



Assessment of ferrocyanide intake from food-grade salt in the Korean population



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ARTICLE INFO

Keywords:

Ferrocyanides
HPLC
Estimated daily intake
Acceptable daily intake
Food-grade salt

ABSTRACT

A simple high-performance liquid chromatography-ultraviolet (HPLC-UV) method for estimating the daily intake of ferrocyanide (FeCN) ion from food-grade salts was developed and validated. The levels of FeCN ion in food-grade salts available in Korean markets were analysed. FeCN ion was detected in 8.9% of 801 samples, with concentrations ranging from ND (not detected) to 9.6 mg/kg. The FeCN-ion levels were below the legal limit (10 mg/kg) specified by the Korean Food Code. The FeCN-ion intake of the Korean population was evaluated by combining food-intake data from the 2012–2014 Korea National Health and Nutrition Examination Survey (KNHANES), and the concentration data. The mean estimated daily intakes (EDIs) of the general population and consumers were 0.010 and 0.012 µg/kg bodyweight (bw)/day, respectively, when the mean values from the tested samples were used. The 95th percentile intake levels of both groups were 0.036 and 0.039 µg/kg bw/day, respectively. The EDIs of FeCN ion for all groups were much lower than the acceptable daily intake (ADI) of 0.025 mg/kg bw/day. These results provide useful information about the use of FeCNs as anticaking agents for consumers and manufacturers.

1. Introduction

Calcium, potassium, and sodium ferrocyanides (FeCNs) are complex cyanides that are used as anticaking agents in food-grade salts (European Commission, 2001). Food-grade salt is added to enhance the taste of foods and to preserve a variety of food products when used in large amounts. The assessment of FeCN intake from food-grade salts is therefore important for determining the potential risk to health (Sarmugam & Worsley, 2014; Sivasankar, 2009).

FeCNs are authorized in food-grade salts for human consumption by the regulations of the European Union, Japan, and Korea. However, in the United States, only sodium ferrocyanide is permitted. In Korea, FeCNs may be used in food-grade salts at levels within the legal limit of 10 mg/kg (as ferrocyanide ion), in accordance with the Korea Food Additives Code (MFDS, 2015a); with the exception of solar sea salts (MFDS, 2015b), this limit applies to food-grade salts. No ferrocyanide ion is permissible in solar sea salts. According to food labelling regulations (MFDS, 2016a), FeCNs are required to be labelled on salt products as “calcium ferrocyanide”, “potassium ferrocyanide” or “sodium ferrocyanide”. In the European Union, FeCNs are permitted in food-grade salts and their substitutes at levels of 20 mg/kg (calculated as anhydrous potassium ferrocyanide), individually or in combination (European Commission, 2011). In the United States, sodium

ferrocyanide should not exceed 13 mg/kg as anhydrous sodium ferrocyanide in food-grade salts (USFDA, 2016). In Japan, the usage level of 20 mg/kg is set for food-grade salts (MHLW, 2016), whereas in the CODEX Alimentarius, ferrocyanides are set at < 14 mg/kg for food-grade salts and < 20 mg/kg for substitutes and sauces (CODEX, 2016). Hence, the accurate and simple determination of FeCN-ion content is important in order to ascertain compliance with these standards as well as to ensure food safety.

When FeCNs are consumed, they are excreted in the urine through glomerular filtration (European Commission, 2001). Short-term studies have demonstrated kidney damage attributed to crystal formation at 5% and to a lesser extent at 0.5% dietary incorporation over 13 weeks. The no-observed-adverse-effect-level (NOAEL) was set at 50 mg/kg (equivalent to around 2.5 mg/kg bodyweight (bw)/day) in the diet based on urinary cell excretion in a chronic rat toxicity study. Finally, an acceptable daily intake (ADI) of 0–0.025 mg/kg bw was set by the Scientific Committee on Food (SCF, 1991); the Joint FAO/WHO Expert Committee on Food Additives (JECFA, 1974) also established the same levels. Given these guidelines and due to the potential for kidney damage, the reliable quantification of FeCNs is crucial. In addition, it is important to monitor the per capita consumption of such additives to ensure that levels of the general population remain within healthy limits.

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Despite these implications, few studies have been carried out to determine the FeCN-ion content from FeCNs in food-grade salts (Roberts & Wilson, 1968; Saitoh, Soeta, Minamisawa, & Shibukawa, 2013; Yamane, Isawa, & Osada, 2006). The daily intake of FeCN has been estimated in two studies. In the US, the per capita daily intake of soluble sodium ferrocyanide as a food additive (anticaking agent), determined by a poundage method, was reported to be 0.35 mg/person (SCOGS, 1981). Jain and Mathur (2014) recently estimated the daily intake of FeCN ion, assessed using Food Frequency Questionnaires (FFQs) and three day food diaries; the mean intake and the 95th percentile for respondents was estimated to be 0.001 ± 0.001 and 0.002 mg/kg bw/day, respectively.

Recently, the amounts of produced and imported food-grade salts in Korea have increased. The estimation of FeCN intake is necessary because ferrocyanides are used as anticaking agents in food-grade salts. To date, there are no data available on the dietary intake of FeCN ion in the Korean population. In order to determine any potential health risks from its intake, an accurate knowledge of FeCN consumption levels is required. Analytical data of FeCN-ion content in regularly consumed salts are required in order to estimate human exposure to ferrocyanides.

The present study was designed with the objective of combining analytical data on FeCN-containing food-grade salts and consumption survey data for the estimation of FeCN-ion intake in the Korean population, and to assess its potential risk by comparing the estimated daily intake to the ADI. To afford optimal analytical conditions for the determination of FeCN content, different parameters that influence the analytical procedure were optimised using high-performance liquid chromatography (HPLC). A total of 801 food-grade salt samples were analysed, and the daily intake of FeCN ion was estimated from the measured concentrations of FeCN ion and the amounts of consumed salts taken from the survey data.

