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Research Paper

Assessment of Eleven Priority Phenols in Surface Water of Ughelli River, South-Southern Nigeria

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ABSTRACT : *The identification and quantification of individual phenols is essential for proper understanding of the impacts of phenols on the environment and human health. In this work, surface water concentrations of eleven (11) priority pollutant phenols enlisted by United State Environmental Protection Agency (USEPA) were determined in Ughelli River, using Gas Chromatography (GC) coupled to Flame ionization Detector (FID). The mean values of 27.54, 10.64, 0.182, 0.085, 0.342, and 0.423 µg/L were obtained for phenol; 2-chlorophenol; 2-nitrophenol; 2,4-dimethylphenol; 2,4-dichlorophenol; and 4-chloro-3-methylphenol respectively. While for 2,4,6-trichlorophenol; 2,4-dinitrophenol; 4-nitrophenol; 2-methyl-4,6-dinitrophenol; and pentachlorophenol, the mean values were: 0.232, 0.572, 0.972, 11.14, and 7.16 µg/L respectively. The total for the eleven priority phenols in the various sites range from 41.09 – 116.57µg/L with a mean of 59.29 ± 32.53µg/L. These values compared to the European Union (EU) standard of 0.5 µg/L for total phenols content and 0.1 µg/L for individual phenols in drinking water, are quite high and deserves attentions.*

KEYWORDS: *Ughelli river, priority pollutants, phenols, water quality, health status*

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I. INTRODUCTION

The health status of man is largely influence by the quality and quantity of water available to him. Pollution resulting from anthropogenic activities have resulted in decrease in the qualities of most surface water throughout the world. Among the pollutants of increasing concern is phenol and its related compounds – generally called phenols; a collection of aromatic compounds characterized by hydroxyl (-OH) functional group directly attached to a carbon atom that is part of the aromatic ring. They have wide range of applications in household products and industrial synthesis. They are used as disinfectants (antibacterial and antifungal agents) in household cleansers, creams, lotions, ointments and mouth wash. In the dye industries they are used to make coloured azo dyes. Others such as creosols (methyl phenols) and chlorophenols have functions as wood preservatives. Phenols are use in agricultural practice, for the productions of herbicides and insecticides and can be derived from degradation of the chlorophenoxycarboxylic herbicides and organophosphorous insecticides [1]. Also, alkylphenols can result from the transformation of alkylphenol polyethoxylates, present in detergents as non-ionic surfactants. Phenols are also used in the production of medicinal and industrial organic chemicals. Phenolic resins which have wide applications in moulded articles, insulation in electrical equipment (such as circuit board), household laminates, gluing and bonding building materials etc., are products of condensation polymerization of phenols and formaldehydes [2]. Phenols though occur naturally in water and soil environment as a result of the decomposition products of plants, vegetation and animal waste, their excessive presence in the environment usually results from domestic, agricultural and industrial activities.

The excessive presence of phenols in water environment, represent great treat to aquatic life and human health. Many researchers have implicated phenols to be highly toxic, exhibiting carcinogenic, teratogenic and mutagenic properties with resultant adverse effects on human health [1, 3, 4]. A more recent concern is the estrogen-mimicking nature of several environmental phenols. The estrogen receptor (ER) does bind with the hydroxyl residues of phenols instead of the 17β-hydroxyl group of the hormones thus disrupting the sexual hormones functions. This can result in several conditions such as feminization of the male species, development of breast and endometrial cancer, decreased libido, lower sperm count, cryptorchidism, prostates enlargement, and subsequent sterility of animals and humans [5 – 8].

