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Quality control in the production of fluoridated food grade salt

Summary

Fluoridated food grade salt has been manufactured in Switzerland for 50 years. Since correct dosing is important not only for effective caries prophylaxis but also in order to guarantee food safety, the production of fluoridated salt must be accurately monitored. The authorities do not impose any specific requirements as regards the purity of the fluoride compounds that are used, nor the homogeneity or dosing accuracy that should be attained during the manufacture of fluoridated salt. The quality requirements to be observed and the means by which these standards are to be ensured must largely be determined by the producer himself as part of the "self-monitoring" that is stipulated by the law.
Depending on whether fluoridated salt is manufactured in a continuous or discontinuous process and on whether the fluoride is added as a solution or in solid form, a plant-specific testing plan must be drawn up for the implementation of quality monitoring. On the basis of statutory requirements, a food manufacturer must subject all the processes which he carries out to a risk analysis (HACCP study). Monitoring of the dosing of fluoride must be classified as a Critical Control Point (CCP).
Three well-established testing methods which have been validated in ring tests are available to determine the fluoride content in food grade salt (a potentiometric, an ion-chromatographic and a photometric method). In practice, the potentiometric method has proven to be a simple, accurate and comparably low-priced process and is widely used.

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Introduction

Since 1995, the Ordinance on Nutritional Value (NwV 1995) has regulated the enrichment of foodstuffs with essential or physiologically beneficial substances such as vitamins or minerals throughout Switzerland. One of these "physiologically beneficial substances" is fluorine, which may be added to food grade salt in the form of fluoride in order to prevent dental caries. In Switzerland, the content of fluoride in fluoridated food grade salt is 250 mg/kg.
The accurate addition of such a small quantity of fluoride per kilogram of food grade salt is not a simple matter in terms of technology. However, accuracy is very important because excessive intake of fluoride can cause toxic effects. Conversely, since

one kilogram of food grade salt is sufficient for an average of more than six months in a two-person household, too low a dosage (or in the extreme case, the total absence) of the declared admixture of fluoride can cause a serious gap in individual caries prevention. Producers of fluoridated food grade salt must therefore take suitable technical measures to ensure that precisely the declared quantity of fluoride is added and homogeneously distributed in the salt. Quality control has the task of monitoring correct functioning of the technical plant and the correct implementation of operating regulations. If any divergences are ascertained (instances of over- or under-dosage), the respective batches of fluoridated salt must not be put on sale.

Legal requirements

No special approval is required to manufacture fluoridated salt in Switzerland. However, the principle of "self-monitoring" which applies to all producers of foodstuffs is contained in Article 23 of the Swiss Food Law (LMG 1992, Federal Law on Foodstuffs and Commodities): Anyone who manufactures, treats, distributes, imports or exports foodstuffs must ensure in connection with his activities that the goods satisfy the legal requirements. He must examine them, or have them examined, in accordance with "Good Manufacturing Practice" (GMP).

The term "Good Manufacturing Practice" is not enclosed in quotation marks by chance. Unlike the pharmaceutical sector, the food sector does not yet have any generally recognized guidelines making it possible to deduce the specific measures that have to be taken in individual cases in order to guarantee the safety of food. As regards the production of fluoridated food grade salt, the Swiss Ordinance on Nutritional Value (NwV 1995) merely stipulates the type of fluorine compounds (exclusively fluorides) and the dosage (250 mg fluoride/kg of salt). The quality criteria that must be met by the fluoride compound used and the extent to which the fluoride content in the salt may vary from the desired value remain unclear. Nevertheless, the Swiss Food Manual (SLMB 2004) specifies and describes the methods that may be used to determine the content of fluoride in food grade salt.

