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Artigo

Aqueous Phase Extraction as Sample Preparation Procedure for Sodium Determination in Infant Foods

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Extração em Fase Aquosa como Procedimento de Preparo de Amostras para a Determinação de Sódio em Alimentos Infantis

Resumo: Este trabalho objetivou desenvolver um procedimento de preparo de amostras, empregando a extração em fase aquosa, para a determinação de sódio (Na) em salgadinhos de trigo, salgadinhos de milho, papinhas industrializadas para bebês e macarrão instantâneo. As etapas incluíram extração com água ultrapura, agitação por vortex, centrifugação e quantificação do Na por fotometria de chama. A exatidão e a precisão foram avaliadas por testes de adição e recuperação, comparação com decomposição úmida, método de Mohr e uso de diferentes fotômetros de chama. O teste de Youden foi utilizado na avaliação da robustez, com a massa da amostra, força centrífuga relativa e tempo de agitação como variáveis. Diferenças significativas foram observadas ($p \leq 0,05$) em comparação com o método de Mohr e diferentes fotômetros de chama para as papinhas para bebês e macarrão instantâneo. Essas diferenças foram atribuídas à maior sensibilidade da fotometria de chama e erros instrumentais. As recuperações variaram entre 84-119 % para fotometria de chama e 89-108 % para o método Mohr, com desvios padrão relativos inferiores a 11 %. O tempo de agitação e a força centrífuga relativa tiveram um efeito positivo na robustez. O desempenho do procedimento foi verificado em várias amostras de alimentos, com valores na faixa de 129 a 300 mg Na / 25 g de salgadinhos de trigo e de milho, respectivamente; 1,5 a 81 mg Na / 115 g de papinhas para bebês e 1118 a 1962 mg Na / 85 g de macarrão instantâneo. Foi observada discrepância entre os valores obtidos e rotulados. Nesse contexto, o procedimento de extração de fase aquosa desenvolvido fornece uma alternativa simples, de baixo custo e livre de reagentes tóxicos aos métodos tradicionais de quantificação de Na em alimentos com diferentes concentrações.

Palavras-chave: Espectrometria de emissão atômica em chama; alimentos infantis; minerais; cloreto de sódio.

Abstract

This work aimed to develop a sample preparation procedure involving aqueous phase extraction, for the determination of sodium (Na) in wheat snacks, corn snacks, baby food and instant noodles. The steps included extraction with ultrapure water, vortex agitation, centrifugation and Na quantification by flame photometry. The accuracy and precision were evaluated by addition and recovery tests, comparison with wet decomposition, the Mohr method and by using a different flame photometer. The Youden test was used in the assessment of robustness, with sample mass, relative centrifugal force and stirring time as the variables. Significant differences were observed ($p \leq 0.05$) in comparison with the Mohr method and differents flame photometers for baby food and instant noodles. These differences were attributed to the higher sensitivity of the flame photometry and instrumental errors. The recoveries varied between 84–119 % for flame photometry and 89–108 % for the Mohr method, with relative standard deviations less than 11 %. The stirring time and relative centrifugal force had a positive effect on robustness. The performance of the procedure was verified in various food samples, with values in the range of 129–300 mg Na/25 g wheat snacks and corn chips, respectively, 1.5–81 mg Na/115 g baby food and 1118–1962 mg Na/85 g instant noodles. A discrepancy was observed between the values obtained and labelled. In this context, the developed aqueous phase extraction procedure provides a simple, low-cost and toxic reagent-free alternative to the traditional methods for Na quantification in foods with different concentrations range.

Keywords: Flame emission atomic spectrometry; infant foods; mineral; ·sodium chloride.

