



ORIGINAL ARTICLE

Determination of Magnesium, Calcium and Sulphate Ion Impurities in Commercial Edible Salt

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KEYWORDS

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ABSTRACT: Natural elemental impurities are recognized as a threat for safety and quality of edible salt and have adverse effects on public health. In the current study, fifty samples of packages containing 1 kg of salt from 25 different brands were collected from retailers in Semnan city (Iran). The concentrations of main impurities of edible salt including magnesium (Mg), calcium (Ca), and sulphate (SO_4^{2-}) ions were quantified by the aid of an Ion Chromatography with conductivity detector. According to findings, the maximum concentrations of Mg, Ca, and SO_4^{2-} ions in salt samples were 0.067, 0.226, and 0.888 % w/w (dry matter basis), respectively. In addition, the concentration of Mg in 16%, Ca in 4%, and SO_4^{2-} in 28% of samples suppressed the acceptable limit proposed by the Institute of Standards and Industrial Research of Iran (ISIRI) (0.15% for Ca, 0.03% for Mg, and 0.46% for SO_4^{2-}). Moreover, the maximum and minimum levels of purity in the salt samples were recorded as 99.940 and 97.730%, respectively. Moreover, the purity in 12% of the investigated samples was lower than that of the minimum acceptable limit suggested by ISIRI, while the purity of 97% samples met the acceptable Codex Alimentarius limit (97% Min). Based on results of the current investigation, the routine purification processes used in some factories of Iran did not reduce impurities. Hence, purification process besides constant monitoring and safety management should be improved to promote the health quality of edible salt.

INTRODUCTION

Salt is one of the most prevalent and legendary consumed minerals in the world due to its special place in food and vital role in human life [1, 2] while about 30% of the total produced salt is consumed to supply physiological and diet necessities [3]. In 2017, the world salt production was 280 million tons

(Statistica, 2019). The leading salt producer in the worldwide scale was China (58 million tons), and the United States, India, Germany, and Canada are some of the world's leading salt producing countries. Iran produced about 2.800 million tons of salt annually which translate to 1.1% of the world's total salt

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production [4]. Edible salt as a crystalline product predominantly includes sodium chloride (NaCl) extracted from the sea, underground salt rock, or natural brine [5, 6]. In fact, the existence of impurities and their relative levels influence the salt properties [7-9]. Since the edible salt has different health related applications such as an ingredient of food, food manufacturing, food additives and as a carrier of nutrients (i.e. iodine), its chemical composition, quality factors, NaCl content, secondary products and contaminants must be measured and monitored [5, 10]. Naturally secondary products which are generally made of calcium, potassium, magnesium, sodium sulphates, carbonates, bromides, calcium, potassium, and magnesium chlorides may present in different levels in edible salt depending on the origin and the method of salt manufacturing [5]. Some of these ions either enter the crystalline sodium salt or accumulate on the surface of salt crystals, resulting in further contamination of salt as impurities [11-13]. The presence of these impurities in salt pose some negative environmental and economic impacts [1, 12]. The impurities cause moisture absorption and cracking of the salt product, making it difficult to use while considered as a defect in salt quality [11]. On the other hand, the increase in impurities leads to decrease in purity of salt, which could diminish the salty taste of salt. This matter indirectly might increase the consumption/intake of salt [14]. In addition, the presence of sulphate in the form of calcium sulphate, which is insoluble, resulted in further precipitation in food and undesirable appearance [2, 11, 15]. However, calcium sulfate is the main structure naturally found with sodium chloride in environments; due to its low solubility in water, it probably crystallizes alongside with salt. Furthermore, calcium sulfate, as a noticeable risk factor for stomach cancer, can be ingested through high consumption of salted food (contaminated with calcium sulfate and /or other impurities) [16-18]. However, most of literatures concerning the safety of edible salt are focused on iodide and heavy metal determination [1, 10], all impurities have to be reduced during purification process up to acceptable standard level. The ion chromatography is a simple, quantitative, fast, and accurate method for the determination of iodate and sulphate in common iodized salt [19].

