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(54) Title: PROCESS OF INFUSION – USE OF FREEZE DRYING AS PROCESS STEP

(57) Abstract: 1. Title: Process of infusion of vitamins and/or minerals 2. Short Description: 2.1 Technical problem of the invention: Infusion into plant-based slices is ineffective, as the diffusion into the cell structure is limiting both the speed of the infusion process and the amount of material infused in a given infusion time. The new process is meant to improve the process efficiency. 2.2 Solving the problem or technical task: Using an oil-in-water emulsion as infusion liquid, instead of two subsequent infusion steps. 2.3 Field of application: Fortification of food material especially crisps or other snacks with oil- and /or water-soluble micronutrients



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**Description of Invention**

Process of infusion – use of freeze drying as process step

**Background**

[001] Human nutrition is highly complex. Beside the macronutrients: fat, protein and carbohydrates, that mainly provide energy, also micronutrients have to be supplied to the human body in sufficient quantities. On a more detailed level beside vitamins and minerals, also dietary fibres and antioxidants need to be considered. A high number of people do not manage to meet all requirements adequately. Fortification is a way to complement everyday food by components that are not supplied by the standard meals. The complemented material may be natural occurring foods like juices or spices or concentrates or a specially produced material like vitamin C. In this context, the question of bioavailability is also of importance. A fortified food has the advantage over a food supplement, like a tablet or liquid dose, as it does not need specific acceptance, but will be consumed with the regular food. Fortifying snacks is of particular interest, as snacks are often supplied in a dried- and storage-stable form. Therefore, these products are well-suited for fortification with temperature-instable components.

[002] Typical components for fortification could be, without limiting it to, these examples, are vitamins like vitamin A and D, as well as minerals like Iron (Fe) and Zinc (Zn). Base materials of the snack could be, without limiting it to, roots and tubers like potato, sweet potato or carrots.

**State of art**

[003] The standard process is described for example in [1 and 2] and can be summarised as follows: The base material for example a root or tuber is sliced into thin slices. The root or tuber may be, for example, potato, sweet potato, carrot, beet root or cassava without limiting the choice to these materials. The material may be blanched (temperature treatment by steam or hot water) before- or after slicing.

[004] The slicing may be performed using any type of slicing apparatus, e.g. apparatus of Urschel CC Slicer type, Urschel Lab Inc. Chesterton, IN USA: Preferred slice thickness for infusion in the standard infusion process should be low. Preferred thickness is below 5 mm, or more preferred below 2 mm, or more preferred below 1 mm, more preferred below 0.5 mm, or even more preferred 0.2 mm.

[005] The invention is however not limited to thin slices as the diffusion is effectively enhanced, so that also thick slices up are possible. Limited to a preference of below 30 mm, or more preferred below 20 mm, or more preferred below 10 mm, or even more preferred 5 mm

[006] The slices are then either blanched or directly submitted to the infusion bath. The slices are submerged into an infusion liquid, which is a baseliquid which contains the material that shall be infused. Typical infusion material in standard infusion processes are calcium, for texture improvement, or vitamin C, for chemical stabilisation or taste improvement. The infusion liquid may be stagnant or moving, for example flowing in a duct or in a mixer vessel by use of a stirrer. Flowing liquid would show an enhanced mass transport from the liquid to the slices [3]. This infusion step will be characterised by its duration and the liquid temperature. After infusion, the snacks are produced by the standard production process similar to the process for non-fortified products. The process may be adapted to take the sensitivity of the fortification material into account, for example lower temperatures. In the case of crisps, this particularly involves, but is not limiting to, a drying- or a frying step to reduce product moisture and generate a crispy structure. A cooling step is followed by any kind of finishing step, for example flavouring, surface coating or any other finishing step.

#### **Problem to be solved**

[007] The target is to enrich snack base material with a sufficient quantity of the fortification material, i.e. 5% of the daily recommended allowance (RDA), even preferred more than 10% of the RDA, or even preferred more than 30% RDA, even preferred more than 50% RDA or more than 100% of the RDA within a standard portion size. The optimum level may depend on the risk of overdosing of the fortification material. The infusion typically work on the basis imbibing the fortification material into the product by diffusion. This may take a long time especially if slices are thick. In addition, the maximum amount of fortification material that can be added economically is limited. This is particularly true for cell material like slices of roots and tubers. The cell wall poses a diffusion barrier that reduces the diffusion efficiency significantly. This has led to the invention of vacuum impregnation techniques as described for example by Tiwari et al. [2]. The presented process provides a significant improvement over this state of art technology in food infusion or so called impregnation.