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Extração em Fase Aquosa como Procedimento de Preparo de Amostras para a Determinação de Sódio em Alimentos Infantis

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1. Introduction

The dietary habits of a large part of the global population have been modified in recent decades. In particular, the consumption of processed foods, which are highly appreciated by consumers, especially children and adolescents, is increasingly gaining space in the population's diet.[1,2]

Processed foods are commonly eaten together or replace the main daily meals, and account for about 80 % of daily sodium (Na) intake in developed countries. Early childhood is the stage that begins the development of the eating habits of human beings, as well as the knowledge of taste sensations. Excessive Na intake at this stage can influence the preference for salty foods in adulthood, increasing the likelihood of diseases associated with excess Na in the organism.[1-3]

Sodium is an essential mineral to the human organism, mainly in maintaining the plasma volume, acid-base balance, osmotic regulation, Na pump functioning and neurotransmission.[4] A high Na concentration in the human body is associated with cardiovascular diseases, hypertension, osteoporosis, gastric cancer and renal disorders, among others. In this context, researchers and authorities from different areas of science have sought alternatives for lowering Na in the diet of the world population, to reduce the damage to public health.[5-7]

The World Health Organisation (WHO) recommends a daily Na intake of 2000 mg for an adult. For children, there is no set value, but the indication is that it should be adjusted downwards, based on the energy requirements of the children relative to adults.[8] The European Society of Pediatrics, Gastroenterology, Herpetology and Nutrition also does not present specific recommendations but suggests not to add sodium chloride (NaCl) to foods during childhood. In Brazil, the legislation recommends a daily intake of

2400 mg Na.[8-11] Thus, the quality control of these products concerning the Na content allowed by the regulatory bodies, must be guaranteed.

The determination of metal species in foods is based on two stages, the sample preparation and the subsequent detection by quantification techniques. The first step is considered to possess the greatest degree of difficulty due, in most instances, to the need for chemical transformation of the sample, which typically involves dry or wet decomposition.[12] However, these procedures have disadvantages, such as high preparation time, volatilisation losses, high amount of residue generated, use of toxic reagents with high oxidant power and exposure of the analyst to the unhealthy environment.[13]

The high solubility of most Na salts in water[14] indicates that the Na ion can be isolated from the matrix without the need for complex chemical transformations, using extraction techniques. The extraction techniques for the quantitation of metal species can be an interesting and efficient alternative sample preparation procedure compared to the traditional methods.[15] Notably, the use of aqueous phase extraction as a sample preparation procedure for the determination of the concentration of water-soluble metal species, such as Na, may contribute to the decrease in the use of toxic chemical reagents and generation of residues and, furthermore, be compatible with detection by instrumental and classical analytical techniques. Some authors have already used this procedure for quantification of metals in foods.[16-18]

In consideration of the information mentioned above, this work aimed to develop a sample preparation procedure, using aqueous phase extraction, to determine the Na concentration in corn and wheat snacks, instant noodles and industrialised baby food by flame photometry.

2. Material and Methods

2.1. Reagents, solutions and samples

Precipitation titration of chloride ions was achieved by the Mohr method, using silver nitrate (Qhemis, São Paulo, Brazil) to precipitate the ions and potassium chromate 5 % (*w/v*) (Tec-Lab, São Paulo, Brazil) as the indicator. Analytical grade nitric acid (HNO_3 65 %, *v/v*; Qhemis) and hydrogen peroxide (H_2O_2 30 %, *v/v*; Synth, São Paulo, Brazil) were used in the wet decomposition. NaCl (purity ≥ 99.5 %; Quemis) was used for the fortification and spectrophotometric determination of Na. Before use, all flasks and glassware were cleaned with tap water and immersed in 1 % (*v/v*) aqueous alkaline detergent Extran MA01 (Merck, Germany) for at least 24 h. Afterwards, they were rinsed with tap water, soaked in 7 % (*v/v*) HNO_3 aqueous solution for at least 24 h and washed thoroughly with deionised water.

The standards, solutions and samples were weighed using an analytical balance with ±0.0001 g accuracy (Bel Marck model 210, Monza, Italy) and prepared using high-purity deionised water obtained from a Milli-Q instrument (18.2 MΩ cm; Millipore, Molsheim, France).

2.2. Sample preparation procedures

Initially, the samples were homogenised (Philips Walita blender, Minas Gerais, Brazil), aliquoted and, when necessary for solid samples, pulverised (Britania food grinder, São Paulo, Brazil), and dried to constant weight at 60 °C in a kiln with mechanical air circulation (Fanem model Orion 515, São Paulo, Brazil). Next, the weighed corn snack (0.5 g), wheat snack (0.5 g), instant noodles (0.5 g) and industrialized baby food (1.5 g, due to the low concentration of sodium present in this food, a larger sample mass was used in this case) samples were subjected to the sample preparation procedures described below.

