COMPARATIVE STUDY OF LOCAL AND COMMERCIAL TABLE SALTS MARKETED IN WUKARI, NIGERIA

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Abstract: A preliminary study of Akwana mineral salt was conducted using LaMotte testing kit for water quality investigation, Fourier Transform Infrared (FTIR) for characterization of organic contamination and X-ray fluorescence technique (XRF) for quantification of metals and non-metals alike. The study showed that Akwana mineral salt and table salts processed from it contained nitrate, phosphate, iron and ammonia with concentration ranges from 0.4 to 1.0, 1.0 to 2.0, 0.5 to 12 and 0.5 mg/kg, respectively. However, the concentration of iron in Akwana mineral Salt (sample A) is very high and above the WHO permissible limit (1.0 mg/kg) as compared to Akwana Mineral Salt Brine, Uncle Palm and Mr. Chef (sample B, E and F). Akwana Mineral salt local Filtrate and finished Akwana table salt from the local factory in Akwana (Sample C and D) contained the least concentration of Iron 0.5 mg/kg each. The Phosphate concentration was 2 mg/kg for samples B and C while A, D, E and F which had 1 mg/kg. The FTIR results showed prominent peaks for Akwana local salts and the commercial table salts (Uncle Palm) when compared. The prominent peaks include, $O - H$; Phenol or Alcohol at 3700-3584, 3550-3200 cm $^{-1}$; O - H; Carboxylic at 1440-1411 cm^{-1}; $N - H$; Primary Amides at 3500-3250 cm $^{-1}$; $C = C$; Alkene at 1650-1610 cm $^{-1}$; $C - H$; Alkanes at 1440, 1466 cm^{-1}; $C - X$; Alkyl halides at 650-500 cm^{-1} and $C - O$; Carboxylic, ether, ester at 1150-1050 cm^{-1}. The XRF analysis showed presence of some impurities in the Akwana table salt and Uncle palm (commercial salt). Prominent impurities of concern included, Sulphur: 18.8746 mg/kg, Calcium: 4.019 mg/kg, Si: 1.7191 mg/kg and Aluminium: 2.3279 mg/kg. These impurities may be as a result of the addition of calcium aluminium silicate as an anticaking agent in edible salt and it also occurred naturally in soils and rocks.

Keywords: Akwana, mineral, trademarked salts, uncle palm

Introduction

Table salt (sodium chloride) has been with us from the inception of geologic time. The demand for it as a necessity for all living things in one form or another continues today. The body of an adult contain relatively appreciable quantities of salt, as we may infer from the taste of "blood, sweat, and tears" (Dalf, 1971; Johansson, 2008). Sodium (39.34%) and chlorine (60.66%) combine to form sodium chloride (*Zhang et al., 2013*). Sodium, a silver colored metal is unstable and reacts violently with water. Chlorine is a greenish-yellowcolored gas that could dangerous and lethal. Their combined state as a salt is essential to life (Lincoln, 2003). Sodium chloride is used as table salt. WHO recommends a reduction to <2 g/day sodium (5 g/day salt). The recommended maximum level of intake of 2 g/day sodium in adults should be adjusted downward based on the energy requirements of children relative to those of adults (WHO, 2012). It has wide industrial applications and other usage by humans. It is perhaps the only mineral in the world that is used virtually by every human being (Dennis, 2008). The human body depletes salt by sweat, excreted through urine, increased bowel mobility and diarrhea etc. The daily intake of the salts replenishes the essential minerals in humans. Our body primarily consists of fluids having minerals which act as electrolytes on cell membranes allowing transmission of impulses in muscle and nerve fibers and in this way cellular metabolism is controlled (Beard, 1990).

However, now that salt is so cheap we have forgotten that, up until 100 years ago, salt was difficult to obtain, and so it was a highly valued trade item to the point of being considered a form of currency by certain people (Johansson, 2008).

The order of historic development of salt recovery are solar evaporation of brines, quarrying of the solid salt, mining of solid salt, burning of salt-containing plants, evaporation of brine on burning wood, evaporation of brine in vessels, sublimation of sea−water ice or sodium chloride dihydrate in cold climates and recovery of co−product or by product salt from chemical reactions. Sodium Chloride is currently mass-produced by evaporation of seawater or brine from brine wells and salt lakes. Mining of rock salt is also a major source. China is the world's main supplier (Dennis, 2008). Sodium chloride can also be produced from saturated brine by multiple-effect process, saturated brine by open (Grainer) process while rock salt by mining and Sea water by solar evaporation (Davais and Berner, 2005).

