

# CORRELATION ANALYSIS OF REGULATED AND EMERGING TRI-HALOMETHANE DISINFECTION BY-PRODUCTS AND PHYSICO-CHEMICAL PARAMETERS OF A COMMUNITY DRINKING WATER SUPPLY

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## ABSTRACT

Using the Pearson Correlation technique, this paper established significant linear relationship among pairs of analysed physico-chemical parameters with the determined regulated (regTHMsDBPs) and emerging (emergTHMsDBPs) Tri-halomethanes Disinfection By-Products in the drinking water produced by the Ahmadu Bello University water treatment plant. Using standard methods including the USEPA Method 551.1, selected physico-chemical parameters and the levels of the regulated and emerging tri-halomethanes were determined. These were subsequently subjected to statistical correlation analyses. Statistical correlation of pairs of the some of the chemical parameters with total mean tri-halomethanes, show that total mean regulated tri-halomethanes DBPs (TregTHMs) values significantly correlated positively with temperature, total dissolved solids (TDS), total organic carbon (TOC) and residual chlorine values, while significantly correlating negatively with pH. On the other hand, total mean emerging tri-halomethane DBPs (TemergTHMs) significantly correlated with nitrates concentration levels ( $F=0.62578^*$ ) while being highly significantly correlating with residual chlorine ( $F=5.2670^{**}$ ), total organic carbon ( $F=13.3882^{**}$ ), total dissolved solids ( $F=6.2695^{**}$ ) and pH ( $F=2.7783^{**}$ ) and total mean regulated THMs (TregTHMs) with  $F=25.4016^{**}$ . Similarly, the resulting grand total THM (GTTHMs) concentration levels (comprising of the regulated and the emerging THMs) showed highly significant correlation with the total regulated THMs (TregTHMs) ( $F=54.9578^{**}$ ) and with total emerging THMs (TemergTHM<sub>2</sub>) ( $F=289.2349^{**}$ ) but with pH ( $F=3.3757^{**}$ ) as the only physico-chemical parameter. These signify that the concentrations of both emerging and regulated THMs are both significantly affected by the proportions of the total dissolved solids, nitrates, total organic carbon and residual chlorine. The generation of these models has established additional frontier to manipulate the concentrations of DBPs in treated drinking water by controlling independent physico-chemical variables in the drinking water treatment process. These predictive models could be safely used to predict the levels of these DBPs even though additional testing and analyses can enhance the confidence in such recommendation and extrapolation.

**Keywords:** Regulated and emerging Tri-halomethanes Disinfection By-Products, Pearson's Correlation, drinking water, carcinogenicity

## INTRODUCTION

Trihalomethanes (THM), i.e., chloroform ( $\text{CHCl}_3$ ), bromodichloromethane ( $\text{CHBrCl}_2$ ), chlorodibromomethane ( $\text{CHClBr}_2$ ), and bromoform ( $\text{CHBr}_3$ ) are major organic by-products of drinking water chlorination, resulting from the reaction of chlorine with natural organic material and bromine in source waters (Uden and Miller, 1983; Coleman *et al.*, 1984). The discovery of these by-products in drinking water has raised questions about their health hazards (Jolley *et al.*, 1990; IARC, 1991).

Trihalomethanes were the first new drinking water regulation EPA issued after passage of the 1974 Safe Drinking Water Act. The agency had the responsibility to produce all of the supporting information, and in quite considerable detail, and use that information, be it toxicology, analytical chemistry, occurrence, treatment technology, costs, economic impact, to craft its regulation (EPA, 1974).

Among the many tests undertaken in statistical analyses to assess the relationship between variable are the correlation test and regression analysis. While correlation determines the possibility and the degree to which pairs of analysed parameters are related, regression determines if any significant linear relationship exist. Usually, correlation among pairs is either significantly positive or significantly negative relationships in addition to insignificant relationships. Positive correlation arise when large values of one parameter in a pair are associated with large values of the other, while negative correlation occur when large values of one parameter are associated with small values of another. While insignificant correlations show no significant changes in a parameter in a pair as the other member of the pair in parameters changes. These statistical tools can be used to determine the relationships among parameters and undertake a trend

Shaibu-Imodagbe *et al.*, (2015); Correlation analysis of regulated and emerging Tri-halomethane disinfection by-products and physico-chemical parameters of a community drinking water supply analyses among these parameters in an experimental setup.

