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Chemical Composition and Physico-chemical Properties of Traditional Alkaline Salts (Lake Deposits and Plant-based Ash Salts) Sampled in Cameroon and Nigeria

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Abstract: Lake deposits and plant-based ash salts are food additives used in a variety of food preparations throughout the world. They exist in many colours and are found in market stalls in many countries. In the literature, they have been named traditional alkaline salts (TAS). Their colours (as identified by users) are their major quality determinant and are associated with their efficiency when used in food preparations. Up to now, no relationship has been established between their colours and their chemical composition and/or physico-chemical properties. Lake deposits (sampled in Cameroon and Nigeria), "white" plant-based ashes and evaporites of plant-based ash filtrates (sampled in Cameroon) found from the market have a buffer behaviour (pH of 10 and 11) while "green" and "black" plant-based ashes do not (pH = 12 to 14). The buffer behaviour is due to the simultaneous presence of carbonates and bicarbonates, combined with the absence of oxides, the pH of the resulting solutions was increased with increasing carbonates/bicarbonates ratio. The colours of evaporites of plant-based ash filtrates are similar (less than 6% of difference) in their chemical composition (sodium, potassium, carbonates and bicarbonates) and physico-chemical properties while the other TAS are not. "White" lake deposits have higher major ions (sodium, potassium, carbonates and bicarbonates) content than the "black" ones. There is always a toxicity risk (lead and cadmium) associated with the use of plant-based ash salts and sometimes "black" lake deposits while there is not a toxic risk associated with the use of the "white" colour of lake deposits. "Black" lake deposits sampled in Abuja, Nigeria have one and a half times more cadmium and lead than those sampled all over Cameroon when they contain these metals.

Keywords**:** lake deposits, plant-based ash salts, traditional alkaline salts, buffer solution, toxicity risk

1. Introduction

Common edible salt is a crystalline product containing at least 97% of sodium chloride (NaCl) and obtained from different sources: sea, natural brine and underground rock salt deposits. With a world production estimated at 26.1 million tons in 2004, it is probably the most consumed food additive [1-4]. It is used in food processing, both at

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household and industrial scales, to improve the taste and flavour of food preparations, and as a preservative. Out of common edible salt, different other specific salts are traditionally used in food preparations at the household level. They have various names in their areas of occurrence. Their use has mainly been reported in sub-Saharan African countries, Asia (India and Indonesia), Oceania (Papua New Guinea) and South America (Bolivia, Peru, Columbia, and Paraguay).

These salts are lake deposits [5-8], plant-based ashes and their filtrates [9-17], and evaporites of these filtrates [18-21]. They are mainly characterized by the alkalinity of their solutions, are used as technological auxiliaries for emulsification, soften the texture of foods [10-12, 14, 15, 18, 22-29], and as taste improvers [30]. Owing to their alkalinity [10, 14, 15, 17, 21, 24, 30], their long-time usage (up to 4,000 years before the common era) [13, 19, 31], and the fact that they seem to have been developed in areas where common NaCl salt was not available [13], they have been named traditional alkaline salts (TAS) by some authors [26]. Their chemical composition shows that they are a mixture of minerals, mainly of salts and oxides (CaO, K_2O, FeO, Fe_2O_3, MgO, and P_2O_5), and thus of anions (OH^-, CO_3^{2-}, HCO_3^-, SO_4^{2-}, S^{2-}, CN^-, F^-, Cl^- and $H_2\,PO_4^-$) and cations (Na^+, Ca^+, Fe^{2+}/Fe^{3+}, K^+, Mg^{2+}, Li^+, V^{x+}, B^{x+}, Mo^{x+}, Al^{3+}, Mn^{x+}), Ni^{2+}, Co^{3+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, Cu^{2+} and Cr^{3+}) (the letter x in superscript for some cations is used for multivalence cations that the ionic form analysed was not provided in the corresponding studies) [5-9, 12, 13, 17-19, 21, 32-34]. Each type of solid TAS exists in market stalls in a variety of colours ("black" and "white" for lake deposits; "black", "greyish", "white", "cream yellow" and "light brown" for plant-based ashes) [14, 15, 21, 25, 26]. For any given functionality, for the same type of TAS, users have a preference that is oriented toward a given colour. This preference is based on the fact that, for the same type of salt, the same quantity of its respective colours has variable effects when used at the household scale [25]. The differences observed in the usages of these salts show that they have different chemical compositions and physico-chemical properties. Since lake deposits occur naturally, the sampling locations, which might be associated with localities where they are sold, can justify the differences in their chemical composition and physico-chemical properties.

For plant-based salts, their difference in efficiency could be due to plants or plant parts used as well as conditions occurring during the burning process [14, 15, 25, 26]. Moreover, some studies involving the use of TAS (without any mention of their colour) have revealed the presence of heavy metals (lead and cadmium) in variable quantities [5-7, 13, 33] and such determinations are vital in order to restrict their use in food preparations. There is therefore a need to check whether or not there is a relationship between the colour of TAS and their chemical composition and physico-chemical properties.

The present study aims thus at determining the variability of the chemical composition and physico-chemical properties of lake deposits and plant-based salts used in Cameroon and Nigeria, in relationship with their colour.