As a result of their toxicity, some phenols are included in the lists of priority pollutants in many countries and are required to be determined [9, 10]. Eleven common phenols viz: phenol (Ph), 2-chlorophenol (2-CP), 2-nitrophenol (2-NP), 2,4-dimethylphenol (2,4-DMP), 2,4-dichlorophenol (2,4-DCP), 4-chloro-3-methylphenol (4-C-3-MP), 2,4,6-trichlorophenol (2,4,6-TCP), 2,4-dinitrophenol (2,4-DNP), 4-nitrophenol (4-NP), 2-methyl-4,6-dinitrophenol (2-M-4,6-DNP), and pentachlorophenol (PCP) have been enlisted in the United State Environmental Protection Agency (USEPA) priority pollutants list (USEPA) [10]. The official standard method for the determination of total phenols in many countries is by molecular spectrophotometry based on the colour formed by oxidative coupling of phenols with 4-aminoantipyrine (4-AAP) in alkaline solution in the presence of potassium ferricyanide, or with 3-methyl-2-benzothiazoline hydrazone (MBTH) in presence of ammonium ceric sulphate. This method however, provides no information as to the nature of each phenols; the position, type and number of substitutions on the phenolic ring influences its reactions, hence their effects on aquatic lives and human health [11 – 13]. Animal studies for instance, have shown that 4-nitrophenol is more toxic than 2-nitrophenol [14], while, Shang et al. [15], revealed that the amount of ozone required to detoxify the chlorophenols (CPs) solutions into complete non-toxic condition follows the order: 4-CP > 3CP > 2CP. They also noted that the intermediates oxidized CPs induced new toxicity during the early stage of ozonation, and that ozonated 2-CP showed greater degree of toxicity increase than 3-CP and 4-CP. The large inductive effect of chlorine on the hydroxyl group of the ortho-chlorinated phenols makes them more acidic than their other isomers. The toxicity of the chlorophenols have been found to increase with the degree of chlorination [16, 17]. The rate of biodegradation which most time has an inverse relationship with toxicity also, has been shown to vary with the ionization constant (pKa), which is largely a function of the nature of the substituent(s) phenols [18]. Analytical determination using Gas Chromatography couple to Flame Ionization Detector (GC/FID) allows for the identifications of individual phenols in water matrix even at low concentration with good precision and accuracy. In this work, the eleven priority pollutants phenols were determined in surface water of the Ughelli River using GC/FID.

II. METHODOLOGY

2.1. study area.

The Ughelli river flows north-south and empties into the Atlantic Ocean through the Forcados estuary (Fig. 1). Ughelli is located between latitude $5^{o}13^{'}$ and $5^{o}45^{'}$ of the equator, and between longitude $5^{o}51^{'}$ and $6^{o}12^{'}$E of the Greenwich Meridian. The area is characterized by the presence of some industries, such as: oil and gas, petrochemicals, wood processing, metals and alloys, rubber latex, agricultural and allied companies. The presence of this industries, coupled with the establishment of the School of Heath and Technology, Ufuoma – Ughelli, has led to the influx of migrants into the area with resultant increase in population.

2.2. sampling and preparation

Water samples were collected from the surface (0 – 30cm depth) of the study area into an amber glass bottle with Teflon-line screw cap. At each site 3 grabs samples were mixed to form composite samples used for the study. A total of five (5) composite samples were taken from the river; the sample sites were at least 3km apart. The sources of the samples (A, B, C, D, & E) are as shown in Fig. 1