The accretion of food safety awareness of consumers leads to demand of pure products [20]. As one of the raw food stuff, salts must be produced with stringent specifications; otherwise, the safety of product can get affected. Salt upgrading program helps manage the contaminants problems [7, 21]. Nowadays, edible salt has been marketed in countries internationally. Therefore, reliable methodologies are needed to identify and evaluate salt specification. A few efficient indicators have been recommended for salt for its good consumption.

Therefore, in order to monitor the chemical safety of edible salt or table salt distributed in Iran, the current investigation was aimed at measuring levels of major impurities of salt including sulphate (SO_4^{2-}), calcium (Ca) and magnesium (Mg) with the aid of an ion chromatography as an efficient analytical method for the separation and quantification of various ionic compounds.

MATERIALS AND METHODS

Salt samples

Semnan province (longitude $53^\circ, 23'$, latitude $35^\circ, 34'$ with hot and dry climate) is considered as one of the most important regions of industrial production of salt in Iran with about 22 salt purification factories. Fifty edible salt samples in 1 kg packages from 25 different brands with the issued licenses by the Ministry of Health and Medical Education of Iran recognized as edible salt were purchased from supermarkets in Semnan, Iran (from March to July 2018). Regarding the ethics committee approval, the names of brands were not stated in this study.

Reagents

The used analytical grade chemicals including tartaric acid, citric acid, ethylenediamine, acetone, sodium carbonate, sodium hydrogen carbonate, sodium perchlorate, and nitric acid were obtained from Merck (Darmstadt, Germany). Magnesium, calcium, and sulphate ions standard stock solution (1000 mg/L, Accustandard, Connecticut, USA) were prepared by dissolving the determinate amount of standard solution in deionized water. Salt solution samples (1% w/v) were prepared by dissolving 1g of salt sample in deionized water and making up to 100 ml.

Ion chromatography (IC)

The professional IC system (850 Metrohm, Switzerland) equipped with 858 professional sample processor was used for salt sample analysis. The column used for cation was Nucleosil 5SA 125/ 4 with flow rate of 1.5 ml/min and anion column was Metrosep A supp 10-100/4 with flow rate of 1 ml/min. The eluent solvent containing a mixture of 5mM sodium carbonate, 5mM sodium hydrogen carbonate, and 5 μ M sodium perchlorate was conducted for anion and 4 mM tartaric acid, 0.5 mM citric acid, 3 mM ethylene diamine, and 5% acetone were used for cations. Both eluent solvents were prepared in deionized water (total volume 2L). The temperature was 40°C. The anion (SO_4^{2-}) and cations (Mg and Ca) were detected using a conductivity detector 2 (850 professional IC 1). Compiling the data was performed by Magic Net 2.3 software. The limit of detection (LOD) was 0.002 mg/L based on 100 μ l injection volume [19].

Calibration

Prior to sample analysis, the calibration curves were obtained. The standard solution concentrations for preparing calibration curves were in the range of 100, 200, 500, 1000 mg kg^{-1} (for SO_4^{2-}), and 4, 8, 12, 20 mg kg^{-1} (for Mg), and 5, 12, 19, 25 mg kg^{-1} (for Ca).

Purity assay

Purity of salt samples was specified using a Potentiometer Titrator (Titrand 906 Metrohm, Switzerland). A salt sample

(60 g) was placed in titration vessel, then 60 ml deionized water, and 1 ml nitric acid (2 N) were added. The sample solutions were titrated by Ag electrode. The total solid content (purity) of samples was measured by Tiamo software (Version 2.2) of Potentiometer Titrator apparatus.

Statistical analysis

The analysis of all samples was done in triplicate and the data were reported as mean values. ANOVA was conducted to analyze the variance using SAS (version 9.4, SASS Institute, North Carolina State, USA) software. Moreover, the significance level was considered 95% ($p < 0.05$). Principle component analysis (PCA) was used to explore the correlation of the measured parameters [22, 23]. The PCA were carried out to establish which salt quality parameters were more associated to the content of the main impurities of edible salt and whether they permit to establish a more precise distinction between main impurities and the concentrations of magnesium (Mg), calcium (Ca), and sulphate (SO_4^{2-}) ions. The statistical analyses were performed using SPSS software (Version 18.0; SPSS Inc., Chicago, Illinois, USA).