The studies reported here compared linear relationship among pairs of analysed physico-chemical parameters with the determined regulated (regTHMsDBPs) and emerging (emregTHMsDBPs) Tri-halomethanes Disinfection By-Products in the drinking water produced by the Ahmadu Bello University water treatment plant.

## MATERIALS AND METHODS

Materials and methods used in this study are in accordance with the standard methods (APHA, 1998) especially the USEPA Method 551.1 (1995) as reported in Shaibu-Imodagbe, (2011), Shaibu-Imodagbe *et al.*, (2013, 2014). The details of these are hereby presented.

### Materials

Materials used in the study include Sixty millilitres (60-mL) screw cap glass vials equipped with cap fitted with PTFE septa which were washed thoroughly and dried ready for sampling, Micro syringes of the capacities – 10  $\mu$ L, 50  $\mu$ L, 100  $\mu$ L and 1000  $\mu$ L, Glass Pipettes of 10 mL size, Pasteur pipette of about 23cm length to extract solvent and Top loading balance. Others include ACS Grade ammonium chloride de-chlorinating agent, ACS Grade sodium sulphate muffle furnace at 400 °C for a minimum of 30 minutes, Phosphate buffer prepared by mixing 1% ACS Grade sodium phosphate dibasic and 99% ACS Grade potassium phosphate monobasic salts, High purity grade MTBE and Agilent Gas Chromatograph (GC) Model 19091 – 413 calibrated with commercial standards for disinfection by-products supplied by Ultra Scientific Analytical Solutions, North Kingston, RI 02852-7723, USA with a Certificate of Quality Assurance. All the reagents used were of Analar grade.

### Sampling

Two hundred and fifty two (252) samples were collected in duplicates longitudinally along the treatment and distribution system of the Ahmadu Bello University Drinking Water Treatment Plant (ABUDWTP). These samples were taken from the source (raw) water (Kubanni reservoir), the sedimentation tanks, after chlorination, at the booster station and at residential house level (Area E Quarters) (Fig 1). Samples for tri-halomethanes (THMs) analyses were taken in 60mL glass vials with weighed samples of ammonium chloride used as de-chlorinating agent. Care was taken to avoid overtopping or spilling of sampled water from the vials. The samples were subsequently stored at temperature of about 4 °C or less in ice filled jugs in accordance with USEPA method 551.1 (USEPA, 1995).

### Methodology

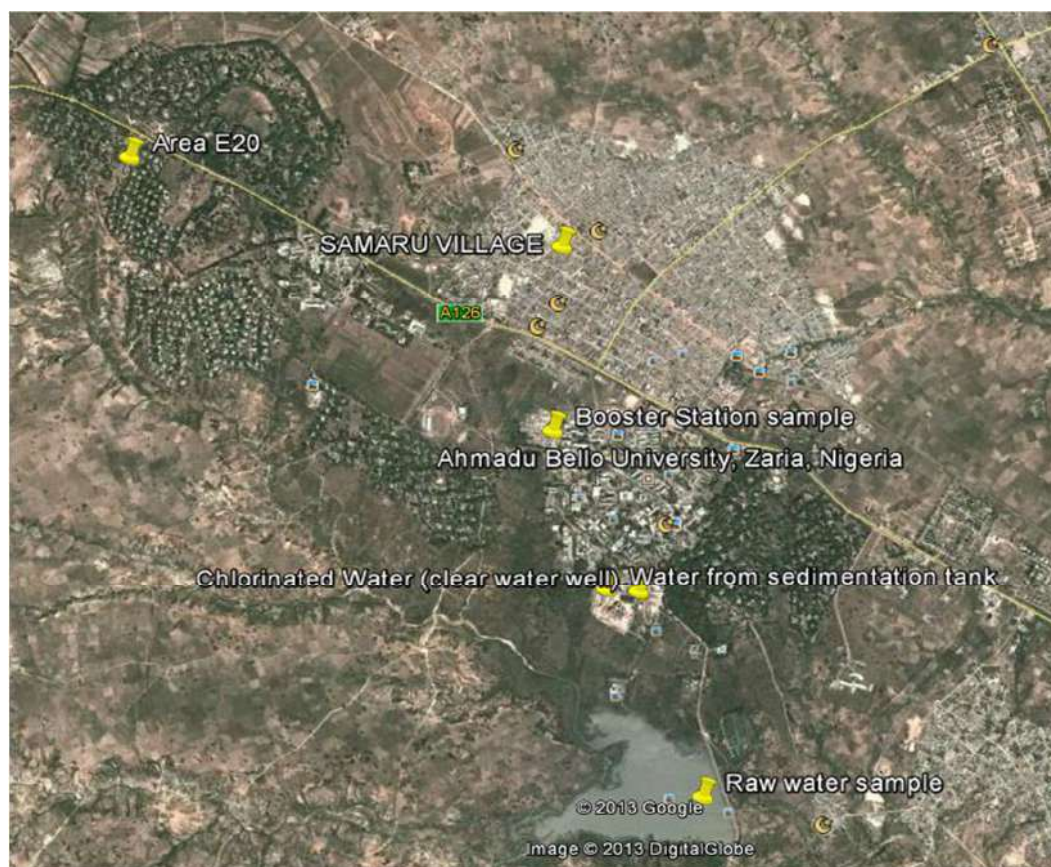
Analyses of the water samples for the THMs were in accordance with standard methods outlined in USEPA method 551.1 using Agilent Gas Chromatograph.

