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Quantification of antimony, iron and other trace elements in PET bottled water under typical use in Kano-Nigeria

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Abstract

Polyethylene terephthalate (PET) bottles are synthesized from ethylene glycol and dimethyl terephthalate using antimony as a catalyst. Antimony is a regulated drinking water contaminant and a non-essential element for plants and animals. The study investigated the release of Sb, Cr, Co, Cd, Fe and Pb in PET bottled water marketed in Kano-north west Nigeria, using MP-AES analytical method. Also the antimony in PET containers and physical properties of water were determined. MP-AES revealed 95±3.30 - 160±7.35 mg/kg antimony in PET containers, the physical properties of water measured were temperature (33 °C - 43°C), pH (7.5 – 8.0) and E. conductivity (0.02 – 0.19 mS/cm). The concentration of antimony released into PET bottled water ranged between below detection limit (0.017) to 50 μg/L, the average concentration of Fe, Co and Cr in PET bottled water were 0.137±0.08 mg/L, 0.005±0.020 mg/L and 0.025±0.007 mg/L respectively. The limit of detection (LOD) and quantification (LOQ) for the elements were found to be: Sb = 0.017 and 0.058 μg/L, Cd = 0.0054 and 0.0179 μg/L and Co = 0.0081 and 0.027 μg/L. Also the percentage recoveries for the method at 0.5 and 1.0 mg/L were found to be: Sb 102.33 ±3.05 % and 111.66 ± 3.05 %, Cd 108.66 ± 7.57 % and 106 ± 8.18 % and Co 122.66 ± 3.06 % and 123 ± 10.44 %. The concentration of antimony released in 29 % of the samples exceeded the maximum acceptable limit of 20 μg/L recommended by World Health Organization. Hence, the study suggests that PET bottled water should be kept away from sunlight and high air temperature

Keywords: Antimony, polyethylene terephthalate, PET bottled water, migration/released

1. INTRODUCTION

Global consumption of bottled water increases consistently for the last few decades, because of its accessibility, better quality and taste. In many developing nations, bottled water is used because of the quality of tap water and health concern (Atkins, 2012). The worldwide production of bottled water reached 391 billion liters in 2017 (Conway, 2020). In Nigeria bottled water constitutes of water in bottles and water in sachets, popularly known as "pure water". Sachet water dominates a large proportion of the population, as it is very cheap, available and affordable. Majority of bottled water in Nigeria are purified table water, only few are either spring or natural waters and have a best before dates of one year (Tukur et al. 2012). Polyethylene terephthalate bottles are well-known material for food contact application, it is used for carbonated soft drink, mineral water, edible oil and juices. Polyethylene terephthalate is a thermoplastic polymer produced by injection stretch blow molding, prepared from the monomers (ethylene glycol and dimethyl terephthalate or terephthalic acid) using antimony (mainly Sb_2O_3) as a catalyst, which catalyzes the poly condensation reaction of the intermediate pre-polymer to polymeric PET.

Regardless of the advantage of antimony in the manufacture of PET, it has a drawback of depositing elemental antimony in PET (Jose, 2014), Consequently, Sb is released into water contained in the PET bottle. Antimony is used as catalyst as well as flame retardant in the production of PET plastic at the concentration of 150-350 mg/kg (Carneado *et al.*, 2015; Hureiki and Mouneimne, 2012). Other metals are also used in the manufacture of PET for a variety of purposes such as catalyst, stabilizers and colorants, iron (Fe) and chromium (Cr) for green colours and cobalt (Co) for blue colours.The European Union (EU) and the United States Environmental Protection Agency (USEPA, 2009), treated antimony as a priority pollutant and regulated in drinking water. Recommending 5 or 6 μgL^{-1} as Maximum Concentration Levels (Westerhoff *et al.*, 2008).

Kano is the capital city of Kano state in North-West Nigeria, it is the commercial nerve center of Nigeria; it is the largest depot Centre of commodities including bottle water from all part of the country. The metropolitan local governments have the highest consumers of PET bottled water as indicated by a research, "PET bottles generation and disposal in Kano metropolis" (Abdulkarim *et al.,* 2012). With the hot climatic condition of Kano, unfavorable situations such as transportation and storage of bottled water in closed rooms/shops with no air conditioning and potential sunlight exposure, could provoke the release of contaminants into bottled water.