RESULTS AND DISCUSSION

Figure 1 shows the chromatograms of standard solution containing 100 mg kg^{-1} SO_4^{2-} ion (Figure 1.a) and standard solution containing 8 mg kg^{-1} Mg ion and 12 mg kg^{-1} Ca ion (Figure 1.b)

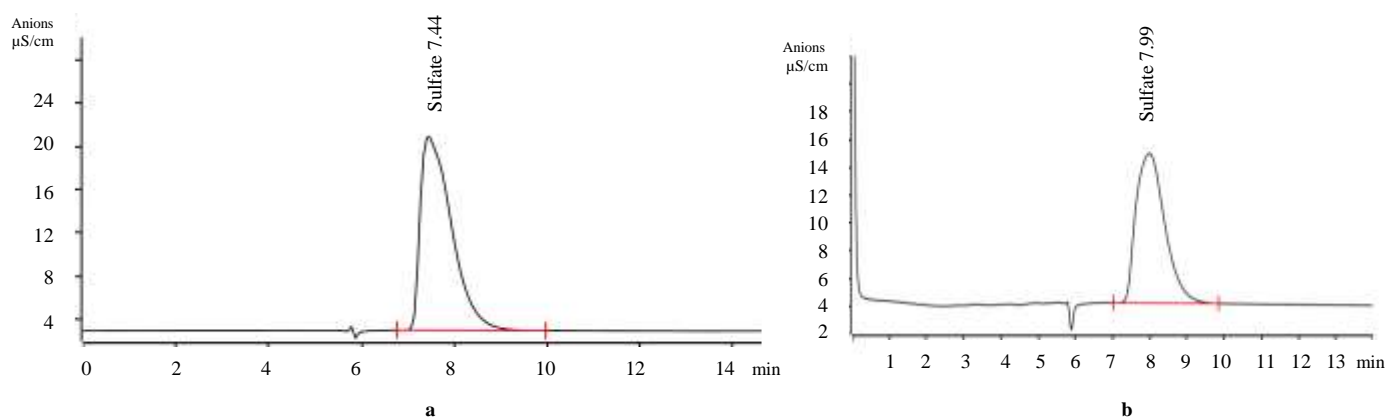


Figure 1. Chromatogram of standard solution containing 100 mg kg^{-1} SO_4^{2-} ion (a), salt sample containing SO_4^{2-} ion (b).

Figure 2(a) shows the chromatograms of an example of salt sample containing SO_4^{2-} ion that exceeded the acceptable limit set by Iran (0.46 %). Figure 2(b) also shows an example of salt

sample containing Mg and Ca ions that exceeded the acceptable limit set by Iran (0.03 % and 0.15 %, respectively) (Figure 2.b).

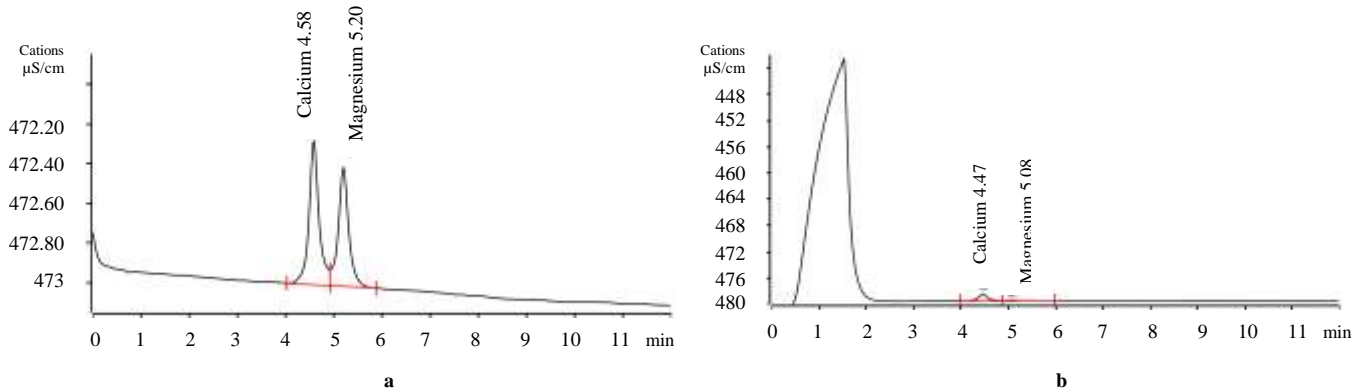


Figure 2. Chromatogram of standard solution containing 8 mg kg^{-1} Mg and 12 mg kg^{-1} Ca ions (a), salt sample containing Mg and Ca ions exceeded acceptable limit (b).