Likewise, the Codex Standard for Food Grade Salt (Codex Standard 150) of WHO (2001) does not provide any specific standards for the quality of the fluoride compounds used to manufacture fluoridated salt or for the dosing accuracy to be observed during manufacture. Section 3.4.3, "Quality assurance", merely states that iodated food grade salt may only be produced by manufacturers who have the appropriate technical equipment and experience in order to ensure correct dosage. This passage may also be applied analogously to the production of fluoridated food grade salt. The Codex Standard does not propose a method to monitor the content of fluoride in food grade salt – in contrast to the determination of the iodine content.

To a large extent, it is therefore left up to the producers themselves to take responsibility for determining the specific measures that must be taken to ensure product quality in the manufacture of fluoridated salt. As regards dosing accuracy, it may be assumed that divergences of 15–20% from the declared value are tolerated by the authorities responsible for implementing legislation on foodstuffs.

In Switzerland, compliance with and implementation of laws and ordinances for health protection and protection against wilful deceit are supervised by the cantonal laboratories. However, since the potential for health hazards is low in the case of food grade salt with or without salt additives, no regular checks are carried out on this group of products. Regardless of this, fluoridated salt has occasionally been examined in connection with special projects at university dental schools. The fluoride dosage was correct in all cases (T. M. Marthaler, G. Menghini, personal communication 2005).

Organization of quality control

The organization of quality control essentially depends on whether the fluoridated food grade salt is manufactured in batches or in a continuous process. Consideration also has to be given to the form in which the fluoride is added. If the fluoride is added as an aqueous solution, it is not only necessary to carry out an initial inspection of the starting material (potassium or sodium fluoride) but also to monitor and supervise the manufacture of the dosing solution. This step is eliminated in the case of solid dosing.

The United Swiss Saltworks on the Rhine have been producing food grade salt with iodine in Switzerland since 1920, and they have also been manufacturing salt with iodine and fluoride since 1955. Traditionally, the continuous manufacturing process with liquid dosing has always been used. At the outset, the iodine was added by means of a dropping funnel and a stopwatch, but nowadays ingenious dosing plants with electronic controls and monitoring systems are in operation. However, there has been no change of the basic manufacturing principle, which is "Add iodine/fluoride as an aqueous solution into a continuous flow of salt, mix and pack the salt".

For the continuous manufacture of fluoridated food grade salt, step-by-step monitoring guarantees the safety and quality of the product (Tab. I).

The intervals at which samples have to be taken and examined in steps 4 and 5 of the continuous production process have to be defined on a plant-specific basis. The intensity of these checks depends on the reliability of the electronic dosing and monitoring system, and on the specific requirements that are set for the safety and quality of the end product. In every case, particular attention has to be paid to the start and end of a manufacturing run because these are the phases of the production process

Tab. I Steps in monitoring the continuous production of fluoridated salt

Step	Check	Sampling
1	Composition of the salt additives (sodium or potassium fluoride)	Batch-by-batch (every time goods are received)
2	Content of the dosing solution	Batch-by-batch (every preparation)
3	Electronic monitoring of the dosing process (ongoing recording of the quantity of salt produced and the consumption of dosing solution)	—
4a	Fluoride content in the salt after the dosing and mixing plant	Continuous
4b	Fluoride content in the salt at the silo intake	Continuous
5	Fluoride content in the packed product (final inspection with product release)	Batch-by-batch (daily packed batch)

where the largest fluctuations must be expected. Testing plans must be compiled to provide a binding record of which checks have to be carried out at which intervals during the production of fluoridated food grade salt.

If the salt is transported unpacked over long distances (within the plant for instance on conveyor belts) after the fluoride has been added, segregation phenomena may occur depending on the particle size and degree of dryness of the salt. If this danger exists, the fluoride content should be regularly checked not only on exit from the mixer and in the packed end product, but also at the silo intake (step 4b). Food grade salt that has already been transferred into cans and packages with a monitored homogeneous distribution of fluoride will not usually segregate during subsequent temporary storage and transport to the customer.