## RESULTS AND DISCUSSIONS

The results of the physico-chemical parameters as determined are presented in Tables 1 and 2 while the correlation analyses are presented in Table 3. Tables 4 show the statistical results for regression analyses. Over the three years period, the sampled water mean temperature values as obtained were relatively consistent among the five weather conditions over the periods (Table 1). The months of June and July when the rains are beginning to set in the study area recorded the highest water mean temperatures averaging 22.8 °C in 2008, 24.1 °C in 2009 and 23 °C in 2010. On the other hand the peak harmattan and beginning of the hot season (January and February) recorded the least mean water temperatures of 12.3 °C, 15.5 °C and 15.8 °C for 2008, 2009 and 2010 respectively. These values are reflections of mean daily temperature values of the study area during the period.

Other results of analysed physical parameters are presented in Table 2. From this table, the measured pH values of the treated and distributed water ranged between weakly alkaline with pH 7.80 $\pm$ 0.98 after chlorination and weakly acid with pH 6.70 $\pm$ 0.51 at household level. These values are within international acceptable limits for drinking water of slightly acidic to slightly alkaline range of pH values.

The increase in acidity towards house level could be due to the intermittent distribution of the produced drinking water. This practice favours biofilms generation and their subsequent decomposition creating intermediate products (Jakubovics, 1998; LeChevallier, 2000) with tendency to acidify the medium (Shaibu-Imodagbe, 2011). Conductivity values of the treated water were 131.00 $\pm$ 16.21  $\mu$ S/cm @ 25 °C in samples after chlorination and 102.00 $\pm$ 27.30  $\mu$ S/cm @ 25 °C. These values are considerably lower than the maximum permitted level of 1000  $\mu$ S/cm @ 25 °C. Highest values were obtained after chlorination in the treatment stage probably due to the calcium hypochlorite used as a disinfectant and the peak content of chlorides at this stage of treatment. The treated water also had higher concentrations of chlorides (12.65 $\pm$ 4.06 mg/L) than the distributed water at household level (0.5 $\pm$ 0.087 mg/L). The variation in chlorides content may also not be unconnected with the disinfectant used at the treatment stage. This is despite the initial high values in the raw water (12.30 $\pm$ 3.59 mg/L) which had been precipitated at the sedimentation tanks.



11°09'12.54" N 7°39'08.97" E

Fig 1: Google Map Showing the Sampling points for the Assessment of THM - DBPs in Ahmadu Bello University Drinking Water Supply

**Table 1: Sampled Water Temperature Variations during the Sampling Periods**

Year	Jan-Feb (Peak Harmattan and Beginning of Hot Season)	March - May (Mid Hot Season)	June July (Onset of Rains)	August - Sept (Peak Rainy Season)	Nov. - Dec. (Beginning of Harmattan)
2008 (°C)	12.3	21.8	22.8	22.8	18.1
2009 (°C)	15.5	21.8	24.1	23.3	16.7
2010 (°C)	15.8	22.6	23	22.8	18.2

**Table 2: Mean Concentration Levels of Physico-chemical Parameters as Measured in the Water Samples from the Treatment and Distribution Systems of the ABU Waterworks.**