Sodium chloride, (NaCl) has a molar mass of 58.44g/mol. It is odourless and off white in colour with a melting point of 801°C and a boiling point of 1,413^{o}C. It specific gravity is 36.79 J/K/Mol and density is 2.165 g/cm^3. The thermal conductivity (as a function of temperature) is at a maximum of 2.03 at 8 K (-265.15°C; -445.27 °F) and decreases to 0.069 at 314 K (41°C; 106 °F) and decreases with doping (Sirdeshmukh *et al.,* 2001; Ruth, 1995).

Uses of NaCl as table salt includes; it's inclusion as an essential constituent of our diet, as a preservative, e.g. in packing and curing of meat and fish. It is used in the industry as a starting material for the manufacture of hydrochloric acid, washing soda, caustic soda and many other sodium compounds. It is also used in the preparation of pottery glaze, soap and salting out. In addition, it is used in Medicine, Agriculture, and firefighting ((Hay and Kyser, 2001; McDonnell, 2003). Sodium chloride is very important during winter for de-icing of roadways in sub-freezing weather. This is the second major consumer of sodium chloride (Gisbert *et al.,* 2002).

Sodium Chloride as table salt is almost universally accessible, relatively cheap and often iodized (Mark, 2003).

This study was embarked upon to determine the chemical composition of Akwana mineral salt in comparison with two selected trademarked or commercial salt found in Wukari market.

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Materials and Methods

Study area

The study area covered selected salt mining area in Akwana district of Wukari Local Government area of Taraba State, Nigeria. Akwana lies between latitudes 7º 51' 0" North and longitudes 9º 14' 0" East and is 72 Km away from Wukari. Wukari is between longitudes 7º 51' 0" North and 9º 47' 0" East

Sampling

Samples used in this study include, Akwana mineral Salt, (sample A), Akwana Mineral Salt Brine (sample B), Akwana Mineral salt local Filtrate (sample C) and finished Akwana table salt from the local factory in Akwana (Sample D). Two trademarked salts (iodized) samples namely, Uncle Palm (Sample E) and Mr. Chef (Sample F) were purchased from Wukari market.

All samples were randomly collected from the mining site and local factory at Akwana and stored in plastic containers, labelled and preserved for processing.

Chemical analysis of salt samples

The determination of phosphate, nitrate, iron and ammonia was carried out using the LaMotte testab water investigation kit, 2010.

A 0.05 g of Nitrate salt tabletlabelled testab #1 was addedinto 10 ml of the analyte solution, the solution was shookfor four minutes and testab #2 was added and shook again, allowed to stand for five minutes. The presence of Nitrate was indicated by colour change. The colour changed was then compared with the LaMotte testab charts to know the concentration thereof. The procedure was repeated for Phosphate, Iron and Ammonia.

The XRF analysis

This was done following the procedure of Neuwirthová *et al*. (2012) and Heena *et al*. (2015). X-ray fluorescence spectrometer applies XRF technique to conduct fast and accurate analysis of complex composition of substances. Energy dispersive fluorescence spectrometer (XRFS) SPECTROXEPOS equipped with 50 Watt Pd X-ray tube was used to excite the samples. The target changer, with up to 8 polarization and secondary targets, offers many different excitation conditions ensuring optimum determination of all elements from sodium to uranium. Measurements were performed in helium atmosphere. The detector was a state-of-art silicon drift detector (SDD). A spectral resolution of less than 160 eV for Mn K-alpha was achieved. The maximum count rate was 120,000 cps. The analyzer can handle samples with diameters up to 32 and 40 mm. A voltage of 40kv and a current of 350 mA were applied to generate the X-ray needed to irradiate the samples in this study for a pre-set period of 100 seconds for each sample. The system detected elements in the salt samples, their concentrations were determined and expressed in percentage.

FTIR analysis

FTIR analysis identifies chemical bond, functional groups by their characteristic absorption of infrared radiation in vibrational modes. May be used in transmission mode, especially capable of identifying the chemical bonds of organic materials, detects and identifies organic contaminants or additives also determines bonds undergoing degradation reactions or changing with processing.