In order to guarantee the safety of food, producers must identify and evaluate all health risks that may occur during the production of a foodstuff (this is known as the HACCP concept: Hazard Analysis and Critical Control Point). Working procedures or production steps in which health risks can be excluded or reduced are designated as Critical Control Points (CCPs). Since underdosage would reduce the cariostatic benefit and overdosage would entail dental fluorosis (a cosmetically undesirable phenomenon), monitoring of the fluoride content has to be classified as a CCP. Accordingly, special attention must be paid to the quality checks during fluoridated salt production processes.

If fluoridated food grade salt is produced in a batch mixer, monitoring is easier than for the continuous process. The mixer can be charged with precisely measured quantities of dosing solution or fluoride in solid form, and the mixture is bound to be obtained in the predetermined composition. One sample per batch is usually sufficient for monitoring purposes. For this reason, the discontinuous production process is the method of choice for those new to the field or for manufacturers with smaller production volumes.

Methods to determine the content of fluoride in food grade salt

A series of tried-and-tested analytical processes are available to determine the content of fluoride in food grade salt. The choice of method is primarily based on the instrumentation of the relevant monitoring laboratory. However, consideration has also to be given to criteria such as the expected incidence of samples, cost of maintenance, user-friendliness, desired level of automation and purely analytical factors such as disruptive influences due to impurities or additives.

Three main analytical processes are used in monitoring laboratories to determine the fluoride content: (a) potentiometric determination using ion-selective electrodes, (b) ion-chromatographic determination, and (c) photometric determination using an SPADNS reagent.

In recent years, all three methods mentioned above have been revised and revalidated in ring tests by a working group of ESPA (1996, 1999, 2002; European Salt Producers' Association, now known as EUSalt). Table II lists the limit of determination, repeatability r and reproducibility R.

The *potentiometric method* (Fig. 1) offers good precision and reproducibility, and is easy to automate if necessary. However, errors may occur in the case of salt varieties that naturally have a higher content of calcium or magnesium, or those to which anti-caking agents containing calcium or magnesium have been added. These can usually be eliminated by adding the complexing agent CDTA (trans-1,2-diaminocyclohexane-NNN'N-tetra-acetic acid). The cost of purchasing simple measuring equipment comprising an ion meter, ion-selective electrode and reference electrode is relatively low, so this process can also be used by laboratories with modest financial resources.

Tab. II Determination limit, repeatability r and reproducibility R of different methods for determining fluoride (in mg fluoride/kg salt)

Potentiometric method (determination limit: 10 mg F/kg salt)			
Fluoride content	296	149	243
Repeatability r	12	5	13
Reproducibility R	17	9	30
Ion-chromatographic method (determination limit: 15 mg F/kg salt)			
Fluoride content	24	104	254
Repeatability r	3	4	6
Reproducibility R	9	9	9

Photometric method (determination limit: 40 mg F/kg salt)				
Fluoride content	96	157	254	247
Repeatability r	10	7	27	21
Reproducibility R	32	29	74	90

Note: Repeatability describes the agreement within a laboratory whereas reproducibility refers to the agreement between laboratories

The advantage of the *ion-chromatographic* process is that bromide, nitrate, sulphate and iodide can also be determined in the same working operation as the fluoride content. Precision and reproducibility of this method are good, but considerably more time is required for a determination from a single sample than in the case of the potentiometric process (the runtime for a chromatogram is approx. 18–30 minutes). In contrast to the potentiometric method, the operation of an ion chromatograph makes relatively high demands on the infrastructure of the laboratory and the qualifications of the employees who are responsible for carrying out the analysis. Moreover, the acquisition cost of an ion

Fig. 1 Measuring equipment for direct potentiometric determination of the fluoride content in food grade salt (Metrohm, Herisau)

chromatograph with a separation column, PC control and evaluation software is considerably higher than the cost of purchasing a simple measuring system for potentiometric fluoride determination.
The *photometric method* is insensitive to disruptive influences due to calcium or magnesium ions. Use of this process is impossible only in the presence of the anti-caking agent tricalcium phosphate (E 341). Photometric determination is substantially inferior to the other two methods in terms of precision and reproducibility. Given that the analysis is comparatively complicated to perform, this process is generally used only in cases where a laboratory has no ion meter or ion chromatograph, but where a spectral photometer is available.
Depending on the analytical method, the specified quantity of salt to be weighed in for each determination is between 5 g and 50 g. Smaller quantities should be avoided because they lead to unreliable measuring values. With test portions of more than 50 g it is not possible to detect inhomogeneities within a small packing unit.

