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Sal rosa do Himalaia: determinação do teor de iodo, pesquisa de corantes e avaliação da rotulagem

Himalayan pink salt: iodine, colorants research and labeling evaluation

ABSTRACT

Marcia Regina Pennacino do Amaral-Mello* 🝺

Jamila Barbosa 匝

Nelson Aranha Dias 🕩

Roberta Franceze Paiva Martins 厄

Regina Sorrentino Minazzi Rodrigues D

Instituto Adolfo Lutz, Centro de Alimentos, São Paulo, SP, Brasil

* E-mail: marcia.mello@ial.sp.gov.br

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Introduction: Himalayan pink salt has stood out in the Brazilian market for its attractive colors, in addition to appeals to the consumer that highlight it as a "more natural" product and with a great diversity of minerals as it is a rock salt. The iodine content in pink salt consumed in the country is still little discussed, but vital to keep lodine Deficiency Disorders (DDIs) under control. Objective: Evaluate Himalayan pink salt samples for iodine contents, presence of artificial colorants and labeling. Method: Seventy-one samples from thirteen cities of the São Paulo State were analyzed for determination of iodine added as iodate, qualitative testing colorants, and labeling evaluation based on Brazilian legislation. Results: A high rate of unsatisfactory iodine content was found (56%), even higher in salts sold in bulk (74%). It was found that among the unsatisfactory samples, the highest percentage of inadequacy (28%) was the absence of iodine, exposing the consumers of this product at risk for DDIs. The most frequent labeling irregularity was the absence of iodine declaration (46%), with contradictions between label declaration and effective presence evaluated analytically. Unforeseen (27%) or superlative (14%) quality expressions were verified, as well as claims not supported by scientific studies, such as 60% reduction in sodium and presence of 84 minerals. No sample showed colorants addition. Conclusions: Himalayan pink salt samples analyzed showed important irregularities for health of the population, especially regarding the iodine content, but also labeling inaccuracies that compromise access to correct information about the product.

KEYWORDS: Sodium Chloride; Iodination; Legislation; Iodine Deficiency

RESUMO

Introdução: O sal rosa do Himalaia tem se destacado no mercado brasileiro por sua coloração atrativa, além dos apelos ao consumidor que o destacam como um produto "mais natural" e com grande diversidade de minerais por ser um sal de rocha. O teor de iodo no sal rosa consumido no país ainda é pouco discutido, mas vital para manter sob controle os distúrbios por deficiência de iodo (DDI). Objetivo: Avaliar amostras de sal rosa do Himalaia quanto aos teores de iodo, à presença de corantes e à rotulagem. Método: Foram analisadas 71 amostras em 13 cidades do estado de São Paulo para determinação de iodo adicionado na forma de iodato, prova qualitativa para corantes artificiais e avaliação da rotulagem com base na legislação brasileira. Resultados: Um elevado índice de insatisfatoriedade dos teores de iodo foi encontrado (56%) e um percentual ainda maior nos sais comercializados a granel (74%). Verificou-se que, dentre as amostras insatisfatórias, o maior percentual de inadequação (28%) foi a ausência de iodo, colocando a população consumidora deste produto em risco para as DDI. A irregularidade de rotulagem mais encontrada foi a ausência da declaração da adição de iodo (46%), com contradições entre a declaração no rótulo e a efetiva presença avaliada analiticamente. Foram verificadas expressões de qualidade não previstas (27%) ou superlativas (14%), assim como alegações não comprovadas por estudos científicos, como a redução de 60% de sódio e a presença de 84 minerais. Nenhuma amostra apresentou adição de corante. Conclusões: O sal rosa do Himalaia analisado apresentou irregularidades importantes para a saúde da população, em especial quanto ao teor de iodo, mas também não conformidades de rotulagem que comprometem o acesso à informação correta sobre o produto.

PALAVRAS-CHAVE: Cloreto de Sódio; Iodação; Legislação; Deficiência de Iodo



INTRODUCTION

Salt is considered a universal consumer good, it was historically used as one of the most traditional forms of conservation, in addition to having as its primary function to salt food¹. Sold in different granulometries, it varies from finely ground powder to small crystals, with uniform granulation, according to the respective classification².