2. Materials and methods

2.1 *Collection and preparation of TAS samples*

TAS (lake deposits, plant-based ashes and evaporites of plant-based ash filtrates) of different colours were sampled in some towns of Cameroon and Nigeria (Figure 1). In each town, sampling was done in markets where they were found. In each market, when a given TAS of a given colour was available in many stalls, some stalls were randomly selected for sampling and it is to the selected traders that these TAS were purchased and packaged. For lake deposits, since their chemical composition and physico-chemical properties can vary in a geographical area, they have been sampled in Nigeria and Cameroon. In Nigeria, they were collected from market stalls in Abuja, a town of Derived savannah agro-ecological area, while in Cameroon, sampling was done in markets of main towns of three agro-ecological areas: Sudano-Sahelian, humid forests and western highlands (Figure 1). For plant-based salts (plant-based ashes and evaporites of plant-based ash filtrates), they have been sampled in Cameroon whenever found in market stalls. Sampled plant-based TAS where generally sold by manufacturers or by sellers who have bought them from the manufacturers. Altogether, 27 samples of lake deposits, 3 samples of plant-based ashes, and 4 samples of evaporites of plant-based ash filtrates were obtained. The considered colour in this study is the one used by users. Regardless of the locality where sampling was done, each sample of TAS was obtained from a mixture of samples of each colour of a given type, obtained from a given trader. These samples were crushed with a mortar and pestle, sieved ($\phi \leq 1$ mm), and then packaged in polyethylene bags until analyses. Owing to their hygroscopicity, evaporites of plant-based ash filtrates were freeze-dried before crushing, sieving and packaging.

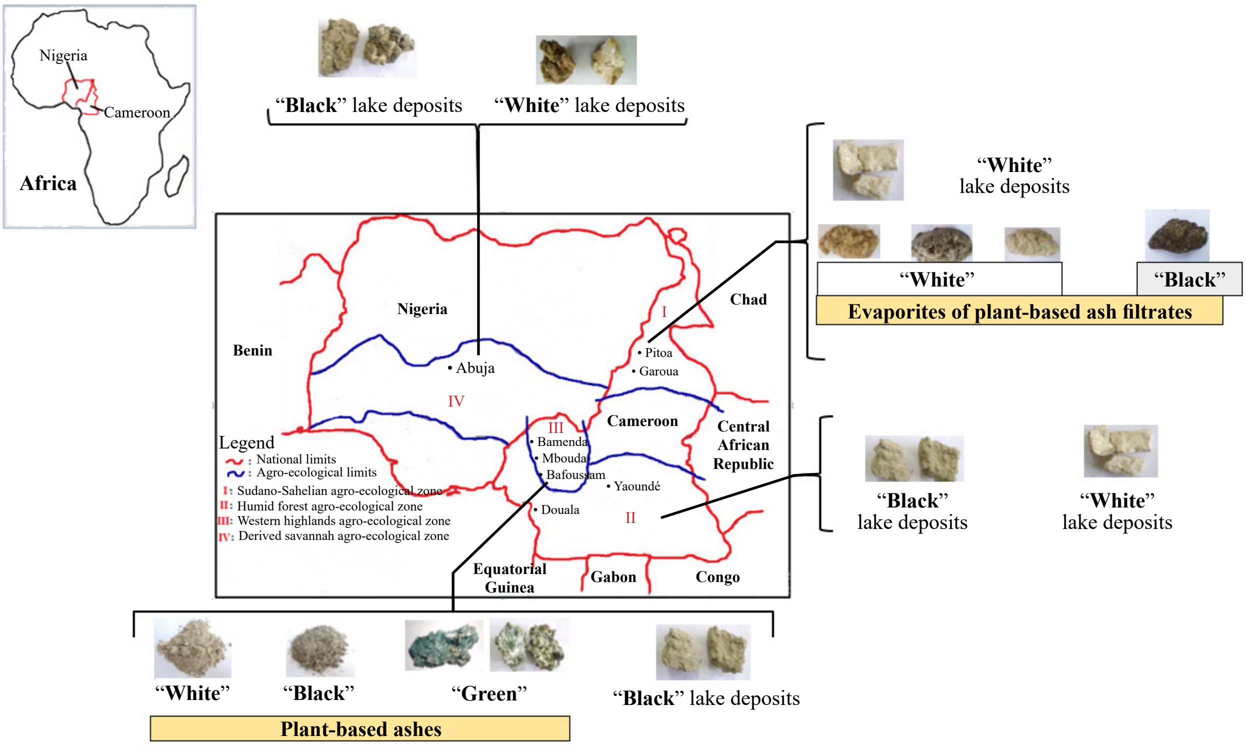

Figure 1. Sampling areas of the main types of TAS

Stock solutions of TAS were prepared in distilled water at concentrations varying between 2.4 g/L and 3.5 g/L, then filtered on Whatman filter paper no. 42 and kept in closed containers before cations and anions analyses.

2.2 *Ionic composition of TAS*

2.2.1 *Determination of cations*

Cations (Na^+, K^+, Mg^{2+}, Ca^{2+}, Cu^{2+}, Fe^{3+}, Ni^{2+}, Cr^{3+}, Zn^{2+}, Pb^{2+} and Cd^{2+}) of TAS were determined using prepared stock solutions. These solutions were diluted (dilution factor of 1/12 for Na^+, K^+, and Ca^{2+}); and 1/4 for the remaining cations) in 0.5N nitric acid before analysis. The standard of each cation was prepared in 0.5N nitric acid using specific salt (see data availability statement) for each cation and diluted to obtain a range of concentrations for the calibration curve (see data availability statement). Atomic absorption spectrometer (PerkinElmer AAnalyst 700) was used at a specific wavelength(s) for each cation (see data availability statement), and cation contents of TAS were determined from standard calibration curves.

2.2.2 *Determination of anions*

The anions that the content was assessed were: chlorides (Cl^-), phosphates ($H_2PO_4^-$), carbonates (CO_3^{2-}), and bicarbonates (HCO_3^-). Chlorides and phosphates were determined using the methods of Barney and Bertolacini [35], and Jeffery et al. [36], respectively. Carbonate and bicarbonate contents were determined by titration. In this respect, a volume, V (≈ 40 mL) of stock solution of each TAS was titrated by progressive addition of 0.1N hydrochloric acid in the stock solution, while agitating this solution using a magnetic agitator. The measurement of pH (Adwa AD1020) was done after each addition of hydrochloric acid solution (between 0.5 mL and 1 mL), up to pH ≈ 2.10. The end point of carbonates and bicarbonates titration (pH = 8.3 and pH = 3.7 respectively) was used to identify the complete titration of each anion, and the corresponding volume of hydrochloric acid solution added was used for quantification.