	Raw Water	Water after Sedimentation	Water after Chlorination	Booster Station Water	Household water
pH	7.8 ±1.34	7.60±1.00	7.80±0.98	8.00±1.01	6.70±0.51
Conductivity (µS/cm @ 25 °C)	83.52±14.75	102.50±25.60	131.00±16.24	121.00± 30.81	102.00±27.30
Chlorides (mg/L)	12.30±3.59	0.80±0.16	12.65±4.06	0.50±0.11	0.50±0.087
Total Dissolved Solids TDS (mg/L)	50.50±10.97	50.50±9.96	51.50±9.88	55.00±14.52	66.00±7.02
Total Organic Carbon TOC (mg/L)	6.57±0.92	4.30±1.73	2.30±0.31	2.40±0.31	2.30±0.28
Residual Chlorine (mg/L)	0.00	0.00	0.05±0.016	0.03±0.016	0.02±0.014
Nitrates (mg/L)	3.00±0.54	2.40±0.54	0.80±0.28	0.80±0.30	1.40±0.50
Sulphates (mg/L)	40.00±11.55	23.00±1.09	22.50±1.35	24.50±2.99	42.00±5.90

<b>Phosphates (mg/L)</b>	0.39±0.021	0.15±0.019	0.25±0.026	0.15±0.017	0.10±0.012
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The total dissolved solids (TDS) ranged between 50.50±10.97 mg/L in the raw water sample to 66.00±7.02 mg/L in the treated household water sample. The higher TDS values were recorded during the distribution of the treated water than during the treatment stage. This may initially be due to the use of flocculating and liming agents during the treatment stage; while the upsurge in the TDS concentration levels could be due to the products of the mineralization of the intermediate products of decomposed biofilms in the distribution system. The proportion of total organic carbon in the raw water (6.57±0.92 mg/L) and the treated water (2.30±0.31 mg/L) and distributed water to household level (2.30±0.28 mg/L) exceeded the maximum total organic carbon levels of 4 mg/L for source water and 2 mg/L for treated water specified in the Disinfectants and Disinfection By-Products Rule of the USEPA (Anon1, 2001). This may have implications for tri-halomethane formation during the drinking water treatment and distribution. The mean concentrations of residual chlorine in the treatment and distribution system were found to vary from 0.05±0.016 mg/L in the samples after chlorination to 0.02±0.014 mg/L at household level. These low mean concentrations have implications for the safe delivery of wholesome and potable drinking water to the consuming community as these may indicate limited ability to maintain secondary disinfection in the distribution system. The mean nitrate concentrations decreased longitudinally along the treatment process from 3.00±0.54 mg/L in the raw water to 0.80±0.28 mg/L in the sample after chlorination but increased slightly to 1.40±0.5 mg/L at house level probably due to the biofilm growth, decomposition and mineralization as a result of intermittent water distribution. These values are considerably lower than the maximum contaminant limit of 50 mg/L allowed in national and international standards because of the risk of methaemoglobinanaemia. The concentrations of sulphates were found to decrease during the treatment process from 40.00±11.55 mg/L to 22.50±1.35 mg/L after chlorination but gradually increased to 42.00±5.90 mg/L at household level. This situation is most probably accounted for by the growth, decomposition and mineralization of biofilms generated in the distribution system due to intermittent water distribution. Despite this, these values are still within permissible limits under the national and international standards which range from 100 mg/L to 500 mg/L. The mean phosphate concentrations in the treatment and distribution stages progressively decreased from the raw water with 0.39±0.021 mg/L to house level water with 0.10±0.012 mg/L. This trend may be connected to the use of phosphates as essential nutrients for the

growth and development of biofilms along the distribution system.

Table 3 shows results of the Pearson's correlations coefficients among the pairs of parameters considered. Some pairs of the physico-chemical parameters correlated significantly among these physico-chemical parameters such as pairs like pH with TDS, TOC and residual chlorine among others. Statistical correlation of pairs with some of the chemical parameters with total mean tri-halomethanes, show that total mean regulated tri-halomethanes DBPs (TTHM<sub>4</sub>) values significantly correlated positively with temperature, total dissolved solids (TDS), total organic carbon (TOC) and residual chlorine values, while significantly correlating negatively with pH (Shaibu-Imodagbe, *et al.* 2013). On the other hand, total mean emerging tri-halomethane DBPs (TemergTHMs) significantly correlated with nitrates concentration levels (F=0.62578\*) while being highly significantly correlating with residual chlorine (F=5.2670\*\*), total organic carbon (F=13.3882\*\*), total dissolved solids (F=6.2695\*\*) and pH (F=2.7783\*\*) and total mean regulated THMs (TregTHMs) with F=25.4016\*\*. Similarly, the resulting grand total THM (GTTHMs) concentration levels (comprising of the regulated and the emerging THMs) showed highly significant correlation with the total regulated THMs (TregTHM<sub>4</sub>) (F=54.9578\*\*) and with total emerging THMs (TemergTHM<sub>2</sub>)(F=289.2349\*\*) but with pH (F=3.3757\*\*) as the only physico-chemical parameter. These signify that the concentrations of both emerging and regulated THMs are both significantly affected by the proportions of the total dissolved solids, nitrates, total organic carbon and residual chlorine. Also, this trend indicates that the proportions of both regulated and emerging THMs are significantly affecting each other. From the point of view of the grand total THMs, this showed highly significant positive correlation with only pH (F=3.3757\*\*) among the physical parameters. This indicates that large values of the total mean emerging and regulated tri-halomethanes-DBPs are associated in this study with large values of temperature, TDS, TOC and residual chlorine and nitrate levels in some instance while large values of the grand total mean tri-halomethanes (GTTHMs) are associated with low values of pH (high acidic condition) as had been documented in literature, (WHO, 2008). Also, the only significant correlation between pH and the resulting mean total of the regulated and emerging THMs could indicate that the degree of acidity is the major physico-chemical parameter determining the formation and proportions of the emerging THMs formed.