The world production of all types of salt in 2017 was estimated at 277 million tons, with Brazil contributing with 2.7% of this production (7.4 million tons) and still importing 757 thousand tons³.

Recently, a large number of unrefined edible salts advertised as gourmet salts have appeared in the Brazilian market, such as *fleur de sel*, Persian pink salt, Hawaiian red salt, black salt, and especially Himalayan pink salt (HPS) which showed growth in the Brazilian market in recent years.

The origin of the HPS is the mines located about 300 km southwest of the Himalayas, mainly the mines of Khewra and Kalabagh, in Pakistan⁴. Brazil is one of the main importers of this salt in South America and has increased the import of salt from Pakistan in recent years, from 17,622 tons, which corresponds to US\$ 432 thousand in 2015, to 83,270 tons, corresponding to US\$ 3.32 million in $2017^{3,5.6}$.

HPS has been highlighted for having an attractive color, pink when ground, and when in larger crystals its color can vary from transparent, white, pink or even reddish, according to the extraction site and minerals present in it^4 .

Its color and other qualities declared on the labels of this product in relation to other types of salt, highlighting it as a "more natural" product, have boosted its use both in haute cuisine and in domestic use. Claims such as reduced sodium content in relation to common refined salt and high diversity and amount of minerals, are inserted in this context, and may mislead the consumer.

As a rock salt, HPS presents variations in its composition. In a study with different portions of rock from the mine of Khewra, site of extraction of the HPS, a great variation in its purity was found, that is, the content of sodium chloride varied from 60% to 90%⁷.

The variations in concentration also affect other minerals present in greater amounts in the region called the potassium layer or Kallar, a portion of the rock where potassium can reach 9.13%, sulfates 27%, 4.13% magnesium, and 1.3% calcium⁷. Despite these variations, most of the samples from the Khewra mine studied showed a low concentration of minerals, except for chloride and sodium⁷.

Most of the 28 rock samples presented contents <1%: for potassium in 21 samples, in 26 for calcium, and in 24 for magnesium⁷. Fayet-Moore et al.⁸, evaluating commercially acquired HPS, found similar results to these in relation to mineral composition.

Despite variations in the mineral composition of HPS, special attention should be given to the amount of iodine present in the product consumed in the country. According to the Brazilian National Health Surveillance Agency (Anvisa), only salt containing 15 mg or more up to the maximum limit of 45 mg of iodine per kg of product will be considered suitable for human consumption⁹.

In terms of public health, adequate iodine intake is the most viable way to avoid important health problems caused by its deficiency, the so-called iodine deficiency disorders (IDD)^{10,11}.

Although salt can naturally contain traces of iodine, the amount is insufficient to guarantee the adequate intake established by Brazilian public iodination policies, being controlled through the lodination Monitoring Program (PMI) of salt intended for human consumption, which oversees the efficiency of iodination in the country^{12,13}.

The objective of the present study was to evaluate samples of HPS, marketed in the state of São Paulo, from 2016 to 2020, regarding the levels of iodine, artificial dyes, and labeling, in order to verify compliance with current legislation.

METHOD

From March 2016 to December 2020, 71 samples of HPS were analyzed, being 45 samples of ground salt and 26 of coarse salt, sold in establishments in 13 cities in the state of São Paulo. Among these, 19 samples were in bulk and the others from 33 different brands. The samples came from collections carried out by the Health Surveillance of different municipalities in the state of São Paulo, for fiscal analysis, referring to the monitoring of the iodine content in salt for human consumption, part of the National Program for the Prevention and Control of Iodine Deficiency Disorders (Pró-Iodo)¹², and also included samples acquired by the authors in supermarkets in the Metropolitan Region of São Paulo especially for the study.

The quantification of iodine content was performed in an automatic potentiometric titrator based on the technique of determination of added iodine in the form of iodate, described in Physicochemical methods for food analysis, Instituto Adolfo Lutz, Technique 383/IV¹⁴. 10 g of salt was weighed, diluted with 200 mL of water, adding sulfuric acid and potassium iodide, followed by titration with sodium thiosulfate solution in an automatic titrator with a platinum electrode.