2.3 *Solution properties of TAS*

These properties were determined using TAS solutions prepared at different concentrations. Many TAS solutions were prepared from 10 g/L up to concentrations above saturation and kept at 30 °C. From the lower concentrated solution to the next one, the quantity of added salt was 40 g when the solution was brought back to 1 L. Different concentrations of each TAS sample were prepared by mixing a given mass of TAS (dry mass basis) with 25 g of distilled water in a sample vial. The sample vial was closed with an air-tight cover and sonicated (Elma E 30H Elmasonic) at 30 °C until the solution became homogenous (5 to 15 minutes depending on concentration), then incubated into the water bath (DAIHAN Scientific) at 30 °C during 12 to 14 hours in order to allow an equilibrium to be established. The sonication was done in order to speed up the dissolution of these salts as done at a household scale with the help of heat when cooking. After this time, samples were removed from the water bath, filtered on Whatman filter paper no. 1 and incubated again for 12 to 14 hours. After final incubation, TAS solutions were analysed for:

- pH (Adwa AD1020);
- Electrical conductivity (EC, mS/Cm, InoLab Cond7110);
- Turbidity (NTU, Eutech Instruments turbidimeter TN-100);
- Solubility (g/100 g of water) of filtered TAS solution through 24 hours of 105 °C oven drying (Model PH 0305). The formula presented in Equation (1) was used to determine the solubility,

$$\text{Solubility}\left(\frac{\text{g}}{100}\,\text{g H}_2\text{O}\right)=100*\left(\frac{\left(m_{\text{bds}}-m_{\text{b}}\right)}{\left(\left(m_{\text{bds}}-m_{\text{b}}\right)+\left(m_{\text{bds}}-m_{\text{bs}}\right)\right)}\right)=100*\left(\frac{m_{\text{s}}}{m_{\text{s}}+m_{\text{e}}}\right) \quad (1)$$

m_{bds}: mass of the bottle containing the dried salt; m_{b}: mass of the empty bottle; m_{bs}: mass of the bottle containing the solution of salt; m_{s}: mass of dried salt; m_{e}: mass of water.

Insoluble index (*II*, %) [8] through 24 hours of oven drying at 105 °C (Model PH 0305), of undissolved matters retained on filter papers during filtration of TAS solutions (Equation (2)).

$$II(\%)=100*\left(\frac{\left(m_{\text{fs}}-m_{\text{fp}}\right)+\left(m_{\text{fp}}*\left(\frac{W_{\text{pf}}}{100}\right)\right)}{\left(m_{\text{s}}\right)}\right) \quad (2)$$

m_{fs}: mass of dried salt and filter paper (g); m_{fp}: mass of empty filter paper (g); W_{pf}: water content of filter paper (%); m_{s}: dry mass of salt.

Electrical conductivity can be used to measure the mobility of ions in a given solution [37]. A saturated solution is one in which there is no longer an increase in ions solvatation, and thus there is no increase in the number of ions. Using electrical conductivity, the saturation concentration was identified for a given TAS as the first concentration above which there was no increase in its electrical conductivity.

2.4 *Solution properties of TAS*

All the data were obtained from at least three different measurements and results were expressed as average ± standard deviation. An analysis of variance (ANOVA) was undergone to determine the effect of different factors (agro-ecological area and/or colour) on the chemical composition of TAS and Duncan test was used to differentiate samples which are significantly different ($p \leq 0.05$). The software STATGRAPHICS Centurion XVI.I was used in this respect. All the curves were drawn with the software Sigmaplot 12.0.

3. Results and Discussion

3.1 *Chemical composition of TAS*

Sodium, potassium, chlorides, phosphates, carbonates, and bicarbonates appeared as the main common components of all the types of TAS, regardless of the location where they were sampled, as well as their colour (Tables 1 and 2). Iron and copper were not detected in plant-based ash salts, whereas iron was not detected in lake deposits. Plant-based ash salts are richer in potassium whereas lake deposits are richer in sodium. This latter observation complies with published findings [7, 8, 17-19, 21], and may even justify the statements of TAS users, who claim the positive effects of plant-based ash salts and negative effects of lake deposits on health [25]. The regular consumption of potassium is known to improve the functioning of kidneys, bones and cardiovascular system [38, 39], while the consumption of sodium is associated with non-communicable diseases [40, 41]. Due to the fact that bivalent cations are important determinants of the viscosity of charged hydrocolloids found in sticky vegetables, as well as of the maintenance of the green colour of vegetables during cooking [42, 43], it can be hypothesised that their presence in TAS justifies these reported functionalities.

With respect to lake deposits, the ionic composition is significantly related to the colour of salts (Table 1), particularly for major ions (sodium, potassium, carbonates, and bicarbonates). Generally, “white” samples have a higher content of the major components than the “black” ones. When taking into consideration the agro-ecological zones in Cameroon, for a given colour, there was not a significant variation amongst the content of major components. This shows that lake deposits which are used all over Cameroon might originate from the same source. This is also true for samples collected in different market stalls in Nigeria, which have a similar chemical composition, although they are significantly different from those found in Cameroon (Table 1). Lake deposits sampled in Cameroon generally had higher sodium content (two times more for “black” colour), carbonates (three times higher for both colours) and bicarbonates (five times higher for both colours) contents than those sampled in Abuja, Nigeria. This shows that samples found all over Cameroon as well as those found in Nigeria have different sampling origins. The colour seems to be related to the distribution of heavy metals. In this respect, neither lead nor cadmium was detected in “white” samples of lake deposits, regardless of the location where sampling was done. Though they are often found in “black” samples, the occurrence of cadmium (found in 11 of 17 samples) is more important than that of lead (found in 4 of 17 samples). When comparing “black” lake deposits of both countries, those sampled in Nigeria are 1.5 times higher than those of Cameroon, when found.