For the wet decomposition, all of the weighed food samples were transferred, respectively, to digester tubes and then 5 mL of 65 % (*v/v*) HNO_3 and 1 mL of 30 % (*v/v*) H_2O_2 solutions were added. The tubes were placed in a digester heating block (Quimis model A242, São Paulo, Brazil) at 120 °C for about 3 h. After cooling, the digests were transferred quantitatively into a 100 mL volumetric flask and made to volume with deionised water.

For aqueous phase extractions, all of the weighed food samples were placed in 15 mL Falcon tubes (50 mL for instant noodles), and then 10 mL (40 mL for instant noodles) ultrapure water added. Afterwards, the tube contents were homogenised in a vortex shaker (Labnet, São Paulo, Brazil) for 2 min and centrifuged (Hettich model 320R, Tuttlingen, Germany) at 4500 rpm for 10 min at 25 °C (corn snack, wheat snack and instant noodle) and 35 °C (industrialised baby

food), respectively. The obtained supernatants were suitably diluted in ultrapure water, for the determination of Na by flame photometry and Mohr method.

2.3. Analytical figures of merit

Na was quantified by using a flame photometer (Analyser model 910, São Paulo, Brazil), with liquefied petroleum gas as fuel and compressed atmospheric air as the oxidising agent. The aspiration rate was adjusted to 2.0 ± 0.2 mL min^{-1}.

For determination of the instrumental parameters and figures of merit, the analytical curve was obtained by the external standardisation method. Accordingly, an aqueous stock solution (1000 mg L^{-1} Na) was prepared and the standards (0.0, 0.5, 1.0, 2.0, 2.5, 4.0, 5.0, 7.5, 10.0 mg L^{-1} Na) obtained by appropriate dilution.

The instrumental linearity was evaluated by the linear correlation coefficient (*r*) of the analytical curve.[19] The limits of detection (LOD) and quantification (LOQ) were determined according to the Association of Accredited Laboratories of Portugal (Relacre).[20]

The instrumental accuracy was expressed as the relative standard deviation (RSD, %) of the instrumental responses obtained through 20 successive measurements of an analytical standard. The instrumental precision was acquired from 15 measurements of an analytical standard and a researched sample replica, interspersed during the sample readings.

The precision and accuracy of the proposed sample preparation procedure were verified by comparing results obtained by different analytical techniques (flame photometry and titration precipitation) and different flame photometers. For comparison of the equipment, in addition to the flame photometer mentioned above, a Digimed flame photometer (model DM-62, São Paulo, Brazil), located at Mato Grosso Federal University (UFMT,1) and the Brazilian Industrial Apprenticeship Service (SENAI-MT, 2), respectively, was used.[21-22]

The precision and accuracy were also evaluated by addition and recovery tests using the proposed procedure.[21-22]For this, all the food samples were previously homogenised and weighed out into the appropriately sized Falcon tubes (see previous section), respectively. In the fortifications of industrialised baby food and instant noodle samples, different aliquots of aqueous Na solution, at 100 or 1000 mg L^{-1}, were added to the tubes to obtain fortification levels of 25, 50 and 75 % of the initial analyte concentration present in the sample. Conversely, the fortifications of the corn and wheat chips samples were made by the addition of different amounts of solid NaCl, to obtain each concentration level. The spiked samples were allowed to rest for 24 h, thereby ensuring interaction between the analytes and the sample matrices. Next, the aqueous phase extraction was applied to the fortified samples. All experiments were done in sextuplicate and followed by an analytical blank.

Analysis of variance with the post-hoc Tukey test ($p < 0.05$) was used to identify any significant differences among the results. These procedures were performed using Assistat software 7.7 beta version.

The robustness was evaluated by the operating conditions changes, using a fractional factorial design 2^{n-1}, where *n* is the number of variables, considering the following factors: the mass of the sample (g), relative centrifugal force (rpm) and stirring time (s). The variables operated on two levels expressed as the high level (+) and low level (-). For mass of the sample, the (-) was 0.30 g and (+) was 0.70 g for corn snack, wheat snack, instant noodle samples and, for industrialised baby food were 1.0 g (-) and 2.0 g (+). For relative centrifugal force the (-) was 4000 rpm and (+) was 5000 rpm; for stirring time the (-) was 1.5 minutes and (+) was 2.5 minutes (Table 1)

The effects of the variables in the determination of Na concentration were calculated by the Youden test, based on the recommendations of Relacre.[20]

The performance of the method was evaluated by flame photometric quantification of Na in various food brands and flavoured samples collected from supermarkets in Cuiabá city, Mato Grosso. The results were compared with the values labelled and with the current national and international legislation.