Automated potentiometric titration in this case was fundamental for evaluating the end point of the titration, because the great sensitivity of the technique allows its application in very dilute solutions and also for colored solutions, such as the pink solution of dissolved HPS, which makes it difficult to visualize the turning point with the use of an indicator as used in conventional titrations.

The analyzes were performed in triplicate and the data were analyzed by descriptive statistics using mean, median,



minimum value, maximum value, and frequency, in Microsoft Excel® software.

To classify the samples as satisfactory and unsatisfactory, the concentration range from 15 to 45 mg of iodine/kg of salt was used, defined by the legislation⁹.

The presence of dyes was evaluated according to the qualitative test for dyes, described in Physico-chemical methods for food analysis, technique $051/IV^{14}$. 50 g of salt was weighed, diluted with 100 mL of water, white natural wool was inserted into the beaker and hydrochloric acid was added. It was heated in a boiling water bath and, after washing the wool in running water, it was verified whether there was color impregnation in the wool. This analysis was necessary to verify the authenticity of the salt color and to avoid possible falsifications with dyes that could be added to the product.

The labeling evaluation was carried out based on the following Brazilian regulations: Federal Decree No. 75,697, of May 6, 1975²; Anvisa's Resolutions of the Collegiate Board of Directors (RDC): No. 259, of September 20, 2002¹⁵, No. 359, of December 23, 2003¹⁶, No. 360, of December 23, 2003¹⁷, No. 269, of September 22, 2005¹⁸, No. 54, of November 12, 2012¹⁹, Decree-Law No. 986, of October 21, 1969²⁰, and Federal Law No. 10.674, of May 16, 2003²¹.

RESULTS AND DISCUSSION

Table 1 presents the results of the iodine levels found in the HPS samples, classified according to the limits established in the Brazilian legislation⁹.

During the period (2016-2020), 71 samples were analyzed (Table 1), of which 31 (44%) were satisfactory, within the iodine range established by legislation, and 40 (56%), unsatisfactory. In the PMI, published in 2019, Anvisa found a lower percentage of dissatisfaction: 46% of HPS samples were unsatisfactory²².

Silva et al.²³ found a similar percentage (53.8%) of inadequate iodine content in a total of 13 HPS samples collected in the interior of the state of São Paulo. Loyola²⁴ found an even higher percentage of inadequacy in 58% of the 12 samples evaluated in Goiás. The PMI reports released by Anvisa in the last 10 years showed a percentage of adequacy of the iodine content in all salts: between 93 and 97%. However, in 2019, this percentage dropped to 88%, due to the large number of unsatisfactory HPS samples 22 .

In our study, it was found that, among the unsatisfactory samples (56%), the highest percentage of inadequacy (28%, n = 20) was related to the absence of iodine, that is, non-fortified samples, followed by samples with iodine content lower than 15 mg/kg (21%, n = 15) and, finally, higher than 45 mg/kg (7%, n = 5).

Ramos²⁵ evaluated the iodine content in six HPS samples (fine, coarse, and bulk), acquired in Campo Mourão (Paraná) and found no iodine in any of the analyzed samples.

Figure 1 demonstrates the trend towards a reduction in dissatisfaction over time. At the beginning of the collections (2016), all HPS samples (100%) were unsatisfactory, with a gradual decrease in unsatisfactory over the years, 55% in 2018 and 40% in 2020.

Also, in common and refined salts, the iodine content evaluated over longer periods (1999-2013) showed the same type of behavior, ie, a decrease in dissatisfaction over time²⁶.

Probably, this fact is due to the greater effort of the importing industries in the control of iodine fortification, as a result of the inspection generated by the PMI¹².

The high percentage of samples not added with iodine in this study (28%) constitutes the greatest risk to the health of the population. Choosing this type of salt, in the search for a more natural and healthier product, may affect the daily consumption of iodine. In the long term, this scenario can put consumers of these products at risk for the development of IDD, especially pregnant and lactating women, due to the greater demand for iodine by the body^{27,28}.