The nutritional risk (due to the unhealthy effects of sodium) is more important for samples of Cameroon while the toxicological risk (due to heavy metals) is more important for samples of Abuja. When comparing both countries and both colours of lake deposits, the carbonate/bicarbonate ratio of “white” lake deposits was two times higher in Nigeria than in Cameroon and therefore, this could be served as a thumb role to identifying the origin of lake deposits.

The higher concentration of ions in “white” samples, which is probably associated with their more important efficiency, is the justification of the fact that TAS users always prefer this colour of lake deposits whenever they can make a choice [25]. The absence of heavy metals as shown in this study is an extra advantage. In fact, lead and cadmium are metals found in many minerals [44, 45]. Lake deposits are sedimentary rocks formed in water. Radulescu et al. [46] have shown that naturally-occurring water containing mud and silts are generally richer in heavy metals (like lead and cadmium). In light of this finding, it can be hypothesized that “black” lake deposits are richer in mud and silts, while “white” lake deposits do not contain them.

Table 1. Ion contents of lake deposits

Ion	Colour*	Agro-ecological areas			
		Derived savannah	Sudano-Sahelian	Humid forest	Western highlands
Na^+(mg/g)	White	760 ± 7^{Aa}	922 ± 27^{b}	890 ± 33^{Ab}	NF
	Black	296 ± 2^{Ba}	NF	498 ± 72^{Bb}	585 ± 133^{b}
K^+(mg/g)	White	26 ± 1^{Aa}	34 ± 22^{a}	34 ± 11^{Aa}	NF
	Black	39 ± 3^{Ba}	NF	49 ± 16^{Aa}	53 ± 11^{a}
Ca^{2+}(mg/g)	White	1.3 ± 0.2^{Aab}	1.9 ± 0.4^{a}	0.9 ± 0.2^{Ab}(3/4)	NF
	Black	0.62 ± 0.04^{Ba}	NF	1.7 ± 0.4^{Bb}	1.6 ± 0.5^{ab} (10/13)
Mg^{2+}(µg/g)	White	50 ± 20^{Aa}	80 ± 30^{a}	50 ± 30^{Aa}	NF
	Black	10.5 ± 0.4^{Ba}	NF	30 ± 20^{Ab}	30 ± 30^{b}
Cu^{2+}(µg/g)	White	ND	ND	14 ± 4 (1/4)	NF
	Black	ND	NF	ND	19 ± 4 (1/13)
Zn^{2+}(µg/g)	White	ND	4.2 ± 0.3^{a} (1/3)	7 ± 1^{Ab} (1/4)	NF
	Black	ND	NF	4 ± 1^{Ba} (1/3)	7 ± 1^{b} (6/13)
Cr^{3+}(µg/g)	White	60 ± 5^{Aa}	35 ± 7^{b} (1/3)	24 ± 5^{Ab} (1/4)	NF
	Black	76 ± 2^{Ba}	NF	40 ± 6^{Bb}	40 ± 7^{b}
Ni^{2+}(µg/g)	White	24 ± 3^{A}	ND	ND	NF
	Black	44.5 ± 0.4^{Ba}	NF	ND	31 ± 5^{b} (3/13)
Cd^{2+}(µg/g)	White	ND	ND	ND	NF
	Black	48 ± 5^{a}	NF	34 ± 5^{b} (2/3)	37 ± 5^{b} (8/13)
Pb^{2+}(µg/g)	White	ND	ND	ND	NF
	Black	80 ± 3^{a}	NF	ND	53 ± 8^{b} (3/13)
Cl^-(mg/g)	White	1.1 ± 0.4^{Aa}	0.4 ± 0.1^{b}	0.5 ± 0.3^{Aab}	NF
	Black	0.2307 ± 0.0001^{Ba}	NF	0.5 ± 0.2^{Ab}	0.8 ± 0.4^{b}
$H_2PO_4^-$(mg/g)	White	0.31 ± 0.05^{Aa}	3 ± 2^{b}	1.6 ± 0.8^{Ab}	NF
	Black	1.41 ± 0.04^{Ba}	NF	0.59 ± 0.03^{Bb}	1 ± 1^{ab}
CO_3^{2-}(mg/g)	White	133 ± 14^{Aa}	380 ± 20^{b}	378 ± 63^{Ab}	NF
	Black	94 ± 2^{Ba}	NF	187 ± 47^{Bb}	222 ± 60^{b}
HCO_3^-(mg/g)	White	84 ± 6^{Aa}	440 ± 24^{b}	426 ± 49^{Ab}	NF
	Black	133 ± 11^{Ba}	NF	223 ± 59^{Bb}	256 ± 72^{b}
CO_3^{2-}/HCO_3^- ratio	White	1.59 ± 0.09^{Aa}	0.88 ± 0.02^{b}	0.86 ± 0.04^{Ab}	NF
	Black	0.70 ± 0.03^{Ba}	NF	0.84 ± 0.03^{Ab}	0.84 ± 0.6^{b}

*Note: The colour here is the one defined by stakeholders involved in TAS; NF: Not Found in the corresponding agro-ecological zone; ND: Not detected at the corresponding threshold detection for this metal; A, B: For the same agro-ecological area, the same upper case letter in the index for the two colours shows that their ions contents are not significantly different ($p = 0.05$); a, b: For a given colour of all the considered agro-ecological area, the same lower case letter in the index shows their ions contents are not significantly different ($p = 0.05$); In the case of agro-ecological areas of Cameroon, for some cations, the number in the parenthesis represents the number of samples out of the total analysed samples, for which this cation has been detected