3. Results and Discussion

In the flame photometry determinations, the linear correlation coefficients (*r*) were higher than 0.99, indicating an excellent correlation between the emission intensity and Na concentration. The LOD and LOQ were 0.08 and 0.25 mg L^{-1},

Table 1. Experimental conditions selected to evaluate robustness *(n = 3)*

Sample	Variables		
	Sample mass (g) Low (-) - High (+) levels	Stirring time(min) Low (-) - High (+) levels	Relative centrifugal force (rpm) Low (-) - High (+) levels
Corn Snack	0.30 – 0.70	1.5 – 2.5	4000 – 5000
Wheat Snack	0.30 – 0.70	1.5 – 2.5	4000 – 5000
Industrialized Baby Food	1.0 – 2.0	1.5 – 2.5	4000 - 5000
Instant Noodles	0.30 – 0.70	1.5 – 2.5	4000 - 5000
Nominal condition / Industrialized Baby Food	1.5	2.0	4500
Nominal condition/another samples	0.5	2.0	4500

respectively, which were sufficiently low to quantify Na in the samples.

The RSD values, obtained by measuring 20 successive measurements of an analytical standard and 15 readings of an analytical standard and a researched sample replica interspersed during sample readings, were less than 5 %, demonstrating high instrumental accuracy.[23]

In the comparison between the analytical techniques of flame photometry and Mohr method using aqueous phase extraction for the various samples, only instant noodle and industrialised baby food samples presented significant differences ($p \leq 0.05$) (Table 2). This result can be justified due to the greater sensitivity of the instrumental flame photometry technique to the classical precipitation titration technique, which is an indirect method of Na quantification. However, the RSD obtained for both quantification techniques were inferior to 5%, indicating precision in the results.[23]

In the comparison of aqueous phase extraction with a wet decomposition, no significant differences were observed in any of the matrices (Table 2). The sample preparation by wet decomposition used 5 mL HNO_3 and 1 mL H_2O_2, and a preparation time of approximately 4 h. In contrast, the aqueous phase extraction only used deionised water as the solvent and required a preparation time of around 30 min, which eliminated the generation of toxic waste and minimised the exposure of the analyst to harmful substances. Moreover, the aqueous extraction procedure presented a shorter preparation time than traditional methods described in the literature for the same purpose.

In the comparison of the different instruments for aqueous phase extraction, significant differences ($p \leq 0.05$) were found for the corn snack, wheat snack and industrialised baby food samples (Table 3).

The observed differences can be justified by the variations in the instrumental and operational conditions during the tests that can generate errors[24], since the classical sample preparation, the wet decomposition also presented significant differences ($p \leq 0.05$) for all matrices.

Table 2. Results (mean ± %RSD) obtained in the comparison between the sample preparation procedures using flame photometry; and comparison between flame photometry and Mohr titration using aqueous phase extration procedure *(n=6)*

Samples	Sample preparation procedures		Analytical Techiniques		Concentration
	Aqueous phase extraction	Wet decompostion	Flame photometry	Mohr method	
	Mean ± %RSD*		Mean ± %RSD*		
Instant noodle	2134.5^{a} ± 2.5	1994.3^{a} ± 0.3	2134.5^{A} ± 2.5	1936.1^{B} ± 0.2	mg de Na/85g
Wheat snack	253.6^{a} ±3.4	261.7^{a} ± 1.8	253.6^{A} ± 3.4	268.4^{A} ± 1.8	mg de Na/25g
Corn snack	306.9^{a} ± 7.2	269.9^{a} ± 4.6	269.9^{A} ± 1.2	261.9^{A} ± 4.7	mg de Na/25g
Industrialized baby food	8.6^{a} ± 0.7	8.6^{a} ± 1.7	8.6^{B} ± 0.7	11.0^{A}± 0.1	mg de Na/115g