The mean concentration of Mg, Ca, and SO_4^{2-} ions and purity of salt samples with different brands were summarized in Table 1. The maximum and minimum levels of determined ions were recorded as 0.067 and 0 % for Mg, 0.226 and 0% for Ca, and 0.888 and 0.013 % for SO_4^{2-} . Moreover, a range from 97.730 to 99.940% was obtained for purity assay in salt samples (Table 1). Figure 3 demonstrated the effects of Mg, Ca, and SO_4 ions variation on salt purity. As result of increasing Mg, Ca, and

SO_4^{2-} ions concentration, the purity of salt reduced. Variation of salt purity is demonstrated in high gradient of the curve (Figure 3.a, b, c) indicating that the slight presence of ions had a significant effect on salt purity. In fact, the edible salt with lower purity ($p < 0.05$) significantly contained more impurities such as Mg, Ca, and SO_4^{2-} ions. It also seems that in different brand names of salt, the purity depended on the Mg, Ca, and SO_4^{2-} content.

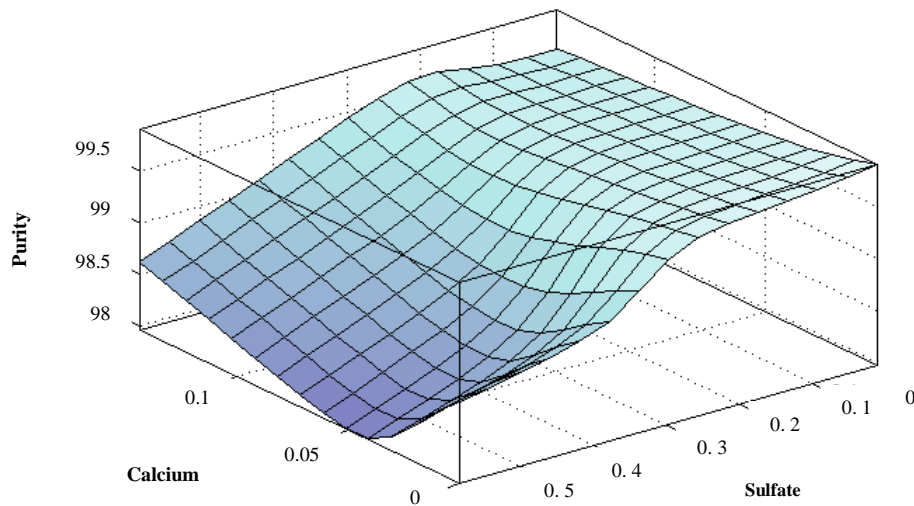


Figure 3. The effect of Magnesium, Calcium, and Sulphate ion contents on purity of salt.