With respect to plant-based ash salts, although there is a significant difference in major cations between colours of the same type of salt, this difference is generally less than 8%. The main difference between the colours of plant-based ash salts is in terms of their anion contents (chloride, phosphates, carbonates and bicarbonates). Heavy metals are always present in all types of plant-based ash salts, showing the potential toxicity of these salts for food consumption. The presence of heavy metals in plant-based ash salts can be due to environmental conditions of the place where these plants have been obtained, the nature of plants used, agricultural inputs used during farming (fertilizers, pesticides, herbicides, insecticides) or processing of plants (petrol to light the fire). In fact, it has been shown that some plants like sorghum (*Sorghum bicolor*) or maize (*Zea mays*), which were reported to be used for the manufacture of plant-based ash salts [13, 25, 29], have the ability to take up heavy metals from soils where they grow [47, 48]. Fertilizers are an important source of cadmium whereas herbicides, insecticides, petrol or car gazes are an important source of lead [49]. Heavy metals (cadmium and/or lead) were sometimes found in plant-based ash salts made from different plants (corn cob, banana and plantain peels, empty palm oil bunches) [9, 12, 17], the quantity being sometimes 50 times the quantity found in the current study (37.4 to 445.5 µg/g of salt) [9]. This high toxicity risk which is systematically associated with plant-based ash salts shows the necessity to study their toxicity as well as to train people on the effect of agricultural practices and burning conditions on their health. Regardless of the type of plant-based ash salt studied, the concentration of cadmium is similar and lead is always found in higher quantities than cadmium (Table 2). The difference in carbonate content of plant-based ash salts is not more than 10% while evaporites of plant-based ash filtrates have three times more bicarbonate (Table 2). When comparing the carbonate/bicarbonate ratio as well as the potassium content of different samples of evaporites of plant-based ash filtrates, there is not a significant difference, showing that for these samples, the colour seems not to be an element of differentiation. This might also traduce the fact that the same plant or plant part is used for the manufacture of evaporites of plant-based ash filtrates in their area of sampling.

Table 2. Ion contents of plant-based ashes and evaporites of plant-based ash filtrates

Ions	Evaporites of plant-based ash filtrates		Plant-based ashes		
	Black	White	Green	Black	10 ± 3a
Na^+ (mg/g)	11 ± 1^a	10 ± 3^a	11 ± 2^a	$14 \pm 1a^b$	15.43 ± 0.30^c
K^+ (mg/g)	232 ± 1^a	224 ± 1^b	207 ± 1^a	189 ± 1^b	209.10 ± 0.50^c
Mg^{2+} (µg/g)	249 ± 1^a	230 ± 8^b	ND	252 ± 1^c	ND
Ca^{2+} (µg/g)	700 ± 100	ND	ND	1090 ± 40^a	570 ± 30^b
Zn^{2+} (µg/g)	6.0 ± 0.8^a	4 ± 2^b	5.40 ± 0.40^a	3.80 ± 0.60^b	4.20 ± 0.90^{ab}
Cr^{3+} (µg/g)	47 ± 4a	44 ± 6^a	64 ± 4^a	83 ± 3^b	82 ± 5^b
Ni^{2+} (µg/g)	35 ± 1^a	29 ± 4^b	32.60 ± 0.50^a	34 ± 3^a	30 ± 4^a
Cd^{2+} (µg/g)	33 ± 2^a	33 ± 2^a	36 ± 3^a	33 ± 3^a	37 ± 2^a
Pb^{2+} (µg/g)	63 ± 1^a	45 ± 7^b	47 ± 1^a	66 ± 9^b	56 ± 2^b
Cl^- (µg/g)	350 ± 1^a	333 ± 79^b	195.70 ± 0.10^a	241.30 ± 0.10^b	370.60 ± 0.40^c
$H_2PO_4^-$ (µg/g)	865 ± 1^a	4000 ± 200^b	855 ± 4^a	2620 ± 30^b	200 ± 10^c
CO_3^{2-} (mg/g)	278 ± 3^a	235 ± 17^b	315 ± 5^a	251 ± 2^b	304 ± 1^c
HCO_3^- (mg/g)	168 ± 5^a	143 ± 17^b	ND	62 ± 3^a	61 ± 4^a
CO_3^{2-}/HCO_3^-	1.66 ± 0.60	1.67 ± 0.28	315/0	4.06	4.94

*Note: ND: Not detected at the corresponding threshold detection for this ion; a, b: for different colours of the same type of TAS, values with the same letter as index are not significantly different ($p = 0.05$)

The presence and importance of carbonates and/or bicarbonates in TAS, which are known to form basic solutions when considered alone in a given solution [50, 51], allows understanding that they either determine or contribute significantly to the alkalinity of TAS.

3.2 *Solution properties of TAS*

3.2.1 *The behaviour of TAS in solution: solubility and turbidity*

Since properties of TAS are observed in solution, their solubility and/or insoluble index can be considered indicative of their behaviour in solution. In this respect, evaporites of plant-based ash filtrates display a higher solubility and an obviously lower insolubility. It was shown that the insoluble index of evaporites of plant-based ash filtrates can vary between 0.82% and 8% [21]. Lake deposits and plant-based ashes have a lower solubility and thus, a higher insolubility (Figure 2). This behaviour can be explained by the fact that solutions of lake deposits contain insoluble minerals [26] and probably mud (for “black” samples), plant-based ashes have charred organic matter, while evaporites result from filtrates. In addition, the colour of salts appears as a discriminant factor of solubility since lake deposits of “white” colour display higher solubility than samples of “black” colour, regardless of the sampling locality. For plant-based ashes, this discrimination is observed around saturation concentrations.