2. Materials and methods

2.1. Materials and reagents

Sodium ferrocyanide was used as a standard and was purchased from Sigma-Aldrich (St. Louis, MO, USA). Sodium hydroxide was used as a solvent during sample preparation and was purchased from Wako Pure Chemical Industries, Ltd. (Osaka, Japan). Sodium perchlorate, used in the preparation of the mobile phase, was supplied by Sigma-Aldrich. All water used in this study was obtained from a Milli-Q ultrapure water system (Millipore, Bedford, MA, USA; resistivity ≥ 18.2 M Ω cm).

2.2. Preparation of the standard solution

A stock FeCN standard solution was prepared by transferring 50 mg of sodium ferrocyanide to a 100-mL volumetric flask and adding 0.02 M NaOH solution to a total volume of 100 mL. The working standard solutions were produced by diluting the stock solution in 0.02 M NaOH solution to form 0.1, 0.5, 1, 5, and 10 mg/L aliquots. The stock solutions were prepared weekly and stored at 4 °C; working standard solutions were prepared daily.

2.3. Food sampling and preparation

A total of 801 salt samples were purchased from food markets in large cities including Seoul, Daejeon, Gwangju, Daegu, Busan, Cheongju, and Sejong in Korea. The samples were separated into six categories according to the Korea Food Code: reworked salt (42), burnt-molten salt (175), refined salt (30), solar sea salt (169), other salt (33), and processed salt (352). Reworked salt refers to a product prepared by dissolving raw salt (100%) in purified water, sea water, or concentrated sea water followed by processing it through filtration, precipitation, recrystallization, dehydration, or by adjusting the concentration of salt.

Burnt-molten salt refers to a product prepared by modifying raw salt (100%) through burning, melting, or other methods; however, it excludes those products processed by washing, grinding, and compression. Refined salt refers to a product of concentrated brine that has been obtained by purifying sea water (including deep ocean water) through an ion exchange membrane, or a salt product processed by evaporating raw salt (100%) dissolved in water with a vacuum evaporator. Solar sea salt refers to crystals of sodium chloride (NaCl) obtained through the natural evaporation of sea water in a salt field and products obtained by their grinding, washing, dehydrating, or drying. Other salt refers to powdered or crystallized products manufactured by processing rock salt or lake salt to edible standards. Processed salt refers to a product made by adding foods or food additives to reworked salt, burnt-molten salt, refined salt, or other salt (not less than 50%), followed by processing. Six FeCN-free salts were selected for method-validation purposes. All samples were stored at room temperature (20–25 °C).

Each salt sample (2–5 g) was transferred into a 50-mL volumetric flask, and dissolved in a 0.02 M NaOH solution to a total volume of 50 mL. The sample was incubated in an ultrasonic bath for 5 min. The extract was then filtered with a 0.22- μ m polyvinylidene difluoride (PVDF) membrane filter for HPLC analysis. For the processed salt containing pepper and flavour, 2 mL of the extract was subjected to an enrichment procedure using a Sep-Pak C18 cartridge (Waters Co., Milford, USA; 500 mg) at a flow rate of ~ 3 mL/min. The cartridge was preconditioned with 5 mL of methanol followed by 5 mL of water. After loading the sample, the flow-through solution was collected into a 15-mL conical tube. The solution was then filtered with a 0.22- μ m PVDF membrane filter.

2.4. HPLC conditions

HPLC analysis was performed on a Shiseido Nanospace SI-2 (Shiseido Co., Tokyo, Japan) instrument coupled to a photodiode array detector (PDA). The HPLC system included an online degasser, a binary pump, an autosampler, and a column oven. An AG11-HC guard column (4 mm \times 50 mm, 9 μ m) and an AS11-HC analytical column (4 mm \times 250 mm, 9 μ m) were used for chromatographic separation. Both columns were purchased from DIONEX (Thermo Fisher Scientific, Sunnyvale, USA). The FeCN chromatograms were analysed by Ezchrom Elite software, Shiseido Corporation. All separations were carried out isocratically at 35 °C with a mobile phase comprising 200 mM sodium perchlorate and 20 mM sodium hydroxide. The flow-rate was maintained at 0.6 mL/min and a 10 μ L sample volume was injected for all experiments. The FeCN eluted from the columns was monitored by a PDA detector at 221 nm. The FeCN absorption spectra were recorded over the 200–600 nm wavelength range. Peaks were identified by comparing the retention times and absorption spectra of the samples with those of the standard solutions.

2.5. Method validation

The HPLC-UV method for the determination of FeCN in food-grade salts was validated for linearity, selectivity, accuracy (recovery), precision, LOD and LOQ according to AOAC guideline (2012). Selectivity was determined by examining the chromatograms to verify the absence of interfering peaks arising from the food-grade salts. Matrix-matched calibrations were performed by spiking five concentrations (0.1, 0.5, 1, 5, 10 μ g/mL) of FeCN in reagent-grade NaCl; linearity was determined using these five concentrations in triplicate. The LOD and LOQ values were calculated from calibration curves constructed using each reagent-grade NaCl sample spiked with FeCN at five concentrations; these were determined to be 3.3 and 10 σ /S, respectively, where σ is the standard deviation of the intercept and S is the slope of the regression line determined from the calibration curve. Accuracy (recovery) and precision (expressed as relative standard deviation, %RSD) were evaluated by analysing six samples spiked with the stock solution at final

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