### Description of invention

[008] The impregnation efficiency has been improved by adding an additional process step to the impregnation- or infusion procedure. Before impregnation, the product is freeze dried to generate a preferred structure for infusion. Freeze drying is a process whereby the product is dried while maintaining its solid form. The water is not in liquid form, so that no or only little shrinkage occurs during drying. Voids remain at places where ice crystals had been before drying. The process comprises a freezing step, followed by a drying or evaporation step to remove the solidified water. This drying step is performed at conditions avoiding a melting of generated ice crystals, which is given at drying conditions below the triple point of water. The triple point of water is defined by a water vapour pressure of 611 Pa (Pascal) and a temperature of 273 K. Often the drying is divided in a main drying step, as described, and a final drying step at a higher temperature, to remove so-called "bound" water. More details can be found, for example, in [4, 5]. The freeze drying can, for example, be performed in a two chamber freeze dryer, like the Zirbus Sublimator 15, Zirbus *technology* GmbH, Bad Grund Germany, or any other freeze dryer installation, including so-called atmospheric freeze dryers.

[009] In drying technology it is known that the drying speed in freeze drying of plant material can be accelerated by freezing the product slowly. This is explained by stronger growth of the crystals that consequently break the cell wall, allowing water to leave the cell more easily [4]. The basic concept of this invention is to use this mechanism to generate a pathway for the infusion liquid in the specific task. As the ice crystals are of considerable size (several  $\mu\text{m}$ ) this should also allow oil drops to penetrate the structure that is generated by freeze drying. Typically, the product will be dried completely, i.e. most frozen water being evaporated or even dried to a standard dried material, having a water activity  $a_w$  (at 25°) of below 0.6, or more preferred of below 0.4, or even more preferred of below 0.3 or most preferred below 0.2. The structure generation proceeds from the outside of the dried product into the material. Therefore, also an incomplete drying will generate structures well-suited for impregnation, as long as the generated porosity has a higher or equal volume than the volume of the infusion liquid, containing the intended amount of infusion material.

[010] The dried material will then be exposed to the known infusion- and production process as already described. The process comprising specifically, but is not limited to: infusion, drying or frying, plus optional steps like layering or finishing.

[011] A particularly efficient variant of the technique is the combination of the described pre-processing with a so-called vacuum impregnation [2] as an infusion technique. This process comprises the following steps: placing the product in a vacuum impregnation room, removing parts of the atmosphere by drawing an appropriate vacuum given as a pressure difference to the starting pressure

of more than 100 hPa (10 000 Pa) or more preferred more than 200 hPa, or more preferred more than 500 hPa, or more preferred more than 700 hPa. This pressure difference stands for a removed volume of gas i.e. air from the pore space. The above data should be read in the case of starting gas pressures higher than the standard atmospheric pressure of 0.1 MPa as pressure ratios (ratio of reduced pressure to starting pressure) of 0.9, 0.8, 0.5 and 0.3 respectively. The infusion liquid is injected into the infusion room and is imbibing into the dried slices. This imbibition can be enhanced by increasing the gas pressure in the room i.e. breaking the vacuum. The higher pressure on the outside of the slice, compared to the inner gas pressure, pushes the infusion liquid into the slices.

### Example 1

[012] Fresh potato was cut into slices of 1.5 mm thickness using a manual slice cutter "Allesschnieder Master M90 Graef, Arnsberg, Germany". The slice thickness was determined using an eddy-current thickness measurement device. Freeze drying was performed in a Zirbus Sublimator 15, Zirbus *technology* GmbH, Bad Grund Germany. The infusion was performed with water-based mineral solutions. The concentrations of fortifying minerals in the infusion liquid are given in the table below. The sample marked "blank" were infused with water. Vacuum infusion as well as standard infusion at ambient gas pressure were conducted. Infusion time was set to 3 min. The final drying was conducted in a Philips Airfryer XXL. Results are shown in figure 1 and figure 2. A clear improvement can be seen when freeze drying was used as a pre-processing step.

