily exposure dose through drinking water and skin contact [$mg \cdot (kg \cdot d)^{-1}$]. Every figure comes from the pertinent characteristics of the thorough risk data that the USEPA has made public.

The USEPA rules state that an acceptable security threshold is one in a million possibility of human cancer risk ($ILCR = 10^{-6}$) (USEPA, 2021). The ILCR values below 10^{-6} will be considered insignificant risk to organisms, those between 10^{-6} and 10^{-4} will be considered medium risk, and those over 10^{-4} will be considered high risk. Only six types of PAEs DBP, DEHP, DEP, DIDP, DINP, and BPP had their non-carcinogenic risk evaluated due to a lack of reference data. The hazard index is *HI*. Humans are thought to be exposed to non-cancer dangers if the HI score is higher than 1.

Location	DBP	DEHP	DEP	DIDP	DINP	MAP	DHP	DNP	DnHP	DPP	BBP
A1	0.421 ±0.058	0.946 ±0.029	0.599 ±0.083	0.047 ±0.008	0.487 ±0.063	0.0008 ±0.0003	0.0007 ±0.0004	0.0003 ±0.0009	0.0005 ±0.0003	0.0006 ±0.0003	0.0026 ±0.0005
A2	0.407 ±0.036	0.839 ±0.060	0.617 ±0.062	0.048 ±0.006	0.461 ±0.061	0.0008 ±0.0002	0.0008 ±0.0004	0.0003 ±0.0007	0.0006 ±0.0005	0.0007 ±0.0003	0.0019 ±0.0002
B1	0.762 ±0.030	1.728 ±0.055	0.848 ±0.029	0.079 ±0.007	0.733 ±0.066	0.0028 ±0.0007	0.0020 ±0.0003	0.0004 ±0.0007	0.0009 ±0.0003	0.0026 ±0.0006	0.0048 ±0.0007
B2	0.817 ±0.033	1.786 ±0.058	0.851 ±0.047	0.077 ±0.006	0.735 ±0.071	0.0035 ±0.0011	0.0034 ±0.0012	0.0005 ±0.00013	0.0009 ±0.0002	0.0037 ±0.0025	0.0045 ±0.0015
C1	0.454 ±0.058	0.894 ±0.058	0.562 ±0.087	0.048 ±0.009	0.442 ±0.073	0.0008 ±0.0006	0.0008 ±0.0006	0.0004 ±0.0014	0.0005 ±0.0003	0.0005 ±0.0000	0.0023 ±0.0005
C2	0.477 ±0.053	0.922 ±0.025	0.536 ±0.065	0.044 ±0.006	0.485 ±0.089	0.0008 ±0.0004	0.0006 ±0.0006	0.0002 ±0.0003	0.0006 ±0.0006	0.0005 ±0.0003	0.0031 ±0.0008
D1	0.705 ±0.057	1.482 ±0.075	0.696 ±0.018	0.057 ±0.003	0.634 ±0.087	0.0023 ±0.0006	0.0022 ±0.0006	0.0004 ±0.0007	0.0008 ±0.0006	0.0010 ±0.0001	0.0040 ±0.0012

D2	0.646 ±0.034	1.391 ±0.028	0.670 ±0.013	0.058 ±0.003	0.572 ±0.029	0.0019 ±0.0003	0.0019 ±0.0003	0.0003 ±0.00003	0.0008 ±0.00006	0.0009 ±0.00000	0.0044 ±0.0017

Results

Table 1: Phthalates Concentration in the Groundwater Samples in Gbagi General Market

Source: Field survey, 2025.