With respect to lake deposits, for samples of the same colour, there is a variation of the insoluble index at the same concentration. This complies with the report of Saidou et al. [8], who found that the insoluble index of lake deposits varied between 1.29% and 36.64% at the concentration of 20 g/L. In general, and particularly for lake deposits, samples displaying higher solubility (“white” colour) are also those with higher ions contents (Table 1).

Lake deposits display variable and higher turbidity, mainly for samples of “black” colour, while turbidity of solutions of plant-based ashes and evaporites of their filtrates are relatively low, constant and stable, regardless of the concentration (Figure 3). In fact, lake deposits are sedimentary rocks containing mud and silts, resulting in a higher insoluble index and turbidity values [46, 52]. Higher turbidity values for “black” samples of lake deposits when compared to “white” ones, might traduce their higher content of mud and silts, and this might justify the presence of heavy metals and lower ion content of samples of this colour (“black”) [46, 52].

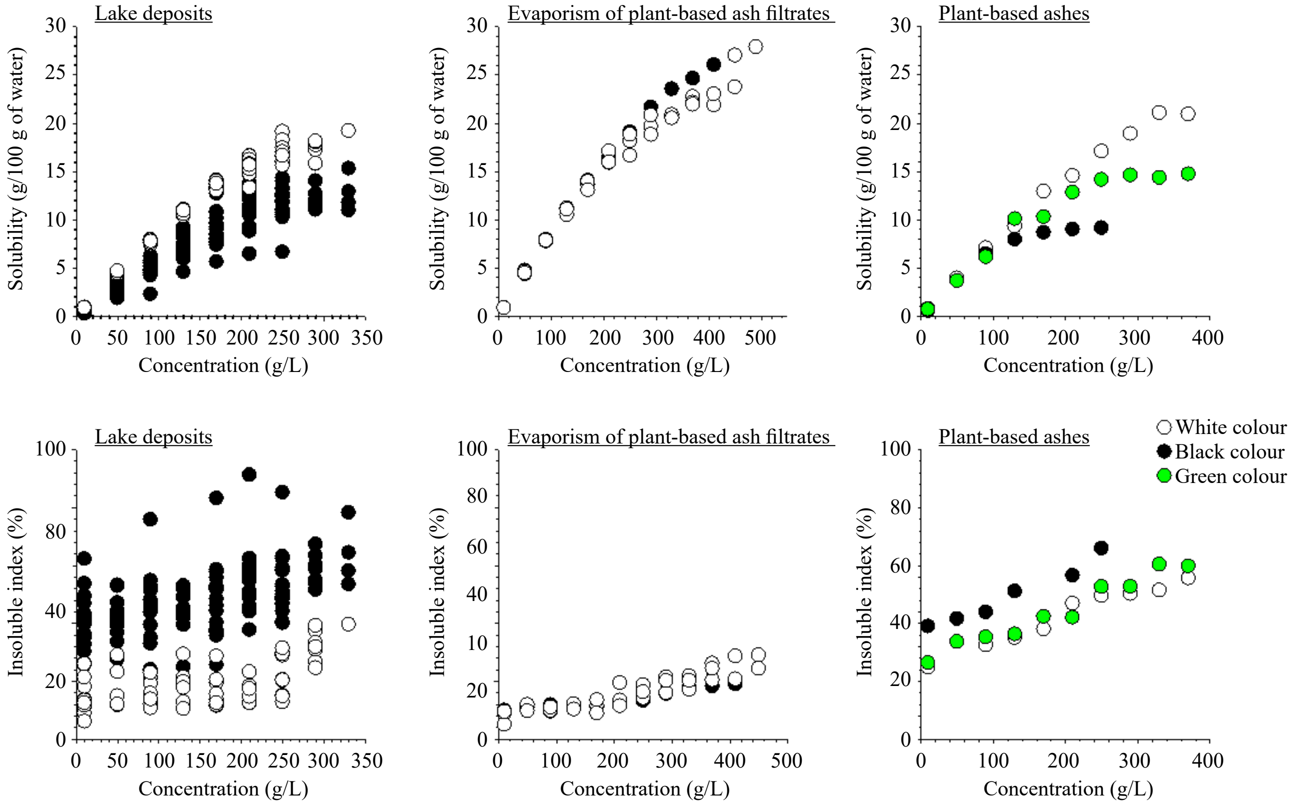

Figure 2. Solubility and insoluble index of TAS

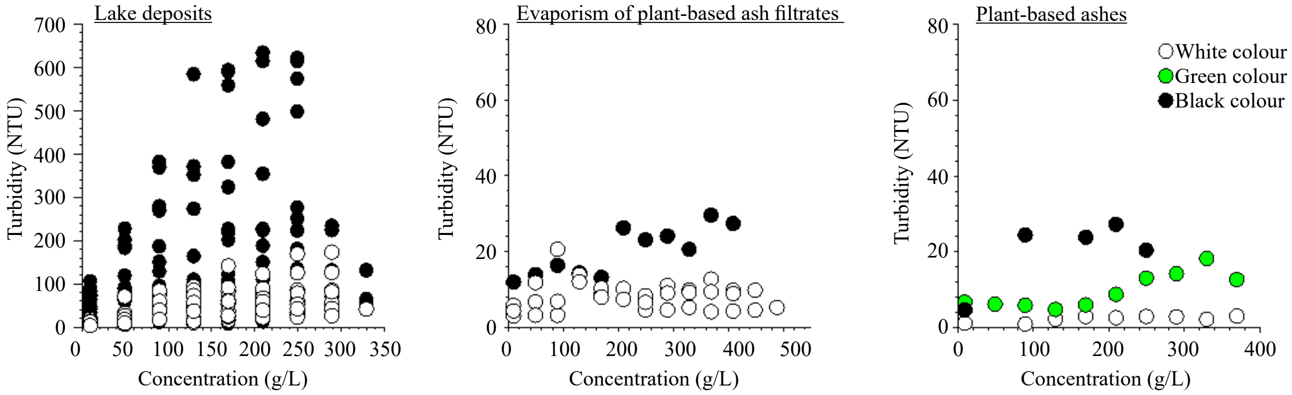

Figure 3. Turbidity of TAS

3.2.2 *Concentration dependency of pH*

From a general point of view, the pH of prepared TAS solutions is alkaline and varies between 9.83 and 14.4, depending on the concentration range (Figure 4; for each type of salt, the colour used to draw the figure is the colour of the salt as designated by users). These pH values are coherent with the findings of different authors [7, 8, 10, 12, 14, 15, 17, 21, 24]. Meanwhile, the pH of solutions of lake deposits, evaporites of plant-based ash filtrates and “white” plant-based ashes display buffer behaviour, with pH (10 to 11) remaining relatively stable when increasing the concentrations of salts in the solution up to saturation. The present study seems to indicate that buffer behaviour is a general characteristic of lake deposits found in Cameroon and Nigeria (“white” lake deposits of Nigeria seem to have higher pH than those found in Cameroon) as well as of evaporites of plant-based ash filtrates. The behaviour is different for “black” and “green” plant-based ashes, for which a concentration dependency of pH is displayed.

In fact, buffer behaviour is due to the simultaneous presence of an acid/base couple in the solution. Since almost all the TAS analysed in the present study contain bicarbonates and carbonates (Tables 1 and 2), which are known to form an acid/base couple with a pKa of 10.35 [50, 51], providing solutions of pH between 10 and 11, it could be assumed that they are responsible for the buffer behaviour. In naturally-occurring water, carbonates and bicarbonates are almost exclusively of biogenic origin and result from the microbial anaerobic oxidation of methane, and the dissolution of atmospheric carbon dioxide in water [53-55]. When these water sources are evaporated away, these anions are concentrated in sedimentary rocks like lake deposits. In plant-based ashes and derivatives, the presence of carbonates and bicarbonates results from the combustion of plants, where they are mixed with metal oxides, depending on combustion conditions (temperature and time) [50, 51]. In general, CO_3^{-}/HCO_3^{-} ratio defines the pH level of TAS solution (Equation (3)) [25], with a higher concentration of carbonates (higher ratio) leading to higher pH, while a higher concentration of bicarbonates reduces pH values (Table 3).

$$\mathrm{pH} = \mathrm{pKa} + \log\left(\frac{C_{\mathrm{car}}}{C_{\mathrm{bic}}}\right) \tag{3}$$