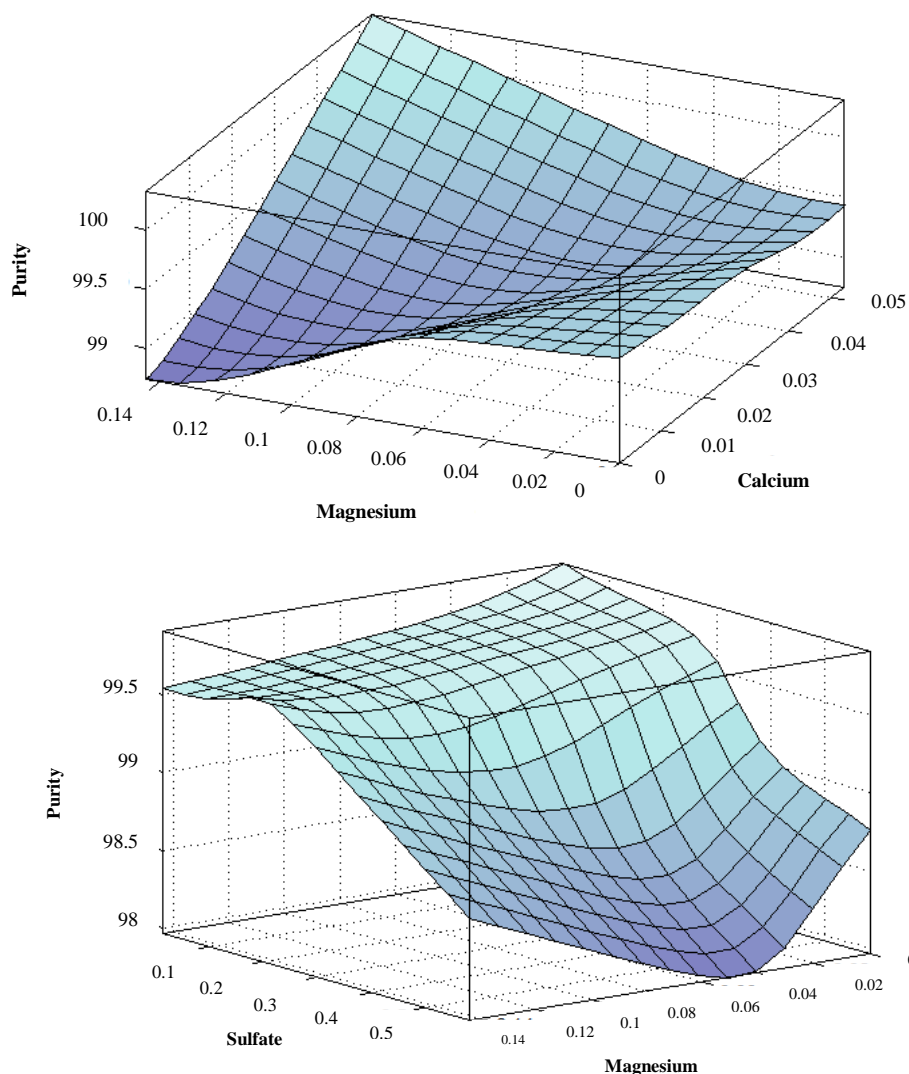


Figure 3. Continued.

Salt had major impurities that must be eliminated during efficient purification. Since the edible salt should meet the mandatory standard, the maximum acceptable limit for food grade salt specifications has been proposed by Codex Alimentarius and Institute of Standards and Industrial Research of Iran (ISIRI). The acceptable limit of Mg, Ca, and SO_4^{2-} ions and purity are presented in Table 2 [5, 24]. According to the results, the levels of Mg, Ca, and SO_4^{2-} ions in 16, 4, and 28% of edible salt samples were more than acceptable limit of ISIRI. Furthermore, the purity in 12% of the samples was lower than minimum acceptable limit of Iran. However, all samples were in the recommended ranges by Codex Alimentarius. There is no reported data about determination of these ions in edible salt, neither from Semnan nor from other cities of Iran. However, our research showed that only 15 brands of evaluated salt (60% of samples) had Mg, Ca, SO_4^{2-} , and purity lower than

maximum limit suggested by ISIRI; while, 10 brands of assayed edible salt were not in accordance to ISIRI. It is obviously found that clarification and purification have not been carried out efficiently in producer factories. In respect of the origin of salt manufacturing, it is observed that 20 brands were manufactured in factories located in Semnan province and five of them were produced in other provinces of Iran (Table 1). However, three brands of salt produced in other provinces of Iran failed to meet the required criteria proposed by ISIRI. Therefore, it seems that mineral impurity is not only related to salt producer of Semnan province.

In the samples, the first component and the second component calculate 48.8% and 23.3% of the total variability, respectively. The contribution of each of the parameters in two factors and distribution of brands in plot are shown in Figure 4.