2. LITERATURE REVIEW

Different authors had reported the detection of Antimony in PET bottles, with various analytical methods. Among the earliest researches on antimony were that reported by Shotyk and Krachler (2007), analysis of 132 PET bottles from 28 countries and migration experiment on 3 bottles by storing pristine water at room temperature for 6 months using ICP-SF-MS analytical method. Tukur et al. (2012), reported a comprehensive study on antimony carried out in the UK, on PET bottle samples from Britain and Nigeria using ICP-MS. Another research reported by Fan et al. (2014) on 16 brands of PET bottles using Milli - Q water at different temperatures for 7 days with ICP-MS analytical method. Among the recent researches reported in the literature were that of Al-otoum et al (2017), analysis of antimony on 22 PET brands including foreign brands in Qatar using ICP-MS. Marcinkowska et al. (2017), analysis of 35 bottles from 19 brands from Polland using HPLC-ICP-MS. Others were that of Markwo et al. (2017), analysis of antimony on 6 popular brands of PET bottles from Accra region, of Ghana, and monitored the migration of antimony under various storage conditions over time. And that of Qiao et al (2018), 10 brands of PET bottles from China using ICP- MS.

The aim of this research is to determine the presence and quantity of Sb, Cr, Cd, Co, Fe and Pb in polyethylene terephthalate bottle containers using Microwave-assisted acid digestion of the polymer and the elements are measured simultaneously by Microwave Plasma-Atomic Emission Spectrometry. And monitor the release of these substances into PET bottled water, randomly selected within the metropolitan local governments of Kano state Nigeria.

3. MATERIALS AND METHOD

3.1 Reagents and Standards

Analytical grade reagents were used, concentrated nitric acid (65% analytical reagent), hydrochloric acid, perchloric acid and hydrogen peroxide for acid digestion of PET polymer and water samples. Milli -Q water (purified by synergy UV Milli- Q water purification system from Millipore). Antimony and a multi-element standards, concentration of 1000 mg/L ± 4 mg/L. Working standards were prepared from these stocks.

3.2 Samples and sampling

Polyethylene terephthalate (PET) containers

Ten different brands of commercially available PET containers used for bottling drinking water in Kano were obtained, weight, thickness and colour of each bottle was taken and recorded. We characterized the bottles and bottle cap materials with FTIR.

PET bottled water samples

Hundred samples of different PET bottled water were randomly purchased from wholesalers at Singer market and retail stores and street vendors within the eight Kano metropolitan local governments. The water samples were purchased in August 2019, they were all within their shelf life, from the manufacturing and expiry dates on the bottles, which ranged from January, 2019 to October, 2019. All bottled water samples purchased have registration numbers from National Agency for Food, Drug, Administration and Control (NAFDAC), which is the Nigerian regulatory body for such items. The bottled water samples were 0.50, 0.75 and 0.80 L volume capacity. Five water samples were obtained from different manufacturers before bottling, which serve as the control and analyzed together with the bottle water samples. Temperature, Conductivity and pH of water the samples were measured for the purpose of assessing the relationship with antimony leaching. The temperature of the water samples were taken on the spot during samples collection using Mercury bulb thermometer. Conductivity and pH were measured using portable pH/EC/TDS/Temp. meter HI-9813-6 (Hanna instruments), made in Romania. Three different samples of well water, Borehole water and rain water were also collected to compare the antimony content with bottle water.

Preparation of Samples

Optimization of Acid digestion procedures

The use of different acids with hydrogen peroxide was investigated both in an open vessel (hot plate) and closed system digester. The fragments of the same bottle was used throughout the optimization procedure, 0.2 g was loaded in microwave digestion tubes followed by the addition of 12mL of the acids in the ratio 5:1 as in Tukur *et al,* (2012) and Markwo *et al*, (2017) in the following order: HNO_3 and H_2O_2 10: 2mL, HNO_3 and $HClO_4$ 10:2mL and 4mL each of the triacids HNO_3 : $HClO_4$: H_2O_2, each combination in triplicates. The same combination was used for hot plate digestion for 2hrs at 230 °C to obtain a clear and yellowish color filtrates which was diluted to 100cm^3 with deionized water and kept for MP-AES analysis. The best combination was picked based on the results obtained and henceforth used for digestion of PET bottles.

PET bottled water evaporation and digestion

5 Liters of each sample was gradually evaporated to 200cm^3 on a hot plate and sand bath using large volume glass beakers. We digested the water samples by adding 5cm^3 concentrated HNO_3 acid under reflux at 105 °C for 4

hours to 25 cm^3, which was cooled, filtered and made up to 50cm^3 with deionized water. The same volume of deionized water was taken as blank and digested like the samples. The samples were transferred to amber glass bottles prior to MP-AES analysis.