In this respect, a comparison of the value of this ratio for different TAS (Table 3) shows that solutions of plant-based ash salts have higher ratios than those of lake deposits, justifying the higher alkalinity of their solutions, and thus their higher efficiency for food tenderisation as claimed by TAS users [25]. This seems to show that alkalinity is the determinant of TAS efficiency. It has already been shown that the pH of solutions of plant-based ashes found in market stalls varies between 10.95 and 13.04 [14]. Moreover, solutions of plant-based ashes are more alkaline than solutions of their evaporites, with “green” plant-based ashes displaying an absence of bicarbonates (ratio = 315/0). This finding, coupled with the concentration dependency of pH of solutions of “black” and “green” plant-based ashes, implies the intervention of a supplementary factor, which may be linked to the combustion conditions of plants used for salt processing. It has been shown that the modification of combustion temperature and time of plants directly impacts the pH of resulting plant-based ash salts [14]. Since the variation of pH is a consequence of the variation of carbonates and bicarbonates contents of a sample as shown with Equation (3) as well as many other authors [7, 50, 56], it can be concluded that combustion conditions directly affect the chemical composition of plant-based ash salts.

The effect of combustion conditions can be understood with the example of calcium bicarbonate. In fact, calcium bicarbonate ($Ca(HCO_3)_2$) is known to be decomposed into carbonates on heating, which can further be decomposed at 840 °C to form calcium oxide, carbon dioxide, and water (Equation (4)).

$$Ca(HCO_3)_2 \longrightarrow CaCO_3 + H_2O \longrightarrow CaO + H_2O + CO_2 \qquad (4)$$

Metal oxides (like calcium oxide) are known to form strong alkaline solutions which display pH dependency to concentration when dissolved in water [50, 51]. Carbonates, bicarbonates, and hydroxides (which are formed from metal oxides) have been shown to be present in plant-based ashes [31]. In the current study, some solutions of plant-based ashes show pH dependency on concentration, and carbonates are always found in these salts, generally in combination with bicarbonates. It can therefore be suggested that components which are responsible for the solution behaviour of plant-based ashes are obtained from combustion. Thus, during combustion, carbonates and probably bicarbonates are produced, and when combustion is going on, bicarbonates react to give carbonates, which later react to give oxides, which in presence of water, can become hydroxides. Based on the results obtained through the determination of pH, it can be said that the buffer behaviour of "white" plant-based ashes can be due to the absence of formation of oxides, combined with the presence of carbonates and bicarbonates, due to combustion conditions (not too advanced). Despite the presence of carbonates ("green" plant-based ashes), and eventually bicarbonates ("black" plant-based ashes), the dependency of the concentration of their solution to pH can only be explained by more complete combustion and thus, the presence of metal oxides. In this case, in solution, the hydroxides derived from metal oxides increase significantly the pH, acting thus in competition with the carbonate-bicarbonate couple. In the particular case of "green" plant-based ashes, the absence of bicarbonates, combined with the more alkaline behaviour of its solution seems to indicate the more important formation of oxides and conversion of all the bicarbonates to carbonates, due to more severe burning conditions when compared to other plant-based ashes.