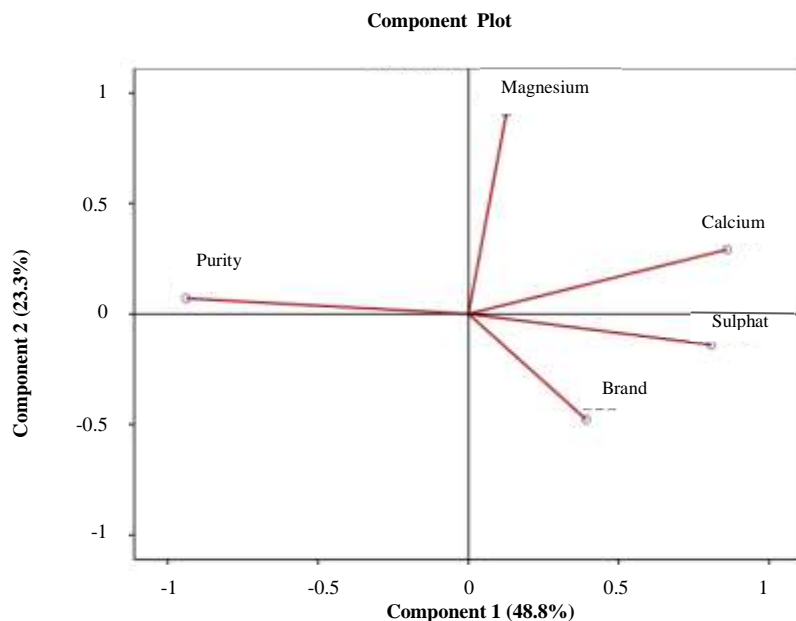


Figure 4. Biplot of the PCA for edible salt.

The concentrations of calcium and sulphate in brands had a positive, high, and significant correlation with PC1 and purity had negative correlation coefficients. The results show that magnesium and calcium were positively correlated with PC2, and sulphate; and brand had negative correlation coefficients. The results obtained from the correlation coefficients confirm the results of the analysis of the main components.

Natural salt known as unrefined salt has been extracted mechanically from salt mines[11]. Brine is a saturated solution

of water and extracted salt that must be evaporated to be prepared for crystallization. Existence of impurities could affect the formation of the crystal of salt. Even the chemical component of unrefined salts can be considered as authentic fingerprint for geographically discriminating the origin of salts [25]. Among the industrial salt purification methods primarily performed in Semnan, the re-crystallization method reserved a notable rank. The process of this method is shown in Figure 5.

Table 1. The percentage of Mg, Ca, and SO_4^{2-} ions in salt samples with different brand name quantified using Ion chromatography with electrical conductivity detector.

Brand of table salt	Origin of salt production	Mg (%)	Ca (%)	SO_4^{2-} (%)	Purity (%)
1	Semnan province	0.004±0.0003 ^f	0.019±0.0007 ^{ghi}	0.534±0.049 ^b	99.558±0.120 ^{fg}
2	Semnan province	0.007±0.012 ^{def}	0 ⁱ	0.014±0.002 ^f	99.9±0.031 ^{ab}
3	Semnan province	0.008±0.0004 ^{def}	0.004±0.0018 ⁱ	0.037±0.002 ^f	99.867±0.120 ^{abc}
4	Semnan province	0.003±0.000 ^f	0 ⁱ	0.051±0.004 ^f	99.862±0.063 ^{abc}
5	Semnan province	0.006±0.0018 ^{ef}	0.024±0.0027 ^{fghi}	0.49±0.077 ^b	99.517±0.110 ^g
6	Semnan province	0.001±0.0002 ^f	0.049±0.0029 ^{def}	0.13±0.018 ^{ef}	99.710±0.061 ^{cdef}
7	Semnan province	0.047±0.0038 ^b	0.052±0.0032 ^d	0.21±0.04 ^{de}	99.272±0.012 ⁱ
8	Semnan province	0 ^f	0.015±0.0029 ^{ghi}	0.022±0.001 ^f	99.880±0.017 ^{ab}
9	Semnan province	0.053±0.0047 ^b	0.025±0.0002 ^{defghi}	0.302±0.034 ^{cd}	99.565±0.008 ^{fg}

Table 1. Continued.