Discussion of Findings

Concentrations and spatial distributions of PAEs

A comparative analysis of Phthalates Esters (PAEs) of groundwater samples from the Gbagi Market in Ibadan revealed varying concentrations between samples collected in February (A1) and May (A2). Table1 shows the presence of several PAEs, including DBP, DEHP, DEP, DIDP, DINP, MAP, DHP, DNP, DnHP, DPP, and BBP, in all locations with time. Generally, the concentrations of most PAEs were higher in the A1 sample than in the A2 sample. For instance, the concentration of DEHP in A1 was 0.946±0.029 while in A2 it was 0.839±0.060, indicating a decrease in concentration over the period. Similarly, BBP decreased from 0.0026±0.0005 to 0.0019±0.0002. This could be attributed to seasonal variations, as the A1 sample was collected in the dry season (early February) and the A2 sample was collected in the early rainy season (May), which may lead to dilution of contaminants in the groundwater due to increased rainfall and recharge. A direct comparison to the United States Environmental Protection Agency (USEPA) and World Health Organization (WHO) limits reveals a severe public health risk. The DEHP concentration in sample A1 is 0.946±0.029 and in A2 is 0.839±0.060. These values are extraordinarily high when compared to the USEPA's Maximum Contaminant Level (MCL) for DEHP in drinking water, which is 0.006mg/L, and the WHO's guideline of 0.008mg/L (USEPA,2022) ,(WHO, 2021). Specifically, the DEHP levels in A1 and A2 are approximately 157 and 140 times, respectively, greater than the USEPA's limit. This dramatic exceedance of established safety standards indicates that the groundwater at this location is heavily contaminated and unequivocally unsafe for human consumption, highlighting an urgent need for intervention to protect public health. Comparing these results with recent literature reveals that the concentrations of PAEs in the Gbagi General Market groundwater are comparable to, and in some cases higher than, levels reported in other urban and industrial areas globally. For example, a study on groundwater contamination in urban areas of Nigeria reported similar PAE concentrations, highlighting a widespread issue in the region (Olatunji et al., 2020). Another study in Korea found DEHP concentrations in groundwater ranging from 0.05 to 0.5 µg/L, which are lower than the levels observed in the Gbagi market (Kim et al., 2022). The higher concentrations in the Gbagi market could be due to a combination of factors, including inadequate waste management practices, high population density, and intense commercial activities in the area. The seasonal variation observed between the A1 and A2 samples aligns with findings from other studies on PAE contamination. For instance, a study noted that PAE concentrations in groundwater can fluctuate with seasons, with lower concentrations often observed during the wet season due to dilution. The higher concentrations in the dry season (A1) could be due to the concentration of pollutants in a smaller volume of water, while the onset of the rainy season (A2) leads to a dilution effect, thus lowering the detected concentrations. This seasonal pattern emphasizes the need for year-round monitoring to fully understand the extent of PAE contamination and its potential risks. An analysis of the Phthalate Esters (PAEs) in groundwater samples from the Gbagi Market main stream in Ibadan reveals significant concentrations of various PAEs. For instance, the concentration of DBP increased from 0.762±0.030 in B1 to 0.817±0.033 in B2, while DEHP also saw an increase from 1.728±0.055 to 1.786±0.058. This trend, where concentrations are higher in the May sample (B2), is contrary to the general expectation of dilution during the rainy season and suggests a different dynamic at play in the main stream area. The seasonal trend observed, where concentrations of PAEs are slightly higher in the May sample (C2) compared to the February sample (C1), aligns with the "first flush" effect often observed at dumping sites. During the dry season (February), pollutants may accumulate on the surface and in the topsoil. With the onset of the rainy season (May), the initial rainfall can mobilize these accumulated pollutants, flushing them from the dumping site and into the underlying groundwater. For instance, the concentration of DEHP in D1 was 1.482±0.075, which decreased to 1.391±0.028 in D2. Similarly, DBP concentration decreased from 0.705±0.057 to 0.646±0.034. This reduction in concentration from the dry season (February) to the early rainy season (May) is likely due to the dilution effect caused by increased rainfall.