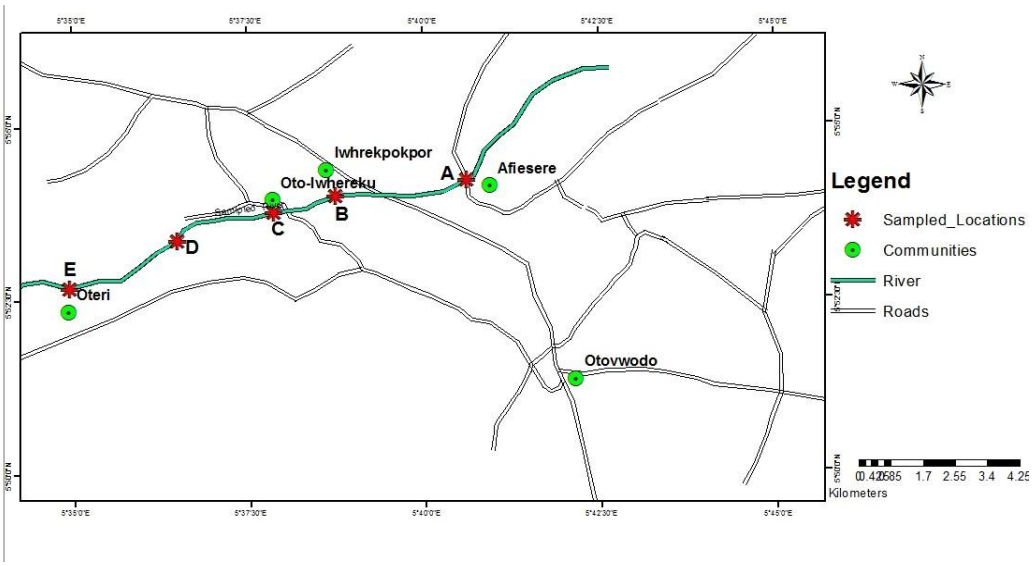

Fig.1: Map of the study area showing the sampled locations

2.3. determinations of physico-chemical properties

Analysis of physico-chemical properties were carried out using standard procedures [19, 20, 21]. The pH was determined with pH meter (model 410A) after standardization of the associated electrodes with buffer 9 and 4 respectively, temperature was determined by 100^{o}C range thermometer, turbidity by the turbidity meter after standardization with 0 and 10 NTU polymer standard solutions, conductivity were determined with the aid of conductivity meter (model RE387TX), Total Dissolved Solids (TDS) were measured using the same conductivity meter with TDS functional mode selected, while the five-day Biochemical Oxygen Demand (BOD_5) were evaluated from depletion in oxygen content after a five-days incubation period using the azide modification of Winkler's method. The Chemical Oxygen Demand (COD) were determined by oxidation with potassium dichromate followed by titration of the excess dichromate with ferrous ammonium sulphate.

2.4. extraction and GC-FID analysis of water samples

The water samples were extracted by liquid-liquid extraction (LLE) technique. 600mL of water sample that have been acidified to a pH 2 by addition of sulphuric acid (1:1v/v), was measured into a separating funnel and extracted three (3) times by 60mL of dichloromethane (DCM). The combined extracts were dried over Na_2SO_4 and concentrated to 2mL by a rotary evaporator. The extract solvent was then exchange to 2-propanol, and reconcentrated to 2mL by rotary evaporation/gentle N_2 blowing. 1µL of the concentrated extract was injected into the GC/FID for phenol separation and analysis.

2.5. analysis

The separation was performed on a fused silica capillary column (DB-5, 20m X 0.15mm X 0.15µm). the carrier gas was helium at a flow rate of 1.5mL/min. Samples were injected in split less mode and the volume of sample injected was 1µm. The injector and detector temperature were 250^{o}C and 300^{o}C respectively. The oven temperature was programmed as follows: initial column temperature was 90^{o}C, increased to 240^{o}C at a rate of 15^{o}C/min. and held for 5min. The method completion time was less than 15minutes. The phenols were identified by comparing their retention time with those of corresponding standards and quantification was done by evaluating the area under the peaks.

2.6. quality control

Pesticide grade solvents were used for the analysis. Mix standard solutions of phenols in propan-2-ol were used to performed method validation and quality control with correlation coefficients for calibration curves all higher than 0.9964. Recovery test was performed by spiking blank samples with the mix standards. The recoveries range were within 92 -107% indicating the suitability of the method.

III. RESULTS AND DISCUSSION

3.1. Some Physico-chemical Properties of the River water

The pH of the river water was slightly acidic; with mean value of 5.49 ±0.23. The five-day Biochemical Oxygen Demand (BOD_5) and Chemical Oxygen Demand (COD) were in the range of 3.53 – 11.89mg/L and 5.79 – 27.32mg/L respectively suggesting that the pollution load of the river was quite low. Other physicochemical properties of the river water are as shown in Table 1.

Table 1: Some Physico-Chemical Properties of the River Water.