Table 3: Results of Correlation Analyses of the Determined Parameters

TEMP	SULPHATES	pH	TDS	TOC	RES CHLORINE	CONDUCTIVITY	NITRATES	PHOSPHATES	CHLORIDES	TRegTHMs	TEmergTHMs	GTTHMs
TEMP	0.043647*											
SULPHATES												
pH	0.3684445	1										
TDS	-0.4551253*	-0.87124**	1									
TOC	-0.422491*	-0.4073957*	0.977227**	1								
RES CHLORINE	-0.4064082	-0.61939**	0.92446**	0.906826**	1							
CONDUCTIVITY	-0.02891	0.763764**	-0.62247**	-0.5942*	-0.7825634**	1						
NITRATES	-0.18817	0.7563599**	-0.72195**	-0.67417**	-0.847447**	-0.85174**	1					
PHOSPHATES	-0.12085	-0.4034518*	0.380422	0.382191	0.68465334**	-0.8792578**	-0.8300569**	1				
CHLORIDES	-0.04178	0.9781309**	-0.53889*	-0.47822*	-0.5474653*	0.87099692**	0.87050963**	-0.558	1			
TRegTHMs	0.427367*	0.1308645	0.755564**	0.826595**	0.63084925**	-0.0762414	-0.1677677	-0.00433	0.087925	1		
TEmergTHMs	0.302203	-0.02064	0.822409**	0.903848**	0.798191**	-0.35305	-0.41544*	0.311944	-0.10102	0.945713**	1	
GTTHMs	0.344784	0.026352	0.811256**	0.890409**	0.755422**	-0.27091	-0.34312	0.217016	-0.0433	0.973776**	0.994854**	1

Table 4: Regression Characteristics of Tri-halomethanes and Physico-Chemical Parameters of sampled drinking waters

Variables		Regression Coefficient	ANOVA F values	Significant F	Regression Model	
		(R <sup>2</sup> )				
pH	y	TEmergTHMs-DBPs	0.4808	2.7783**	0.1941	$y = 2.00E - 04x + 0.5864$
		GTTHMs-DBPs	0.5295	3.3757	0.1635	$y = 2.00E - 04x + 0.6174$
Total Dissolved Solids (TDS)		TEmergTHMs-DBPs	0.6764	6.2695**	0.0874	$y = 2.00E - 05x + 0.5845$
		GTTHMs-DBPs	0.6581	5.7753**	0.0956	$y = 2.00E - 05x + 0.6147$
Total Organic Carbon (TOC)		TEmergTHMs-DBPs	0.8169	13.3882**	0.0353	$y = 2.00E - 04x + 0.5854$
		GTTHMs-DBPs	0.7928	11.4805**	0.0428	$y = 3.00E - 04x + 0.6146$
Residual Chlorine		TEmergTHMs-DBPs	0.6371	5.2670**	0.1055	$y = 2.4E - 03x + 0.5851$
		GTTHMs-DBPs	0.5707	3.9875**	0.1398	$y = 3.30E - 03x + 0.6155$
Nitrates		TEmergTHMs-DBPs	0.1726	0.6258*	0.4867	$y = 2.00E - 04x + 0.5851$
		GTTHMs-DBPs	0.1177	0.4003	0.5719	$y = 2.00E - 04x + 6162$
TemergTHMs-DBPs		TRegTHMs-DBPs	0.8944	25.4016**	0.0151	$y = 4.21E - 01x + 0.2161$
		GTTHMs-DBPs	0.9897	289.2349**	0.0004	$y = 1.42E + 00x - 0.2151$
TRegTHMs-DBPs		GTTHMs-DBPs	0.9482	54.9578**	0.0051	$y = 3.12E + 00x + 0.5206$