In addition, salts sold without the addition of iodine, or that do not contain iodine at the established levels, are in disagreement with the requirements defined in the following legislation: Federal Law No. 6,150, of December 3, 1974²⁹, art. 17 of Federal Decree No. 75.697/1975² and art. 5 of RDC No. 23, of April 24, 2013⁹.

Table 1	Indine contents	$(m\sigma/k\sigma)$ in	Himalayan	nink salt samples	sold in the state	of São Paulo	from 2016 to 2020
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Year	Samples	< 15	15 ≤ [lodine]≤ 45	> 45	ND*	Satisfactory		Unsatisfactory	
	No.	mg/kg	mg/kg	mg/kg	No.	No.	%	No.	%
2016	3	1	0	0	2	0	0	3	100
2017	16	4	6	2	4	6	38	10	62
2018	29	4	13	0	12	13	45	16	55
2019	13	2	6	3	2	6	46	7	54
2020	10	4	6	0	0	6	60	4	40
TOTAL	71	15	31	5	20	31	44	40	56

Source: Elaborated by the authors, 2021.

ND*: not detected.



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Source: Elaborated by the authors, 2021.

Figure 1. Percentage of samples of Himalayan pink salt, satisfactory and unsatisfactory, in relation to iodine content, in the period from 2016 to 2020.



Source: Elaborated by the authors, 2021.

Figure 2. Distribution of iodine contents (mg/kg) of pink salt samples, marketed in the state of São Paulo, analyzed from 2016 to 2020, based on the legal limits of 15 to 45 mg/kg.

It is prohibited, throughout the national territory, to expose or deliver for direct consumption common or refined salt that does not contain iodine at the levels established in an Ordinance of the Ministry of Health⁹.

The only exception for the use of salt without the addition of iodine provides for the use of non-iodized salt in industrialized food products, provided that iodine is proven to interfere with the sensory characteristics of the product⁹.

Among the samples that were iodized (72%), the minimum iodine content found was 1.17 mg/kg and the maximum, 61.52 mg/kg, with a median of 15.06 mg/kg.

In Figure 2, we can see that the iodine levels in the HPS samples are very dispersed and, when presented satisfactorily, are in the concentration range between 18 and 35 mg/kg.

The fact that the maximum limit contemplates three times the minimum limit of iodine facilitates the iodination process by the salt industry, which seeks to reach iodine values in the middle of the established range.

A major contradiction was observed between the information on the label and the effective addition of iodine, analytically evaluated by titration (Chart). Despite the mandatory addition of iodine and the need for this information to be clear on the



Chart. Contradictions between the levels of iodine declared on the label and those determined in Himalayan pink salt.

	lodine declaration	lodine detection
		n = 6 (8%) lodine not detected.
n = 38 (54%)	Declare iodine in nutritional information	n = 16 (23%) lodine detected with variability between declared and found levels from 27% to 222%
Dectare logine	n = 16 (23%)	n = 9 (13%) lodine not detected.
	Declare iodine in the labeling	n = 7 (10%) Iodine detected
n = 33 (46%)		n = 11 (15%) lodine not detected.
Don't declare iodine	-	n = 22 (31%) lodine detected

Source: Elaborated by the authors, 2021. Note: Percentages of total samples (n = 71).

labeling, the information contained on the label must match the characteristics and composition of the product, at the risk of inducing the consumer to error or mistake.

Among the 33 different brands evaluated, seven were collected and analyzed more than once and showed variability in iodine content between different batches. These results demonstrate lack of standardization in the iodination process. For example, a brand with three batches analyzed showed two satisfactory results (25.40 and 39.80 mg of iodine/kg) and one unsatisfactory (51.26 mg of iodine/kg), revealing a coefficient of variation between batches of 33%.

Salts sold in bulk had a higher percentage of dissatisfaction (74%) compared to branded samples (48%), bulk salts usually have a lower cost, however, according to the results presented, they showed less care with iodination by the producers and traders.

All samples showed no artificial or natural dyes. Although they were not found, the search for dyes was important due to the fact that the high cost of this product⁸ could encourage fraud.