Zusammenfassung

Seit 50 Jahren wird in der Schweiz fluoridiertes Speisesalz hergestellt. Da eine korrekte Dosierung für eine wirksame Kariesprophylaxe – aber auch zur Gewährleistung der Lebensmittelsicherheit – wichtig ist, muss die Produktion von Fluorsalz genau überwacht werden. Bezüglich Reinheit der eingesetzten Fluoridverbindungen sowie der zu erreichenden Homogenität und Dosiergenauigkeit bei der Fluorsalzherstellung machen die Behörden keine konkreten Vorgaben. Welche Qualitätsanforderungen eingehalten und mit welchen Mitteln die Einhaltung dieser Vorgaben sichergestellt wird, muss im Rahmen der gesetzlich vorgeschriebenen «Selbstkontrolle» weitgehend vom Produzenten selber bestimmt werden.
Abhängig davon, ob Fluorsalz in einem kontinuierlichen oder diskontinuierlichen Prozess hergestellt und ob das Fluorid als Lösung oder Feststoff zugesetzt wird, ist für die Durchführung der Qualitätskontrolle ein anlagenspezifischer Prüfplan zu erstellen. Aufgrund gesetzlicher Vorgaben muss der Lebensmittelhersteller alle von ihm durchgeführten Prozesse einer Risikoanalyse unterziehen (HACCP-Studie). Die Überwachung der Fluoriddosierung ist als Critical Control Point (CCP) einzustufen.
Für die Bestimmung des Fluoridgehaltes in Speisesalz stehen drei bewährte und in Ringversuchen validierte Prüfmethoden zur Verfügung (potentiometrische, ionen-chromatographische und photometrische Methode). In der Praxis hat sich die potentiometrische Methode als einfaches, präzises und vergleichsweise kostengünstiges Verfahren bewährt und durchgesetzt.

Résumé

En Suisse, la fabrication de sel de cuisine fluoré est connue depuis 50 ans. Or, du fait que le dosage correct du fluorure est important pour une prophylaxie efficace contre la carie – mais également pour des raisons de sécurité alimentaire – il est impératif de surveiller de manière précise la production du sel fluoré. Les autorités ne fournissent pas d'indications concrètes concernant ni la pureté des composants fluorés mis en œuvre, ni l'homogénéité et la précision du dosage à atteindre lors de la production du sel fluoré. Dans une large mesure, il incombe dès lors au fabricant de déterminer lui-même, dans le cadre de l'autocontrôle imposé par la loi, les exigences en matière de qualité auxquelles il s'astreint, de même que les moyens permettant de les respecter.
Suivant que le sel fluoré est fabriqué dans un processus continu ou discontinu, ou que le fluorure est incorporé sous forme de solution ou de composant solide, il y a lieu d'établir un plan de contrôle spécifique aux installations pour le contrôle de la qualité. En accord avec les prescriptions légales, le fabricant de denrées alimentaires est tenu de soumettre l'ensemble des processus qu'il met en œuvre à une analyse des risques (étude HACCP). La surveillance du dosage de fluorure doit être qualifiée de Critical Control Point (CCP). Pour la détermination du contenu en fluorure, trois méthodes éprouvées et validées dans des essais comparatifs interlaboratoires sont à disposition (méthode potentiométrique, photométrique et par chromatographie ionique). En pratique, la méthode potentiométrique a fait ses preuves et s'est imposée en tant que procédé simple, précis et relativement peu cher.

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