### Relationship Assessment

Further relationship assessment was undertaken using regression analysis to determine the rate of change of the determined concentration levels of the regulated and emerging tri-halomethanes disinfection by-products as a function of the concentration levels of assessed physico-chemical parameters. Among these paired assessments, the rate of change of the mean regulated total tri-halomethanes were found to vary significantly with changes in temperature, pH, total dissolved solids, total organic carbon, residual chlorine and nitrates. These, generated respective models by which the level of tri-halomethanes can be predicted with some degree of certainty based on fixed or predetermined level of these physico-chemical parameters. These characteristics are presented in Table 4

Table 4 also show that average values of six of the determined physico-chemical parameters create statistically significant conditional expectation in the average values of total mean tri-halomethanes for any fixed value of these physico-chemical parameters (Shaibu-Imodagbe *et al.*, 2013). Among these parameters are temperature, pH, total organic carbon, and residual chlorine already identified in literature as factors affecting concentration levels of tri-halomethanes in drinking water. In addition, this study increased the frontier of categories of these parameters to include total dissolved solids (TDS), and nitrates. It is noteworthy that seasonal influence was found insignificant despite temperature effect. This is likely due to the fact that only temperature is the influencing factor across seasons influencing the levels of the total mean tri-halomethanes.

It also determined predictive models showing the dependent relationship between the total mean tri-halomethanes and these physico-chemical parameters. Based on these models, it is possible to predict the concentration of the total mean tri-halomethanes with any predetermined concentration of the independent physico-chemical parameters in the sampled waters.

However, the means of the total THM values were found to be higher in all the treatment segments and finished water (in the Booster station and Household) than the Nigerian standard maximum permissible limit of the compounds (0.001 mg/L) SON, (2007). In addition, this study identified additional tri-halomethanes disinfection by-products (emerging THM-DBPs) in the treated drinking water which are known for their suspected and recognised carcinogenicity and genotoxicity. They are also known to cause narcosis, nausea, dizziness, headache, damage to the liver and kidney damage as well as central nervous breakdown. They also cause reproductive disruptions and irritations on the skin, the mucous membrane, the eyes and the respiratory tract (WHO, 2008). The proportions of these emerging THM-DBPs were found to account for over 90% of the

concentrations of the total tri-halomethanes determined at each stages of the treatment and distribution system where they were observed. This adds to the worry in addition to the health impacts of THMs in our drinking water supply. Statistically, the range of measured total mean regulated tri-halomethanes measured as TTHM<sub>4</sub> showed significant linear regression with temperature, pH, total dissolved solids, total organic carbon, nitrates and the residual chlorine of the sampled waters.

In this study, predictive models were found for most of the regulated and emerging THMs and the Totals of the THMs and their concentrations. This opens up additional avenue or alternative to manipulating the concentrations of the THMs in our drinking water supply. This additional alternative requires the control or manipulation of some of independent physico-chemical properties of the water under treatment to restrict the levels of THM DBPs in drinking water. This alternative may hold the key to controlling the levels of these DBPs in drinking water supply in addition to the control of DBPs precursors like dissolved organic matter through physically controlling the wastewater discharges from Samaru Village and effluent from waste dump behind ICSA Ramat hostels. This is geared towards minimizing and controlling the concentrations of these DBPs in the treated water. These generated predictive models which could be safely interpolated to predict the levels of the regulated and emerging tri-halomethanes disinfection by-products at any pre-determined conditions of these independent physico-chemical variables of temperature, pH, total dissolved solids, total organic carbon, nitrates and residual chlorine of the Ahmadu Bello University treated drinking water during the sampling period. However, care must be taken in extrapolating the results of these models to the treated drinking water outside the sampling period and indeed to other drinking water supplies unless backed with additional sampling and assessment.

### CONCLUSION

This study found Ahmadu Bello University treated drinking water to be USEPA and EU compliant with respect to the regulated THMs since the total mean tri-halomethane (THM) values were lower than the MCL (maximum permissible levels) for the TTHMs stipulated by both USEPA and EU (and WHO) standards of 0.08mg/L and 0.1 mg/L respectively.

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Shaibu-Imodagbe et al., (2015); Correlation analysis of regulated and emerging Tri-halomethane disinfection by-products and physico-chemical parameters of a community drinking water supply

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