In transmission mode, area of analysis was 1 x 1 mm. The thickness are from sub-micrometer to a few millimeters. Sample materials are dispersed on KBr pellets. The sample lateral dimensions were at least 12in x 12in. The Specac Golden Gate MKII Diamond single-reflection ATR with KRS-5 lenses, the area analyzed was about 0.6 x 0.6 mm. The width of a sample were less than 108 mm or about 4¼ inches. The window used was rugged type IIIa diamond. Pressure was applied on the sample at a uniform setting of 80lbs by a sapphire anvil.

Variable angle specular reflectance allows the depth of analysis to be varied and can be useful for looking deeper into a surface. Resolution up to ½ cm^{-1}, though most spectra of solids or liquids are taken with 4 cm^{-1} resolution. Signal-to-noise ratio greater than 42000:1, temperature controlled DLATGS detector, 62° Michelson interferometer with retro-reflecting corner cube mirrors, KRS-5 interferometer and DTGS detector windows, KRS-5 lenses are thallium bromoiodide lenses and have a nearly flat response in the entire IR range from 400 to 4500 cm^{-1} range (Anderson, 2018)

Results and Discussion

Each of the salts processed from Akwana mineral salt samples (A, B, C, D) contained nitrate. The concentrations were 1.0, 0.4, 0.5 and 5 mg/Kg, respectively. Nitrate was not detected in the trademarked samples E and F (Table 1). The concentration of Nitrate in sample A, B, C and D were below the WHO permissible limit (45 mg/Kg) for consumption. The concentration of iron and phosphate were within the permissible limits 1.0 and 0.1 mg/kg, respectively. However, the concentration of iron in sample A (which is the raw mineral salt unprocessed) is far above WHO permissible limits (BIS, 2010). The presence of nitrate in Akwana salts could be due to fertilizer residues on farmlands, decomposition of organic matter which can be transported from one point to the other as reported by O'Shea (2002). The high concentration of Phosphate may be due the facts that it occurs in traces in many natural waters, application of inorganic fertilizers and often in appreciable amounts during periods of low biologic productivity (Leena, 2010).

Table 1: Concentration of nitrate, phosphate, iron and ammonia

Samples	Nitrate (mg/kg)	Phosphate (mg/kg)	Iron (mg/kg)	Ammonia (mg/kg)
A	5.0	1.0	12.0	0.5
B	1.0	2.0	1.0	0.5
C	0.5	2.0	0.5	0.5
D	0.4	1.0	0.5	0.5
E	ND	1.0	1.0	0.5
F	ND	1.0	1.0	0.5

However, the concentration of iron in sample A, B, C and D were 12, 1, 0.5 and 0.5 mg/kg while the trademarked samples (E and F) contained 1 mg/kg. The concentration of ammonia in Akwana salt samples and trademarked salt were the same, 0.5 mg/kg. The presence of ammonia could be from the accumulation of decomposed organic matter. Ammonia is present naturally in surface and wastewaters. Its concentration is generally low in ground waters because it adsorbs in soil particles and clays and is not leached readily from soils. It is produced largely by de-amination of organic nitrogen containing compounds and by hydrolysis of urea (Leena, 2010). A representative sample of Akwana table salt and one trademarked salt (uncle palm) was studied. In Akwana representative salt, peaks were observed at 3367.00 (O-H$_{alc}$), 2159.00 (N=N=N$_{azide}$), 1635.56 (N-H), 1411.00 (O-H$_{carboxylic}$), 1082.09 (C-O), 506.00 (C-X$_{halo\ cpd}$) and 390.35 cm^{-1} (Fig. 1). Corresponding peaks were noticed for the two samples at 3459.00, 3419.00, 3367.00, 1635.60, 1636.47, 1635.56 and 1440.60, 1466.81, 1411.00 cm^{-1}, 1143.00 and 1103.00 cm^{-1}.