Calibration curve/Recovery of the method

Six points standard curve was obtained by diluting the stock of 100 mg/L, earlier prepared from 1000 mg/L Antimony and a multi-element standard for chromium, cobalt, cadmium and lead (10 ml in 100). The calibration standard solutions of 2, 4, 6, 8 and 10 mg/L was prepared by appropriately diluting: 0.2, 0.4, 0.6, 0.8 and 1.0 mL of 100 mg/L stock with 10 mL distilled water respectively. To detect the performance of the method, the recovery was tested by spiking the ultra-pure water with a known concentration at 0.5 and 1.0 mg/L Sb and other metals.

4. RESULTS

All plastic bottle water samples characterized were found to be polyethylene terephthalate, based on their FTIR spectra, They are all bluish in color with the exception of PET 7; which is colorless. Their weight ranged from 20 – 26 grams and thickness of 0.18 - 0.24 mm, the bottles capacity were between 500 mL to 800 mL.

Optimization of Digestion Acids

Microwave assisted digestion system with acid combination of HNO_3/H_2O_2 in the ratio 5:1 was chosen as the best method, because it involves complete mineralization of the PET as maximum concentration for antimony and other elements were obtained and henceforth used for the digestion of PET bottles.

Recovery of the method

The result is presented in table 1, percentage recoveries for Sb and Cr were within the acceptable limits of 80 – 120 % as set in EPA Method (6010D). However, the percent recovery of Co for both concentrations were outside the range of acceptability, giving 122 and 123% at 0.5 and 1.0 mg/L respectively. This shows that the method may exaggerate Co concentrations below 1.0 mg/L (ppm).The % RSD were within the range of 2.7 – 8.4%.

The limit of detection and quantification

The detection capability of the MP-AES was calculated as three times the standard deviation of ten measurements of reagent blank, divided by the slope of the calibration curve for the limit of detection (LOD), the limit of quantification (LOQ) was calculated as 10 times the standard deviation, divided by the slope of the calibration curve. LOD and LOQ) were found to be: Sb = 0.017 and 0.058 µg/L, Cr = 0.0054 and 0.0179 µg/L and Co = 0.0081 and 0.027 µg/L.

Table 1: Recovery of the method (at 0.5 and 1.0 mg/L)

Element	Amount added (mg/L) n=3	Amount recovered mg/L)	% Recovery	% RSD
Sb	0.50 1.00	0.51 ± 0.02 1.12 ± 0.031	102.33 ±3.05 111.66 ± 3.05	3.00 2.70
Cr	0.50 1.00	0.54 ± 0.04 1.06 ± 0.82	108.66 ± 7.57 106 ± 8.18	6.9 7.70
Co	0.50 1.00	0.61 ±0.02 1.23 ± 0.10	122.66 ± 3.06 123 ± 10.44	2.50 8.40

Antimony and some trace elements in PET bottles

Total concentrations of Sb, Co, Cr, Cd, Pb and Fe in ten PET containers (PET-1 to PET-10) were shown in form of a bar chart in figure 1. The Sb concentration ranged from 95 ± 3.30 to 160 ± 7.35 mg/kg with the average and median value of 125 mg/kg. The concentrations of Co ranged from 10 ± 1.50 to 25 ± 2.4 mg/kg, with an average and median value of 17.5 mg/kg and 15 mg/kg respectively. Cr ranged from 5 ± 1.40 to 20 ± 2.20 mg/kg with the average concentration of 8.0 mg/kg and median of 5.00 mg/kg. The concentration of Fe cannot be estimated because of large contamination in the blanks and digestion acids, while Cd and Pb were below the detection limit. The concentrations of Sb varied among different brands of PET containers. The detection of Sb in plastic containers can be ascribed to its usage as a catalyst in the manufacturing process of PET plastics. Co and Cr were detected probably due to their usage as colorants and stabilizers. The three caps (white, blue and dark blue) analyzed with the PET containers did not contain any antimony, which is expected as their FTIR spectra were different from that PET bottles. Different technology used in the manufacturing of PET bottles, variable quality and different analytical approach by researchers are responsible for variation in Sb concentration among different brands.