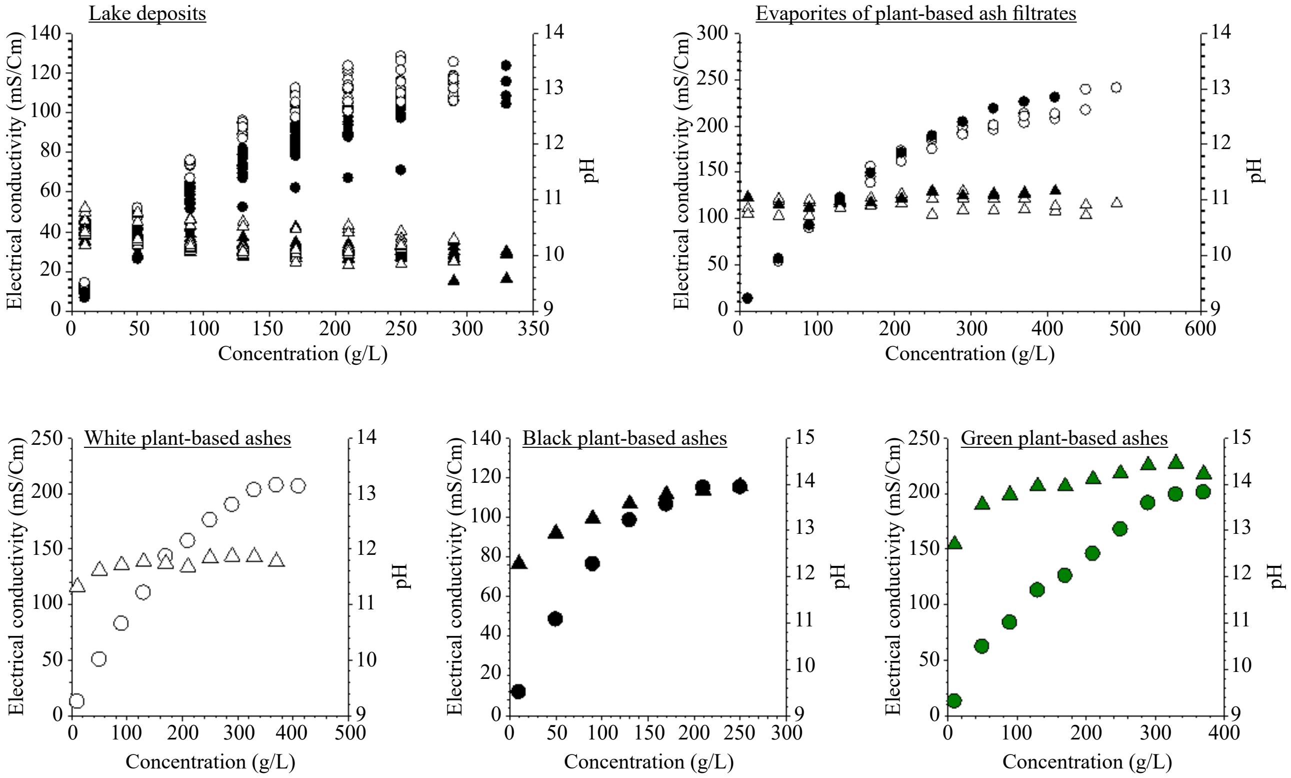

Figure 4. Relation between bulk concentration, electrical conductivity and pH of TAS solutions

Table 3. Solution properties of TAS

Type	Colour	Country	The concentration of TAS at saturation (%)	pH		$\frac{CO_3^{2-}}{HCO_3^{2-}}$ ratio
				At 1% TAS	At TAS saturation	
Lake deposits	Black	Cameroon	21 - 25	10.49 ± 0.12	10.02 ± 0.14	0.84 ± 0.05
		Nigeria	21	10.51 ± 0.02	10.21 ± 0.01	0.70 ± 0.03
	White	Cameroon	17-21	10.42 ± 0.13	10.06 ± 0.11	0.97 ± 0.25
		Nigeria	21	10.65 - 10.86	10.41 - 10.54	1.59 ± 0.09
Evaporites of plant-based ash filtrates	Black	Cameroon	37	11.05 ± 0.01	11.13 ± 0.02	1.66 ± 0.07
	White	Cameroon	29-45	10.81 ± 0.06	10.87 ± 0.19	1.67 ± 0.28
Plant-based ashes	Green	Cameroon	29	12.69 ± 0.02	14.41 ± 0.02	315/0
	Black	Cameroon	21	12.28 ± 0.01	13.86 ± 0.07	4.06 ± 0.01
	White	Cameroon	33	11.32 ± 0.01	11.85 ± 0.01	4.94 ± 0.02

4. Conclusion

Sodium, potassium, carbonates and bicarbonates are major ions of TAS, sodium being the main cation of lake deposits while potassium is the major cation of plant-based ash salts. Lake deposits used in Cameroon and Nigeria have different origins, and those used all over Cameroon have the same source of sampling. Their colour determines their chemical composition, with “white” colour having more major ions (sodium, potassium, carbonates, and bicarbonates) and no heavy metals, while those with “black” colour generally have cadmium and sometimes lead. Lake deposits, “white” plant-based ash and evaporites of plant-based ash filtrates form buffer solution, the buffer behaviour being due to the simultaneous presence of carbonates and bicarbonates, in combination with the absence of oxides and/or hydroxides, the higher the carbonates/bicarbonates ratio, the higher is the pH. “Green” and “black” plant-based ash, despite their carbonates and sometimes bicarbonates content, did not form buffer solutions, the behaviour being probably due to the presence of oxides.

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Author contribution

Robert Ndjouenkeu designed the whole work, did the statistical analysis, drew the curves, read and corrected the manuscript and approved the final manuscript. Franklin Kégah Ngoualem did the work in the laboratory, wrote the manuscript, did the statistical analysis, drew the curves, and approved the final manuscript. Richard Marcel Nguimbou and Asad Muhammad Khan contributed to statistical analysis, read and corrected the final manuscript.

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Conflict of interest

The authors report there are no competing interests to declare.

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