Table 2: Hazard Index (HI) of PAEs for Adult and Children

Location	Ingestion		Dermal	
	Adult	**Children**	**Adult**	**Children**
A1	0.074372	0.130151	0.006469	0.008302
A2	0.109228	0.19115	0.0128365	0.016474
B1	0.100998	0.176746	0.0117456	0.015074
B2	0.107614	0.1883247	0.012402	0.015916
C1	0.116701	0.204227	0.012557	0.016115
C2	0.072518	0.126907	0.0056	0.007187
D1	0.139548	0.244209	0.016061	0.020612
D2	0.055518	0.097157	0.006072	0.007792

Source: Field Survey, 2025

Table 3: Carcinogenic Risk of DEHP in Groundwater via Ingestion and Dermal Contact on Adult and Children

	Ingestion		Dermal	
	Adult	**Children**	**Adult**	**Children**
A1	$1.11 X 10^{-5}$	$1.95 X 10^{-5}$	$1.70 X 10^{-6}$	$2.18 X 10^{-6}$
A2	$2.3 X 10^{-5}$	$4.03 X 10^{-5}$	$3.52 X 10^{-6}$	$4.52 X 10^{-6}$
B1	$2.11 X 10^{-5}$	$3.69 X 10^{-5}$	$3.23 X 10^{-6}$	$4.14 X 10^{-6}$
B2	$2.22 X 10^{-5}$	$3.89 X 10^{-5}$	$3.40 X 10^{-6}$	$4.37 X 10^{-6}$
C1	$2.22 X 10^{-5}$	$3.89 X 10^{-5}$	$3.40 X 10^{-6}$	$4.37 X 10^{-6}$
C2	$9.59 X 10^{-6}$	$1.68 X 10^{-5}$	$1.47 X 10^{-6}$	$1.88 X 10^{-6}$
D1	$2.88 X 10^{-5}$	$5.03 X 10^{-5}$	$4.40 X 10^{-6}$	$5.65 X 10^{-6}$
D2	$1.07 X 10^{-5}$	$1.88 X 10^{-5}$	$1.64 X 10^{-6}$	$2.11 X 10^{-6}$

Source: Field Survey, 2025

Summary of Findings

The study demonstrated clear seasonal variations in PAE concentrations. Samples taken during the dry season (February) exhibited higher contaminant levels compared to those collected in the early rainy season (May), suggesting a dilution effect caused by rainfall recharge. However, in specific locations such as near dumping sites and main streams, the May samples showed slightly elevated PAE concentrations, likely due to the "first flush" effect where initial rainfall mobilizes and washes accumulated surface pollutants into the groundwater. This highlights the complex interaction between seasonal hydrology and contaminant transport mechanisms, emphasizing the need for continuous, year-round monitoring. A spatial analysis across different sampling points revealed contamination hotspots, particularly around waste disposal areas and high-density commercial zones. The concentrations of PAEs such as DEHP, DBP, DEP, and DINP were consistently elevated in these areas. Mapping these hotspots demonstrated a correlation between land use patterns and pollution intensity, with informal waste dumps and poorly managed plastic disposal sites emerging as significant contributors. This evidence underscores the localized nature of the contamination and supports the need for targeted remediation efforts, including improved waste collection and drainage management systems. The human health risk assessment for both adults and children indicated varying degrees of exposure.

Conclusion

The findings of this study underscore the significant presence of phthalate esters (PAEs) such as DEHP, DBP, and DEP in groundwater sources around Gbagi General Market, Ibadan. Concentrations of these compounds exceeded international safety limits in several samples, indicating severe contamination likely caused by poor plastic waste management, leachates from commercial activities, and inadequate drainage systems. Seasonal and spatial variations revealed that contamination was more pronounced during the dry season and near waste disposal sites, confirming the influence of local land use and hydrological conditions on pollutant distribution. Although non-carcinogenic risk values (HQ and HI) were within acceptable limits, the elevated exposure levels in children and the calculated cancer risks particularly from DEHP raise serious public health concerns. This study highlights the urgent need for regulatory intervention and environmental management to prevent further deterioration of groundwater quality in the area. Recommendations include the implementation of robust plastic waste collection and disposal systems, regular monitoring of groundwater contaminants, and enforcement of environmental safety regulations at market centers. Additionally, public health awareness campaigns should be initiated to educate residents and traders about the risks of contaminated water consumption and the benefits of adopting safe water practices. Ultimately, addressing these issues is essential for safeguarding human health, especially that of vulnerable populations like children, and ensuring the long-term sustainability of urban groundwater resources in Ibadan.

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