These results confirm preliminary tests that showed that the HPS solution, when filtered, is clear, and the color is maintained in the residue retained on the filter paper, generated by the mineral compounds present in the salt²⁵.

In the evaluation of the labeling of salts, several irregularities were found based on the legislation applied. Table 2 presents the most frequently found labeling irregularities, with several samples showing more than one nonconformity.

The most common labeling irregularity was the absence of a declaration of the addition of iodine in 33 (46%) samples, of which 22 contained iodine in the required concentrations.

Only one sample (1%) incorrectly declared the unit of iodine content in the nutritional information, 15 milligrams of iodine instead of micrograms, which corresponds to a thousand times the correct concentration. This could lead the consumer to error

or mistake in comparison with other labels (RDC No. 360/2003; RDC No. 269/2005)^{17,18}.

The practice of not declaring the presence of iodine may have the purpose of inducing the consumer to believe that the product is totally natural and without added chemical substances.

The high percentage of unforeseen quality expressions, 19 (27%), among them "100% natural", reinforces this intention. The use of other expressions such as premium and gourmet also lead to the mistaken idea of a superior quality product, without there being a quality standard provided for in the legislation. Probably with the same intention, ten samples (14%) used superlative expressions of quality such as: "the purest" and "the oldest in the world".

The stimulus to consumption generated by food advertising was confirmed by Cuevas et al.³⁰, evaluating articles from specialized magazines for the food industry from 2007 to 2018, verified a strong tendency to invest in promotion and advertising, to the detriment of other types of investments, such as: product formulation, packaging, or equipment to make food healthier.

In addition, more vague and less verifiable claims such as "natural ingredients" were considered potentially profitable³⁰. Several of these claims were featured on HPS labels, such as: "healthy salt", "purer", "preservative-free", and "nutritionally balanced". These claims are likely to present HPS as a "more natural" or less processed product and with additional health benefits, without proper evidence through scientific studies.

Even though it is a new product on the market, HPS falls within the Technical Regulation of salt for human consumption, including in relation to its granulometric classification. However, 27 (38%) samples adopted unforeseen classifications, such as: fine, medium, and granulated, in disagreement with the standardization established in Decree n° 75.697/1975².

Among the irregularities, 18 (25%) samples presented some claim about mineral content, using terms related to quantity, diversity, or even citing some chemical elements in the labeling, such as copper, zinc, and calcium.



Table 2. Number and percentage of samples with information on the labeling in disagreement with current legislation.

Information in disagreement on the labeling	Legislation	Unsatisfactory samples No. (%)
There is no declaration of iodine addition	Federal Decree-Law No. 986/1969, art. 20 RDC No. 23/2013	33 (46)
Unforeseen classification indication: fine, medium, and granulated	Decree No. 75,697/1975	27 (38)
Expressions of unforeseen quality: 100% natural, premium, gourmet	Federal Decree-Law No. 986/1969, art. 22 RDC No. 259/2002, item 3	19 (27)
Mineral claims: rich in minerals, 84 minerals, and more	CNI not foreseen for salt RDC No. 54/2012, item 1.4	18 (25)
Indication of origin versus denomination	Federal Decree-Law No. 986/1969, art. 21 RDC No. 259/2002, item 3.2	12 (17)
Superlative expression of quality: purest, oldest in the world	Federal Decree-Law No. 986/1969, art. 20	10 (14)
Other statements such as: free of pollutants and toxins, nutritionally balanced, and healthy salt	RDC No. 259/2002, items 3.1 and 7.1	8 (11)
Sodium reduction: 60% less sodium and/or low sodium content	CNI not foreseen for salt RDC No. 54/2012, item 1.4	5 (7)
Use of the expression: no preservative	Federal Decree-Law No. 986/1969, art. 21 RDC No. 259/2002, item 3 Anvisa Technical Report No. 70/2016	3 (4)
Gluten: use of expression other than the standardized one	Federal Law No. 10,674/2003	2 (3)
Incorrect unit of iodine declared in the nutritional information	RDC No. 269/2005 RDC No. 360/2003	1 (1)

Source: Elaborated by the authors, 2021.