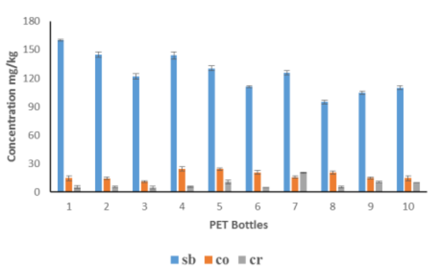

Figure 1: Concentration of Antimony, Cobalt and Chromium in ten different brands of PET bottles.

The results of Sb concentration in PET containers in this study is very much in line with 104 ± 8.81 to 166 ± 5.30 (Fan *et al*., 2014) and 123 to 146.45 mg/kg (Markwo *et al*., 2017) reported in the literature. And on the other hand, lower than some studies (Keresztes *et al*., 2009; Tukur *et al*., 2012, shotyk *et al*.,2006 and Jesus *et al*., 2016). They detected Sb in PET bottles from Hungarian bottles as 210 – 290 mg/kg (Keresztes *et al*., 2009), Tukur *et al*, (2012) reported Sb from Nigeria and Britain bottles within the range of 178 – 287 mg/kg and 195 - 242 mg/kg respectively. The highest level of antimony in PET bottles reported in the literature were 194-323 mg/kg (Jesus *et al.,* 2016) and 351-397 mg/kg (Shotyk *et al.,* 2006)

Antimony in randomly selected PET bottled water

The concentration of antimony in the water produced by the manufacturers before bottling, which was used as the control ranged from ND to 4.25 µg/L. The amount of antimony released in PET bottled water samples in Kano metropolitan ranged between below detection limit (0.017) to 50 µg/L with an average of 23.50 ± 2.50 µg/L (fig.2). The antimony level in 37% of the samples were below the Maximum Acceptable Concentration of 5-6 µg/L (USEPA, 2009) for antimony in drinking water. 34% have antimony concentrations from 6 to 20 µg/L, a value not exceeding WHO maximum level of 20 µg/L, while 29 % have antimony level from 21 to 50 µg/L, which exceeded the maximum contaminant levels. The average concentrations of antimony in well water, tap water and bore-hole water analyzed at three different times were 1.5 ± 0.04 µg/L, 2.6 ± 0.016 µg/L and 2.5 ± 0.012 µg/L respectively. None of the values exceeded the maximum concentration in drinking water and only 25 out of 100 samples of PET bottled water has antimony concentration within the limit of these natural waters which having values from not detected to 2.6 µg/L. The detection of antimony in PET bottled water is attributed to the earlier findings, the presence of antimony in empty PET containers (95 ± 3.30 to 160 ± 7.35 mg/kg) in this research, due to the use of antimony as a catalyst in the manufacturing of PET bottles, 150 to 350 mg/kg Sb was reported to be use in the industrial process. The high amount of antimony in some of the bottled water samples can be ascribed to primary exposure to tropical temperature in Kano before purchase.

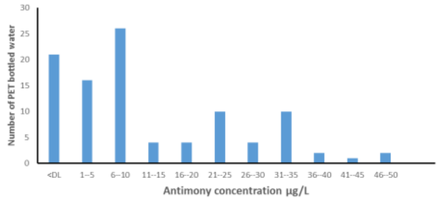

Figure 2: Range of Antimony concentration released into different samples of PET bottle water

The concentration of antimony released into PET bottled water in this study is compared with what was reported from the literature, though most of these studies were based on certain experimental conditions. Tukur *et al,* (2012) reported 0.03 µg/L to 6.61 µg/L Sb in 47 samples of freshly purchased British bottled water. Bach *et al.,* (2013), reported Sb concentration ranged from 3.5 – 10 µg/L after storing at 60-80°C for a seven days. Antimony concentration released at 2°C - 22°C for 7 days was reported to range from 11.70 – 54.10 ng/L and much higher values 1240 ng/L was released at 45°C (Reimann *et al.*, 2012). Similarly Shotyk and Krachler (2007), reported 8.90 – 2570 ng/L for analysis of 69 brands of PET bottled water, kept at room temperature for 6 months. These results could be consistent with Sb released by some samples in this research (from ND - 50 µg/L) and could differ significantly, because the bottle water samples we analyzed in this research were randomly selected, exposed to different unknown storage conditions by the vendors. Conditions such as air conditioned room, room temperature, under the shade, high air temperature, to bright sunlight exposure, for shorter or longer time duration were all possible for the weather conditions and commercial activities in Kano metropolitan.