10	Semnan province	0.028±0.0006 ^c	0.036±0.0114 ^{defg}	0.106±0.001 ^{ef}	99.690±0.059 ^{efid}
11	Semnan province	0.016±0.0004 ^d	0.032±0.003 ^{defgh}	0.129±0.025 ^{ef}	99.761±0.046 ^{bcd}
12	Semnan province	0 ^f	0 ⁱ	0.013±0.002 ^f	99.759±0.134 ^{bcd}
13	Semnan province	0.008±0.0009 ^{def}	0.02±0.0013 ^{ghi}	0.352±0.077 ^c	99.478±0.021 ^{gh}
14	Iran	0 ^f	0.144±0.0509 ^b	0.584±0.058 ^b	97.732±0.0392 ^{bcde}
15	Semnan province	0.067±0.0135 ^a	0.226±0.0197 ^a	0.888±0.204 ^a	99.216±0.026 ⁱ
16	Semnan province	0 ^f	0.014±0.0025 ^{ghi}	0.206±0.035 ^{de}	99.589±0.112 ^{efg}
17	Semnan province	0 ^f	0.007±0.0004 ^{hi}	0.14±0.030 ^{ef}	99.773±0.111 ^{abcd}
18	Iran	0.009±0.0005 ^{def}	0.051±0.0037 ^{de}	0.544±0.051 ^b	98.009±0.028 ^j
19	Iran	0.05±0.0025 ^b	0.09±0.0092 ^c	0.272±0.046 ^{cd}	98.701±0.039 ^j
20	Semnan province	0 ^f	0 ⁱ	0.025±0.002 ^f	99.943±0.0343 ^a
21	Semnan province	0.003±0.0002 ^f	0.009±0.0071 ^{ghi}	0.095±0.011 ^{ef}	99.840±0.051 ^{abcd}
22	Semnan province	0.014±0.009 ^{de}	0.078±0.0024 ^c	0.47±0.058 ^b	99.360±0.008 ^{hi}
23	Iran	0 ^f	0.013±0.0007 ^{ghi}	0.033±0.004 ^f	99.870±0.140 ^{abc}
24	Semnan province	0 ^f	0.05±0.002 ^{def}	0.521±0.07 ^b	99.351±0.001 ^{hi}
25	Iran	0 ^f	0 ⁱ	0.089±0.012 ^{ef}	99.901±0.046 ^{ab}

Table 2. Acceptable limit of Mg, Ca, and SO₄²⁻ ions and impurity of food grade salt.

Chemical specification	ISIRI	Codex Alimentarius	Percentage of samples exceeded acceptable limit (ISIRI)
Calcium (as Ca)	0.15 % * Max	Depending on the origin and method of production of the salt	4
Magnesium (as Mg)	0.03 % Max	Depending on the origin and method of production of the salt	16
Sulphate (as SO ₄ ²⁻)	0.46 % Max	Depending on the origin and method of production of the salt	28
Purity (as NaCl)	99.2 Min	97% Min	12