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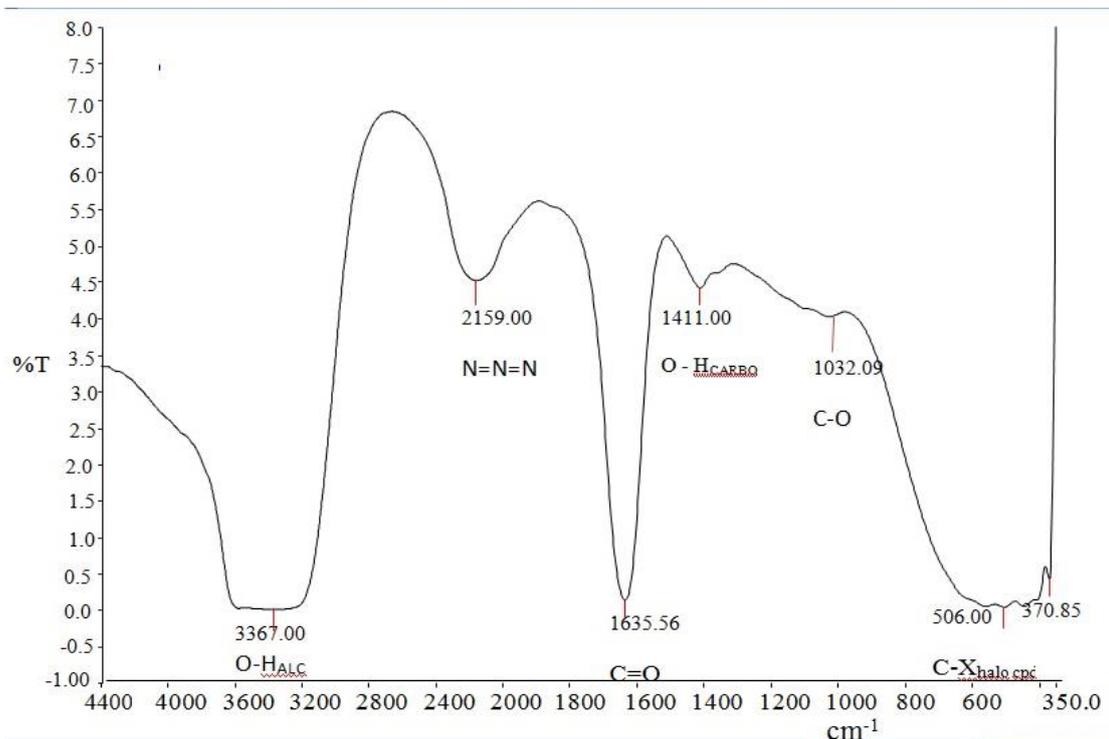
Fig. 1: FTIR spectra of representative sample of Akwana salt
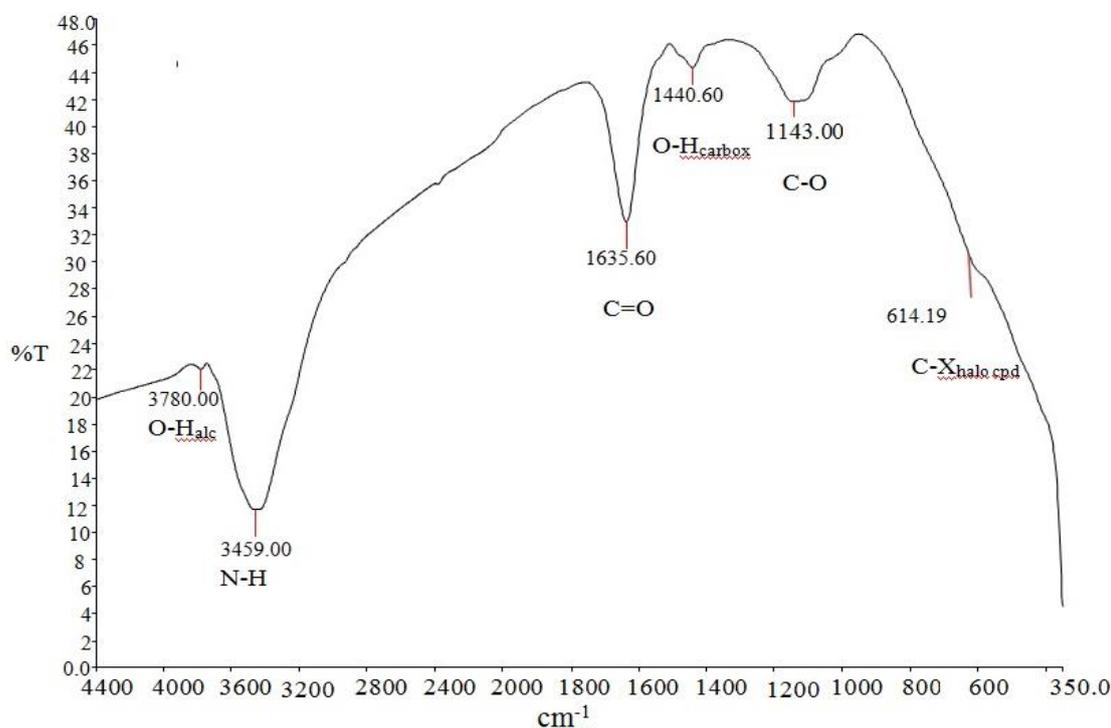
Fig. 2: Spectrum of uncle palm