[a]Values followed by the same letter in the same line indicate no significant difference between the samples at the 5 % confidence level; * Relative Standard Deviation. [A, B] Values followed by the same letter in the same line indicate no significant difference between the samples at the 5 % confidence level; * Relative Standard Deviation

Table 3. Results (mean ± %RSD) obtained in the comparison na comparação between the aqueous phase extraction and wet decomposition in different flame photometers *(n=6)*

Samples	Photometer 1		Photometer 2		Concentration
	Aqueous phase extraction	Wet decompositionn	Aqueous phase extraction	Wet decomposition	
	Mean ± %RSD*				
Instant noodle	2134.5[a] ± 2.5	1994.3[a] ± 0.3	2105.9[a] ± 0.0	1692.2[b] ± 11.1	mg de Na/85g
Wheat snack	253.6[a] ± 3.4	261.7[a] ± 1.8	201.3b ± 3.9	209.0[b] ± 2.7	mg de Na/25g
Corn snack	306.9[a] ± 7.2	269.9[b] ± 4.7	241.0[bc] ± 7.4	221.0[c] ± 9.3	mg de Na/25g
Industrialized baby food	8.6[b] ± 0.7	8.6[b] ± 1.7	8.3[c] ± 2.2	9.1[a] ± 0.7	mg de Na/115g

[a, b, c] Values followed by the same letter in the same line indicate no significant difference between the samples at the 5 % confidence level; * Relative Standard Deviation; Photometer 1: Analyser (UFMT); Photometer 2: Digimed (SENAI)

The recoveries varied between 84–119 % for flame photometry and 89–108 % for the Mohr method, with relative standard deviations less than 11 %.The AOAC recommends recovery values between 80 and 110 %, with RSD lower than 16 % for the concentration range of mg kg^{-1}.[19] Nonetheless, according to the matrix complexity, instrumental analytical techniques and the method purposes, the recovery range can be increased.[25]

Considering the variations that may cause interference in the determination of Na by the proposed preparation procedure, the robustness was evaluated through the Youden test (Figure 1). The relative centrifugal force for all the samples and the sample mass for the corn snack, wheat snack and industrialised baby food samples showed negative effects, indicating that the proposed preparation procedure is robust to these variables.

The stirring time had a positive effect for all matrices, and the sample mass showed a positive effect only for the instant noodle sample. The positive effect suggests that the variables should undergo the least possible change in the application of the aqueous phase extraction proposed in the Na quantification, to avoid potential systematic and random errors.

The Na concentration in instant noodles samples ranged from 1117.6 to 1961.9 mg Na/85 g of sample. The highest amount was verified in the A brand, and only the MA sample presented a Na concentration in agreement with the labelled value (Table 4). Buzzo and co-authors used inductively coupled plasma-optical emission spectrometry to investigate the high levels of Na in industrialised foods (cornflour, biscuit, hamburger, sausage and instant noodles)

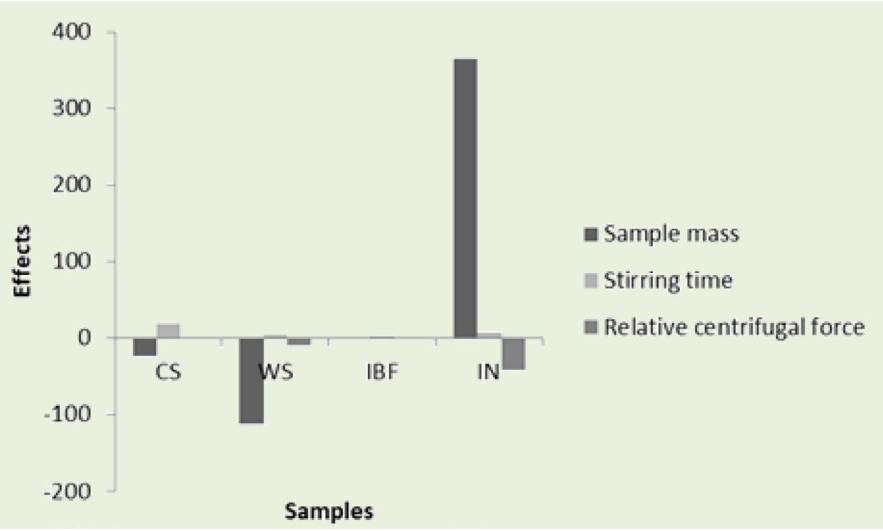

Figure 1. Results of the Youden test for the robustness evaluation. CS: corn snack; WS: wheat snack; IBF: infant baby food; IN: instant noodles

consumed by the Brazilian population and noted that the instant noodles recorded the highest Na concentration.[26]

The Na contents obtained in the corn snack varied from 128.6 to 208.6 mg Na/25 g of sample, respectively, and all the F brand samples recorded results higher than the labelled values. For the wheat snacks, the concentrations ranged from 158.0 to 300.4 mg Na/25 g of sample, and all results were lower than those described on the labels (Table 4).