Properties	Range	Mean ± SD
pH	5.39 – 6.25	5.49 ±0.23
Temperature (^{o}C)	25.94 – 26.23	26.11 ± 0.27
Turbidity (NTU)	0.93 – 9.74	5.33 ± 1.72
Conductivity (µS/cm)	54.57 – 65.70	62.53 ± 3.67
Total Dissolved Solids (mg/L)	35.69 – 43.51	39.27 ± 3.57
BOD_5 (mg/L)	3.53 – 11.89	5.38 ± 1.08
COD (mg/L)	5.79 – 27.32	17.57 ± 2.75

3.2. Concentrations of Phenols in the River water

Table 2 shows the concentrations of each phenols and the total of the eleven (11) priority phenols in the various sampled locations; while Table 3 gives a statistical description of the phenols' concentrations in the sampled locations.

Table 2: Concentrations (µg/L) of phenols in surface waters of Ughelli River at the various locations

Types	Concentrations (µg/L)				
	A	B	C	D	E
Ph	27.01	37.79	21.90	23.89	27.11
2-CP	8.91	18.39	5.990	8.618	11.28
2-NP	0.178	0.376	0.010	0.034	0.314
2,4-DMP	0.296	0.052	BDL	0.012	0.064
2,4-DCP	0.146	1.244	0.084	0.034	0.202
4-C-3-MP	0.324	0.508	1.136	0.070	0.078
2,4,6-TCP	0.278	0.638	0.034	0.198	0.012
2,4-DNP	0.010	2.290	0.098	0.208	0.254
4-NP	0.172	1.244	0.280	0.046	3.118
2-M-4,6-DNP	1.70	35.47	5.354	5.856	7.318
PCP	2.26	18.57	6.204	3.726	5.062
Total	41.28	116.57	41.09	42.69	54.81

Table 3: Statistical descriptions of phenols concentrations in the surface water of Ughelli river

Type	Range(µg/L)	Median(µg/L)	Mean(µg/L)	SD(µg/L)	% contribution
Ph	21.90 – 37.79	27.01	27.54	6.1372	46.45
2-CP	5.997 – 18.39	8.91	10.638	4.7212	17.94
2-NP	0.01 – 0.376	0.178	0.1824	0.1632	0.3076
2,4-DMP	BDL – 0.296	0.052	0.0348	0.1210	0.1430
2,4-DCP	0.034 – 1.244	0.146	0.342	0.5082	0.5768
4-C-3-MP	0.70 – 1.136	0.324	0.4332	0.4385	0.7138
2,4,6-TCP	0.012 – 0.638	0.198	0.232	0.2528	0.3913
2,4-DNP	0.01 – 2.29	0.208	0.572	0.9651	0.9647
4-NP	0.046 – 3.118	0.280	0.972	1.2899	1.6394
2-M-4,6-DNP	1.70 – 35.47	5.856	11.1396	13.7574	18.788
PCP	2.26 – 18.57	5.062	7.1644	6.544	12.08
Total (11)	41.09 – 116.57	42.69	59.29	32.53	100

The value for phenol (Ph) ranged from 21.90 µg/L in sample C to 37.79µg/L in sample B. The mean value was 27.54 µg/L and the median value was 27.01 µg/L. According to the European Community Council (ECC) directive specification [9], the legal tolerance for each phenol intended for human consumption is 0.5 µg/L. The value in this report, compared to that of the standard, is high and connotes contamination with respect to phenol. Also, when compared to the United State Environmental Protection Agency (USEPA) [22] maximum contaminant level (MCL) of 1 µg/L for drinking water, the values are still higher. Zhou et al [4], determined phenol in surface water of China River Basin and obtained a range of 56.73 – 137.35, and 35.72 – 97.85ng/L phenol for wet and dry seasons respectively with mean of 101.68 and 71.04ng/L phenol for the respective wet and dry seasons. Their values are comparatively lower than that of this report.