Chloride and Iodide absorbance peaks were observed in all the samples at 506.00, 535.00 cm^{-1}, respectively. Absorbance peaks of carboxylic acid were also visible in all the samples.

Water had two absorbance peaks in our samples, a sharp peak around 1635 – 1636 cm^{-1} and a broad peak around 3459 – 3367 cm^{-1}. The hygroscopic nature of sodium chloride could be accountable for the water peaks.

The predominant peaks are: O–H; Phenol or Alcohol at 3700-3584, 3550-3200 cm^{-1}; O–H; Carboxylic at 1440-1411 cm^{-1}; N–H; Primary Amides at 3500-3250 cm^{-1}; C=C; Alkene at 1650-1610 cm^{-1}; C–H; Alkanes at 1440, 1466 cm^{-1}; C–X;

Alkyl halides at 650-500 cm^{-1} and C–O; Carboxylic, ether, ester at 1150-1050 cm^{-1}.

The FTIR spectrum (Fig. 2) of uncle Palm trademarked salt showed peaks at 3780.00 (O–H$_{alc}$), 3459.00 (N–H), 1635.60 (C=O), 1440.60 (O–H$_{carboxylic}$), 1143.00 (C–O) and 614.19 (C–X$_{halo cpd}$) cm^{-1} for. Peaks at 3419.00 (O–H$_{alc}$, N–H), 1636.47 (C=O), 1466.81 (C–H), 1108.00 (C–O) and 535.00 (C–X$_{halo cpd}$) cm^{-1}. However, Yalçina and Mutlu (2012) also obtained similar peaks (3419, 3459, 3420, 3446, 3434, 3468 and 1636) in their work. This peaks come from water in the structure of

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the sample. We estimated that 1032.09 and 1143.00 value comes from carbon dioxide in air as shown in Figs. 1 and 2.

The XRF analysis indicated that both the representative Akwana table salt sample and trademarked salt (uncle palm) contain some impurities (Table 2 and Figs. 3 – 4).