In a technical report released in 2015 by the Brazilian National Health Surveillance Agency (ANVISA) showed an average value of 1623 mg Na/85 g instant noodle and 210 mg Na/25 g corn snack and these concentrations were higher than those obtained in the current work.[27]

Table 4. Sodium concentration (mean ± %RSD) and percentage of difference between obtained and labeled results in different brands and flavors of instant noodle, corn snack, wheat snack and industrialized baby food *(n=3)*

INSTANT NOODLE (mg de Na/85g)									
Brands		**A**			**B**			**C**	
Samples	MA*	MB*	MC*	MD*	ME*	MF*	MG*	MH*	MI*
Obtained results	1520.3 ± 3.0	1670.4 ± 2.2	1961.9 ± 3.2	1332.7 ± 0.9	1117.6 ± 1.6	1243.7 ± 6.8	1824.3 ± 2.4	1575.9 ± 8.3	1659.5 ± 3.9
Labeled results	1570	1620	1640	1277	1105	1193	1581	1460	1487
% Difference	- 3.2	+ 3.1	+ 20	+ 4.37	+ 1.0	+ 4.2	+ 15.4	+ 7.9	+ 11.6
CORN SNACK (mg de Na/25g)									
Brands		**D**			**E**			**F**	
Samples	SA*	SB*	SC*	SD*	SE*	SF*	SG*	SH*	SI*
Obtained results	174.1 ± 7.8	208.6 ± 2.5	188.2 ± 6.1	174.9 ± 0.7	141.3 ± 9.2	128.6 ± 13.0	201.7 ± 2.8	185.9 ± 2.6	145.5 ± 0.6
Labeled results	437	422	422	181	146	210	177	174	120
% Difference	- 60.2	- 50.6	- 55.4	- 3.4	-3.2	- 38.7	+ 14.0	+ 6.9	+ 21.3
WHEAT SNACK (mg de Na/25g)									
Brands		**G**			**H**			**I**	
Samples	TA*	TB*	TC*	TD*	TE*	TF*	TG*	TH*	TI*
Obtained results	277.2 ± 3.7	246.6 ± 5.3	252.1 ± 1.4	158.0 ± 1.3	176.5 ± 0.8	159.8 ± 0.9	272.1 ± 5.0	237.3 ± 3.2	300.4 ± 2.9
Labeled results	279	247	260	216	208	257	382	368	385
% Difference	- 0.6	- 0.2	- 3.0	- 27.0	- 15.1	- 38.0	- 28.8	- 35.5	- 22.0
INDUSTRIALIZED BABY FOOD (mg de Na/115g)									
Brands		**J**					**K**		
Samples	PA*	PB*	PC*	PD*	PE*	PF*	PG*	PH*	PI*
Obtained results	81.4 ± 1.8	75.1 ± 1.7	29.2 ± 1.0	41.8 ± 2.5	11.8 ± 2.7	13.7 ± 0.6	9.5 ± 0.7	1.5 ± 0.4	8.8 ± 3.5
Labeled results	123	118	27	24	14	9	10	5.8	6.7
% Difference	- 33.8	- 36.3	+ 8.1	+ 74.2	- 15.6	+ 52.7	- 4.8	- 74.2	+ 30.8