The range for 2-chlorophenol (Table 3), was from 5.992 – 18.39 µg/L. The mean value stood at 10.638 µg/L while the median was 8.91 µg/L. These values are quite high when compared to the legal tolerance level of 0.5 µg/L specified by ECC [9], and the maximum contaminant level of 1 µg/L of the USEPA [22] for drinking water. The concentrations of 2-nitrophenol range from 0.01 – 0.376 µg/L. The maximum value was recorded in sample B, while the lowest value was found in sample C. These values however were lower than the tolerance level of 0.5 µg/L and the maximum contaminant level of 1 µg/L for water intended for human consumption. This shows that the water is not polluted with respect to 2-nitrophenol. The range of values obtained in this report are comparable to the 0.028 – 0.117, and 0.013 – 0.079µg/L for wet and dry seasons respectively recorded by Zhou et al [4] for surface water of China River Basin.

The values obtained for 2,4-dimethylphenol in all samples were lower than ECC [9] and USEPA [22] standards. At site C, the value was below detection limit (0.0001 µg/L). The highest value (0.296 µg/L) was recorded at site A. The range of values for 2,4-dichlorophenol was 0.034 µg/L (Sample C) to 1.224 µg/L (sample B). The mean and the median were 0.342 and 0.146 µg/L respectively. These values are again lower than the [9] and [22] standards; an indication that the river is not polluted with respect to 2,4-dichlorphenol. The mean value obtained in this report is however, higher than the 0.0383 and 0.02584 µg/L obtained by Zhou et al [4] for surface water in China river basin.

4-chloro-3-methylphenol range in the river water was 0.070 – 1.136 µg/L. The mean value was 0.432 µg/L. The range of values for 2,4,6-trichlorophenol was from 0.012 µg/L recorded in sample E to 0.638 µg/L observed in sample B. The average value was 0.232 ± 0.25µg/L. The large standard deviation compared to the mean indicate large variability in concentrations among the sample's sites. Again, the range of value recorded in this report is higher than the 0.00462 – 0.0357 µg/L, and not detectable (ND) – 0.021 µg/L reported by Zhou et al [4] for wet and dry seasons respectively in the surface water of China river basin. The highest value for 2,4-dinitrophenol was 2.29 µg/L (sample B), while the lowest was 0.01 µg/L (sample A). The average value was

0.572 µg/L. Again, the large standard deviation (SD) is an indication of large variability in concentrations of the different locations. The contribution of 2,4-dinitrophenol the total eleven phenol was 0.96 percent.

The mean concentration of 4-nitrophenol was 0.972 µg/L drawn from a range of 0.046 (sample D) to 3.188 µg/L (sample E). The range of 2-methyl-4,6-dinitrophenol in the river water was 1.70 – 35.47 µg/L with a mean of 11.14 µg/L. Pentachlorophenol values ranged from 2.26 – 18.57 µg/L with mean value of 7.16 µg/L. This range is higher than the not detectable (ND) – 0.00734 µg/L, and ND – 0.00392 µg/L respectively reported for wet and dry seasons for surface water in China [4].

In general, the total for the eleven (11) phenols concentrations (Table 3) ranged from 41.09 – 116.57µg/L with a mean value of 59.29 ± 32.53 µg/L. The order of total phenols concentrations in the various locations was: B > E > C > D > A. Among the eleven phenols, the most abundant in the water was phenol (Ph), with an average concentration of 27.54 µg/L corresponding to 46.45% of the total phenols. This was followed by 2-methyl-4,6-dinitrophenol (2-M-4,6-DNP) and 2-chlorophenol (2-CP) with mean concentrations of 11.14 and 10.64 µg/L, representing 18.78 and 17.94% of the total phenols respectively. Pentachlorophenol (PCP) also constituted large portion (12.08%) of the total phenols in the water. The remaining 4.75% of total phenols is made up of 4-nitrophenol; 2,4-dinitrophenol; 4-chloro-3-methylphenol; 2,4-dichlorophenol; 2,4,6-trichlorophenol; 2-nitrophenol; and 2,4-dimethylphenol (listed in decreasing order of percent abundance in the water). There was no regular pattern of increase concentrations downstream or upstream with respects to these phenols, an indication that the contaminants were not from a point source along the river. Sofoniou et al [23], determined total phenols for rivers, lakes, and streams waters located in Northern Greece using spectrophotometric method after steam distillation, and reported a range of 4.0 - 12 µg/L. Ayeni [24] determined total phenols in surface water of Isebo river, south-western Nigeria using same method; found a range of 50 - 2110µg/L. Also, Medjor *et al* [25], using similar method reported total phenols content of the river water in Jalingo metropolis, Taraba state, Nigeria, to range from 306.7 – 407.9 µg/L. The range of values obtained in this report were lower than that of Ayeni [24] and Medjor *et al* [25], but were higher than that reported by Sofoniou *et al* [23]. The range of phenols obtained in this study however, is an indication of anthropogenic inputs which need to be checked.