Table 2: Percentage elemental composition of Akwana salt and trademarked salt

Element	% Composition of Akwana salt	% Composition of Uncle Palm
Mg	0.0000	0.0000
Al$_2$	2.3279	2.5283
Si	1.7191	1.6252
P	0.8719	0.9274
S	18.8746	21.4095
K	0.3307	0.0000
Ca	4.0191	0.0939
Ti	0.0000	0.0000
V	0.0000	0.0000
Cr	0.0000	0.0000
Co	0.0000	0.0000
Mn	0.0000	0.0000
Fe	0.1283	0.1190
Ni	0.1718	0.0101
Cu	0.0211	0.0189
Zn	0.0471	0.04219
As	0.0000	0.0000
Pb	0.0938	0.0027
W	0.0109	0.0320
Au	0.0000	0.0000
Ag	0.0000	0.0000
Rb	0.0014	0.0015
Nb	0.0000	0.0000
Mo	0.3519	0.3129
Cd	0.0000	0.0000
Sn	0.4953	0.3129
Sb	0.5207	0.5014
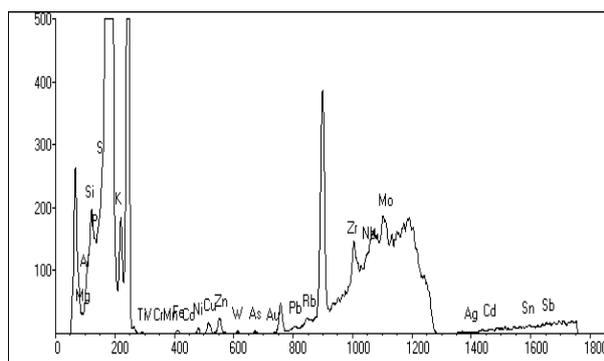
Fig. 3: XRF spectrum of representative Akwana salt
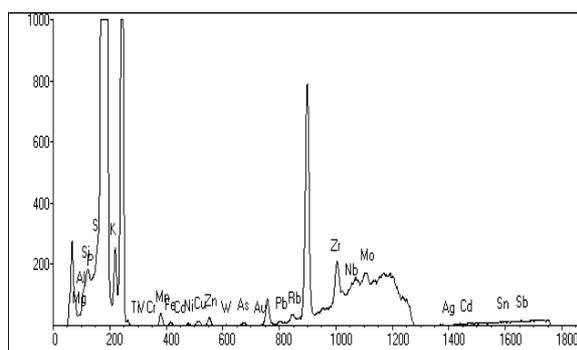
**Fig. 4: XRF Spectrum of uncle palm**

This work agreed with the findings of Amorin and Ferreira (2005), Steinhauser *et al.* (2006) and Peker *et al.* (2007) that, trace elements which include: Aluminum (Al), Arsenic (As), Cadmium (Cd), Cerium (Ce), Chlorine (Cl), Cobalt (Co), Chromium (Cr), Cesium (Cs), Copper (Cu), Iron (Fe), Manganese (Mn), Sodium (Na), Nickel (Ni), Lead (Pb), Rubidium (Rb), Antimony (Sb) and Zinc (Zn) are present in edible salts. Although, direct human use of rock salt has been discouraged by the health authorities, its existence in the markets indicates that this type of salt is still being used by the consumers, especially in developing countries (Soylak *et al.*, 2008; Cheraghali *et al.*, 2010).

The Sulphur content of the representative sample of Akwana table salt was 18.87 while in uncle palm it was 21.41% as the highest. Silicon was found in all the sample with values, 1.72 and 1.63, respectively.

The presence of Si is suspected to be from weathering of rocks and surface run off. A significant percent (4.0%) of Ca was found in the Akwana table salt representative compared to uncle palm's 0.09%. This could be attributed to the abundance of limestone in the study area. The samples contained Aluminium 2.53 and 2.34%, respectively. The presence of Aluminium may result from its relative abundance in the earth crust and weathering of rocks. Gholam *et al.* (2007) and Robert (2012) reported in their work that, Calcium aluminium silicate occurred naturally in soil and rocks and are mostly used as anticaking agent in edible salt during salt preparation. This accounted for the presence of Ca, Al and Si in both samples.

This research revealed that, the levels of Fe, Ni, Cu, Zn, Pb, W, Sb, Rb, Mo, P and Sn in both samples were less than the codex standard while S, Ca, Al are higher than the maximum allowance in food grade (CAC, 2001). Zinc and Cu are essential element in low concentrations and they regulated by the physiological mechanisms in most organisms. However, in an excessive concentration it is known to be a potential hazard that can endanger both animal and human health (Papagiannis *et al.*, 2004). The determined range of these element was 0.0189 – 0.0471% but were lower than the guideline value.

The percentage impurities of Potassium and Phosphorus in the representative sample was 0.04895%. The presence of Potassium and Phosphorus could be because of excessive application of inorganic fertilizers on farmlands.

Conclusion

This study revealed that Akwana mineral salt and the table salt processed from it contained nitrate, phosphate, iron, ammonia. The concentration of nitrate in Akwana table salt samples are below the WHO limit (45 mg/Kg) for consumable salt. However, the concentration of iron and phosphate are within the permissible limits (1.0 and 0.1 mg/Kg) for food (BIS, 2010). The concentration of iron in Akwana mineral salt (12 mg/Kg) is very high as compared to the trademarked salts (1.0 mg/Kg). The concentration of phosphate (2.0 mg/Kg) in Akwana table salts is greater than that of uncle palm.

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