Conductivity, pH and Temperature

The statistical results for the three parameters are presented in table 2, the temperature of the water were found to range from 33 °C – 43 °C, with an average of 37.5 °C. The average temperature of the water samples has exceeded the range agreed by the World Health Organization and Nigerian Standard for Drinking Water Quality. The pH values were found to range from 7.5 – 8.0 with an average of 7.69, forty percent of the samples analyzed have a pH of 7.5 and the remaining 60% have a pH range from 7.6 – 8.0. The pH of the water samples were within the acceptable limit of WHO and NSDWQ. The electrical conductivity range from 0.02 – 0.19 mS/cm, with the average value of 0.072 mS/cm, which is far below the maximum value of 1.00 mS/cm (WHO, 2011 and NSDWQ, 2007)

Table 2: Statistical Results of Temperature, PH and Electrical Conductivity

Physicochemical qualities	Minimum	Maximum	Mean	Standard deviation	WHO (2011)	NSDWQ (2007)
Temperature °C	33	43	37.5	2.73	25-30	25-30
pH	7.5	8.0	7.69	0.19	6.5-8.5	6.5-8-5
Electrical conductivity mS/cm	0.02	0.19	0.072	0.05	1.00	1.00

Effect of physicochemical properties on Sb release

Spearman non-parametric correlation was carried out between Sb in different brands of PET bottles and Sb released into the bottles, a strong positive correlation was indicated (r = 0.9417) which was statistically registered extremely significant (p = .0002 < 0.05 and 0.01) at 95 % confidence limit. The three parameters were also correlated with antimony released into the water, the results with pH indicated moderate positive correlations (r = 0.4987); but statistically registered insignificant (p = .1440 > 0.05).The correlation with temperature indicated no significant relationship (r = 0.5155 and p = .1334 > 0.05), E. conductivity indicated a moderate negative correlation with Spearman correlation (r = -0.4268) but the p value (p = .2182 > 0.05), suggested no significant relationship. The scatter plots are shown in figure 3.

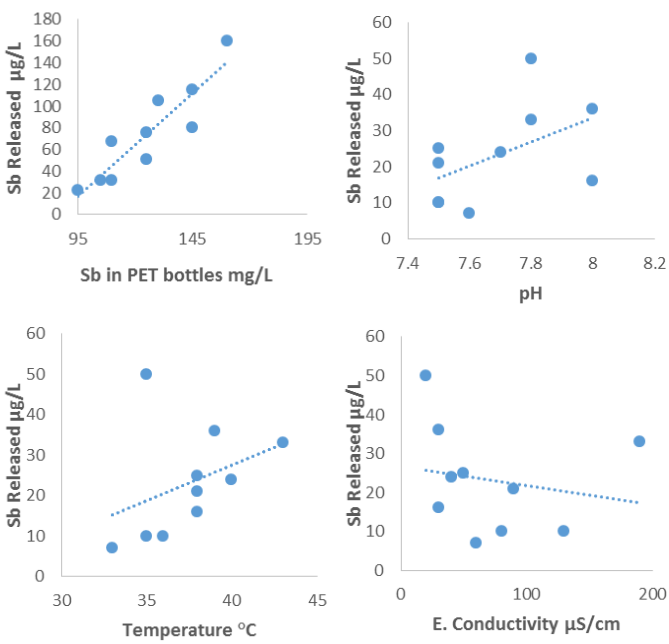

Figure 3: Scatter plots showing the relationship between Antimony released into PET bottled water with Sb in PET bottles and other physical properties of water (pH, Temperature and E. Conductivity).

5. CONCLUSION

The presence of antimony, cobalt and chromium in different brand of PET containers has been detected; MP-AES revealed 95±3.30 - 160±7.35 mg/kg antimony, 10±1.50 - 25±2.40 mg/kg cobalt and 5±1.40 - 20±2.20 mg/kg chromium. The leaching or released of antimony into bottle water has been detected within the range of 0.017 – 50 µg/L in randomly selected bottle water under typical use in Kano metropolitan, 29 % of the samples have antimony level of 21- 50 µg/L which exceeded the highest maximum contaminant level (MCL) of 20 µg/L by WHO. Statistical correlation indicated a strong positive correlation between the Sb released into bottled water and Sb in the PET bottle, and no significant relationship with temperature, pH and electrical conductivity. The antimony and other trace elements analyzed in the majority of bottled water were within the safe limit recommended for drinking water quality, 29 % of the PET bottle water samples analyzed in this study have exceeded the reference dose of 400 ng/kg/day recommended by USEPA.

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