Table 4: Coefficient of relations between the concentrations of the various forms of phenols in surface water.

	2-CP	2-NP	2,4-DMP	2,4-DCP	4-C-3-MP	2,4,6-TCP	2,4-DNP	4-NP	2-M-4,6-DNP	PCP
Ph	0.98**	0.86*	-0.12	0.96**	-0.15	0.88**	0.93**	0.30	0.91**	0.86*
2-CP		0.87*	-0.33	0.94**	-0.24	0.82*	0.93**	0.41	0.92**	0.86*
2-NP			-0.05	0.74*	-0.34	0.54	0.68	0.71	0.67	0.61
2,4-DMP				-0.23	0.30	-0.007	-0.37	-0.31	-0.41	-0.39
2,4-DCP					0.08	0.87*	0.99**	0.21	0.98**	0.97**
4-C-3-MP						-0.05	0.06	-0.35	0.09	0.24
2,4,6-TCP							0.87*	-0.18	0.83*	0.78*
2,4-DNP								0.18	0.99**	0.98**
4-NP									0.21	0.18
2-M-4,6-DNP										0.99**

****significant at the %1 level, *significant at the %5 level.**

3.3. Correlation analysis

Pearson's correlation coefficients and P-values calculated for all possible variables' pairs (Table 4) shows that the concentrations of 2-NP; PCP were positively significantly correlated with phenol (Ph). Ph also was very positively significantly correlated with 2-CP; 2,4-DCP; 2,4,6-TCP; 2,4-DNP and 2-M-4,6-DNP. 2-Nitrophenol(2-NP); 2,4,6-TCP; and PCP showed significant correlation with 2-CP; while, 2,4-DCP; 2,4-DNP; and 2-M-4,6-DNP had very strong correlations with 2-CP. There were also strong correlations between 2-NP and 2,4-DCP; and 2,4-DCP and 2,4,6-TCP. Strong positive correlation was also noted for 2,4-DNP; 2-M-4,6-DNP and PCP with 2,4,6-TCP. Very significant correlations were also observed for the following pairs: 2,4-DCP and 2,4-DNP; 2,4-DCP and 2-M-4,6-DNP; 2,4-DCP and PCP; 2,4-DNP and 2-M-4,6-DNP; 2,4-DNP and PCP; as well as 2-M-4,6-DNP and PCP. The correlations between the various forms of phenols may indicate that they are either from the same source(s) or that the reaction of one form in water environment gives rise to

the other. Lack of correlations, means that the marked presence of one form in water body does not necessarily indicate the presence of the other.

IV. CONCLUSION

The determination of eleven priority phenols in Ughelli river waters reveals a total phenols range of 41.09 – 116.57µg/L. The order of phenolic abundance in the water was: Ph > 2-M-4,6-DNP > 2-CP > PCP > 4-NP > 2,4-DNP > 4-C-3-MP > 2,4-DCP > 2,4,6-TCP > 2-NP > 2,4-DMP. The major contributors to total phenols were phenol (Ph), 2-methyl-4,6-dinitrophenol (2-M-4,6-DNP), 2-chlorophenol (2-CP), and pentachlorophenol (PCP), representing 46.45, 18.78, 17.94, and 12.08 percent of total phenols respectively. The remaining phenols, sum up to only 4.75% of the total phenols. The presence of these phenols in water, and their capacities to accumulate in aquatic organisms represent potential health hazards that required attention.

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