* Mean and Relative Standard Deviation of the triplicates of the analyzed samples. **Difference between obtained and labeled results in percentage. MA*: meat flavor; MB*: roast beef flavor; MC*: country chicken flavor; MD*: yakissoba flavor; ME*: bolognese flavor; MF*: chicken flavor; MG*: seasoned chicken flavor; MH*: cooked vegetables flavor; MI*: seasoned tomato flavor. SA*: pizza flavor; SB*: barbecue flavor; SC*: cream cheese flavor; SD*: cheese flavor; SE*: onion flavor; SF*: ham flavor; SG*: parmesan flavor; SH*: ham flavor; SI*: cream cheese flavor. TA*: barbecue flavor; TB*: parsley and onion flavor; TC*: pizza flavor; TD*: barbecue flavor; TE*: cheese flavor; TF*: pizza flavor; TG*: bacon flavor; TH*: barbecue flavor; TI: pepperoni flavor. PA*: meat with vegetables; PB*: meat with vegetables and rice; PC*: apple; PD*: assorted fruits with yogurt; PE*: meat, vegetables and mandioquinha; PF*: egg yolk, meat and vegetables; PG*: vegetables with meat; PH*: banana with oats; PI: plum.

Four samples of industrialised baby food showed results above those described on the label (PC*, PD*, PF* and PI*), with Na concentrations ranging from 1.5 to 81.4 mg Na/115 g of the sample (Table 4). Values close to this concentration range were obtained by Khamoni and co-authors, in a study on the impact of ingredients on the elemental content of baby food in the United Kingdom.[28]

The ANVISA Ordinance no. 34 of 13 January 1998 describes the technical regulation on the transition from feeding to infants and young children. It recommends less than 200 mg Na per 100 g of the food ready for consumption, and the addition of NaCl to fruit-based products and desserts is prohibited.[29] The fruit-based industrialised baby food evaluated in the current study presented Na concentrations between 1.3 and 71 mg Na/100 g, and PC* (apple flavour) and PI* (plum flavour) samples contained 25 and 8.0 mg Na/100 g, respectively, indicating the possible addition of NaCl in the formulation.

A Student's *t*-test comparing a measured value with a known value in a 95 % confidence interval[30] was applied to the results obtained and to the labelled values described in Table 4. Based on the analysis, all samples, except the industrialised baby food PB*, PC* and PD*, presented significant differences.

The Resolution RDC no. 360 of 23 September 2003, approving the technical regulation on nutritional labelling of packaged foods and making nutrition labelling obligatory[9], indicates a tolerance of +20 % compared to the values of nutrients declared on the label. It also states that for products containing micronutrients in quantities exceeding the tolerance established in the previous item, the company responsible shall keep available the studies that justify such variation.

Among the samples evaluated in the current work, the SI* instant noodle and the PD*, PF* and PI* industrialised baby foods showed Na contents above the permitted tolerance limit. Given this ignorance or disregard of the current Brazilian legislation, a review of the formulations and labels by the manufacturers, as well as the inspection by the competent organisations, is suggested.

Based on the daily recommended intake of 2000 mg Na[11], for the highest Na concentrations recorded, the daily intake of only 100 g of the samples corresponds approximately to 115% (instant noodles), 42% (corn snack), 61 % (wheat snack) and 3% (industrialised baby food) of the daily allowable intake. In this context, the daily intake value quoted is indicated only for adult consumers. For children, as mentioned above, there is no established value, merely an indication that it should be adjusted downwards, based on the energy requirements of children compared to adults. Therefore, the consumption of these foods by children and infants should be made with restrictions to minimise the risk of diseases related to excessive Na consumption in the future.

4. Conclusions

The results obtained in the addition and recovery tests, as well as in the comparisons with different analytical techniques, sample preparation procedures and instruments, indicated that the proposed protocol has the precision and accuracy necessary for the Na quantification in the samples of processed infant foods with different concentrations range evaluated in this study. The robustness tests demonstrated that the stirring time had a positive effect on all the assessed samples and the sample mass had a positive effect only for the instant noodle sample. Therefore, these two variables must undergo the least possible change during the application of the procedure to avoid errors in the results.

According to the data, the use of aqueous phase extraction combined with flame photometric quantification of Na determination in foods may be a promising alternative to classical sample preparation procedures. Moreover, it has several advantages, including the use of a single reagent and the reduction of toxic waste generation, costs and analysis time. In addition, the results suggest that the Mohr method can also be used as an indirect quantification technique.

The evaluated samples presented a discrepancy between the obtained and labelled values, suggesting a revision/update of the formulations and labels by the manufacturers, as well as a greater inspection by the regulatory agencies, aiming to guarantee the food quality and safety of these products.

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