*measured according to dry basis

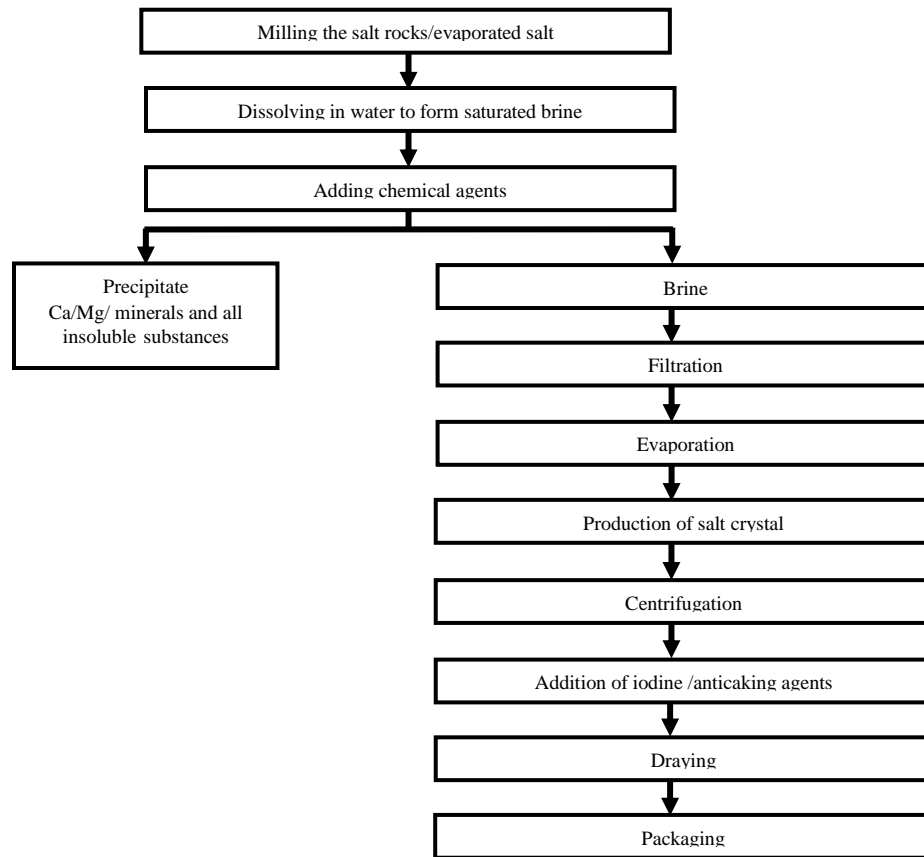


Figure 5. Industrial salt purification based on re-crystallization method.

The results of present study may indicate that some elements of rock salt could have entered in final edible salt and consequently entered in consumers' salt table.

There are two kinds of natural impurities in all crystalline deposits such as rock salt. The concentration of them depends on the salt origin and production method. Generally, these impurities include:

- 1) Surface crystal impurity: soluble (Mg, Ca, and K salt, etc.) and insoluble compounds (sand, clay, etc.)
- 2) Inter crystal impurity: soluble and insoluble compounds due to imperfect crystallization process

In salt production plant, the first category of impurities could be reduced and eliminated by mechanical process like washing with water. However, the second one can be eliminated during dissolving and recrystallizing the salt.

Based on our results, it appears that the separation of impurity had not been efficiently done in some factories. It seems that the dissolving and crystallization processes of salt production

had not efficiently done by some manufacturers in Iran. Nowadays several alternative methods are used in salt industry to produce high purity edible salt. In modern recrystallization process, a series of flash crystallizers with varied pressure levels accompanied by adiabatic flash evaporation could result in production of high vacuum salt quality. Furthermore, it is recommended to upgrade the present technology in Iran to diminish impurities and reaching high purity of edible salt.

Different researches have been conducted all over the world about product's contamination that some of which have the same results as the present work. Rehan et al. (2018) in Pakistan measured the toxic and essential metals in rock and sea salts; they reported the mean levels of the Ca, Mg, and S in sea salt, industrial grade and edible salt were 420, 230, 210 and 400, 200, 200 and 200, 310, 150 ppm, respectively [26]. Furthermore, the elemental composition of salt and their quantities could reflect the efficiency of purification process in

factory [19]. In this regard, the heavy metal concentration in edible salt was determined using atomic absorption spectrophotometry in Tehran (Iran) [10] while the content of aluminum, cadmium, and Iron in packed edible salt produced by Iran factories exceeded the maximum acceptable level of Codex Alimentarius and ISIRI. It is necessary for good manufacturing practice (GAP) to be observed in any production process to have high quality salt.

CONCLUSIONS

In summary, in our study, evaluation of edible salt samples produced in Iran regarding Mg, Ca, SO_4^{-2} and purity showed that the concentration of these impurities in some brand names were not according to acceptable limit of Institute of Standards and Industrial Research of Iran. In the employed PCA analysis, the chosen characteristic of concentrations of Mg, Ca, SO_4^{-2} ions, purity, and brand names were proved to be effective in evaluating the quality of edible salt. Monitoring these chemicals was studied for the first time on edible salt produced in Iran and indicated that the present used recrystallization process need upgrading in order to produce high quality salt. The Mg, Ca and SO_4^{-2} ions are considered as salt contaminant and as critical defects in final product. Implementation of risk assessment on public health regarding presence of impurities in salt is more important and is strongly recommended.

Ethics Approval

The study project had approved by the research ethics committee of Semnan University of Medical Sciences (approval ID: IR.SEMUMS.REC.1397.060).

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Conflicts of interest

All authors have declared that they do not have any conflict of interest for publishing this research.

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