Note: Some samples showed more than one nonconformity.

RDC: Resolution of the Collegiate Board of Directors; Anvisa: Brazilian National Health Surveillance Agency; CNI: complementary nutritional information

The declaration of nutritional properties of a food, such as the declaration of minerals, is carried out through the complementary nutritional information (CNI), regulated by RDC No. 54/2012¹⁹. However, this regulation does not apply to salt and, therefore, in addition to being unforeseen, these claims are misleading, as different studies carried out to date have not identified the diversity mentioned on the labels of 80 or 84 minerals^{7,8,31,32}. In addition, the low concentration of minerals present in the HPS does not present a relevant contribution to the daily needs, based on the recommended salt intake of 5 g daily^{8,11}.

Regarding the reduction of sodium, 7% of the samples were highlighted on the label for a reduction of 60% or "low sodium content", however, this type of claim, according to RDC No. 54/2012¹⁹, does not apply to salt.

For salts with reduced sodium, the so-called hyposodic salts, there is a specific technical regulation: the Ordinance of the Health Surveillance Secretariat/Ministry of Health (SVS/MS) No. 54, of July 4, 1995³³, which defines the use of the declaration of reduced sodium content, in this case, as mandatory and not voluntary.

In addition, studies do not prove such an expressive reduction and labeling should not induce the consumer to error, deceit, or confusion, according to RDC No. 259/2002¹⁵.

Although the sodium content in HPS rocks can vary from 60% to 99%, with the lowest concentration in the region of the potassium layer, most studies with commercial HPS have not found such an extensive variation^{7,8,34}. Regarding the concentration of sodium in the HPS, the levels in different studies ranged from 34,389 mg/100 g and 45,912 mg/100 g, while in refined salt, in the same studies, concentrations varied between 35,870 and $42,764 \text{ mg}/100 \text{ g}^{8,34,35,36}$. Regarding refined salt, research has found a decrease in sodium between 1% and 9%, a much smaller reduction than what is stated on labels^{8,24,35,37}.

It was observed that 12 samples (17%), despite having the sales denomination "Himalayan pink salt", did not clearly indicate the geographical origin of the salt. All packaged food must have its origin described on the label, as established in RDC No. 259/2002, item 3.2, and in Federal Decree-Law No. 986/1969, art. 21^{15,20}.

The geographical indication used in the denomination "Himalayan pink salt" refers to its origin, however some of these samples presented as origin "Brazilian industry" or even "Africa", not clarifying whether the product was imported, where it was imported from, or if it was just processed in another country, which makes this information confusing and subject to errors.

The various claims found on the labeling are probably marketing strategies used to attract the consumer and lead to greater commercialization of the product, because, as seen, they are not provided for in the salt legislation and may contain incorrect information that confuses the consumer.

CONCLUSIONS

The HPS samples sold in the state of São Paulo, from 2016 to 2020, showed a high rate of unsatisfactory iodine levels (56%),



much higher than those found in refined and common salts, and an even higher percentage was found in pink salts sold in bulk (74%). Several irregularities were also found in relation to the labeling of these products, most of them related to the absence of the declaration of addition of iodine, to the classification, to expressions of unforeseen quality, and to declarations about the content of minerals, conditions that may induce the consumer into error, deception, or confusion. No samples were observed with the addition of artificial dye. These data demonstrate that the continuity of monitoring of this product through programs such as Pró-lodo and other government programs is essential to ensure that all salt for human consumption meets the specifications established in the respective technical regulations, aiming at protecting the health of the population.

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Author's Contributions

Amaral-Mello MRP- Conception, planning (study design), acquisition, analysis, data interpretation, and writing of the work. Barbosa J -Planning (study design), acquisition, analysis, data interpretation, and writing of the work. Dias NA - Data acquisition and analysis. Martins RFP - Data acquisition. Minazzi Rodrigues RS - Analysis, data interpretation, and writing of the work. All authors approved the final version of the work.

Conflict of Interests

The authors inform that there is no potential conflict of interest with peers and institutions, politicians, or financial in this study.



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