<b>Slices</b>	<ul style="list-style-type: none"> <li>• <b>Raw material: potato</b></li> <li>• <b>Thickness: 1.5 mm</b></li> <li>• <b>Pretreatment: no treatment, freeze dried</b></li> </ul>
<b>Infusion Liquid</b>	<ul style="list-style-type: none"> <li>• Ca-solution (1 %(w/w) Ca-lactate in distilled water)</li> <li>• Fe/Zn-solution (0.1 %(w/w) Fe(III)pyrophosphate + 0.1 %(w/w) Zn-gluconate)</li> </ul>
<b>Infusion Process</b>	<ul style="list-style-type: none"> <li>• <b>Pressure: ambient pressure, 300 mbar</b></li> <li>• Temperature: ambient</li> <li>• Time: 3 min</li> </ul>
<b>Drying</b>	<ul style="list-style-type: none"> <li>• Device: Air fryer</li> <li>• Temperature: 100 °C</li> <li>• Time: 15 min</li> </ul>

**Example 2**

[013] Fresh potato was cut into slices of 1.5 mm thickness using a manual slice cutter "Allesschnieder Master M90 Graef, Arnsberg, Germany". The slice thickness was determined using an eddy-current thickness measurement device. Freeze drying was performed in a Zirbus Sublimator 15, Zirbus *technology* GmbH, Bad Grund Germany. The infusion was performed with an oil-in-water emulsion. Said emulsion contained 12,5% oil and was prepared using a high-pressure homogenisator type HL I.3-400KX, HST Maschinenbau GmbH, Dassow, Germany, at a homogenisation pressure of 30 MPa. The sample marked "blank" were infused with water. Vacuum infusion as well as standard infusion at ambient gas pressure were conducted. The final drying was conducted in a Philips Airfryer XXL. Infusion time was set to 3 min. Oil is the carrier of oil-soluble fortification material, like oil-soluble vitamins. Oil load is used as a marker for successful fortification with oil-soluble fortification material. Results are shown in figure 3, showing a clear improvement when using freeze drying as a pre-processing step.

<b>Slices</b>	<ul style="list-style-type: none"> <li>• <b>Raw material: potato</b></li> <li>• <b>Thickness: 1.5 mm</b></li> <li>• Pretreatment: <ul style="list-style-type: none"> <li>○ no treatment</li> <li>○ blanching - 75 °C, 2 min</li> <li>○ pre-dried - 75 °C, 10 min</li> <li>○ pre-dried – 75 °C, 30 min</li> <li>○ freeze dried</li> </ul> </li> </ul>
<b>Infusion</b>	<ul style="list-style-type: none"> <li>• O/W Emulsion:</li> </ul>
<b>Liquid</b>	<ul style="list-style-type: none"> <li>○ Oil concentration: 12.5 %(w/w)</li> <li>○ Tween 20 per oil: 0.15 (w/w)</li> <li>○ Homogenization pressure: 30 MPa</li> </ul>
<b>Infusion</b>	<ul style="list-style-type: none"> <li>• Pressure: ambient pressure and 300 mbar</li> </ul>
<b>Process</b>	<ul style="list-style-type: none"> <li>• Temperature: ambient</li> <li>• Time: 2 min</li> </ul>
<b>Drying</b>	<ul style="list-style-type: none"> <li>• Device: Air fryer</li> <li>• Temperature: 100 °C</li> <li>• Time: 15 min</li> </ul>

**Cited non Patent Literature**

- 1 Lusas, E. W.; Rooney, L. W.; Snack Food Processing 2001, CRC Press, ISBN 1-56676-932-9
- 2 Tiwari, P., Joshi, A., Varghese, E., Thakur, M., 2018. Process standardization and storability of calcium fortified potato chips through vacuum impregnation. J. Food Sci. Technol. 55, 3221–3231. <https://doi.org/10.1007/s13197-018-3254-3>
- 3 Mersmann, A.; Thermische Verfahrenstechnik, 1980, Springer Verlag, ISBN 3-540-09903-4

### Claims

What is claimed is:

1. a procedure to promote the infusion into plant-based slices, said procedure comprising the steps of:
  - a. freezing said slice of plant-based material
  - b. drying the frozen slices at conditions below the triple point of water i.e. temperatures below 0°C and water vapour pressures below 6,11 hPa.
  - c. infusing the resulting porous material with a liquid
  - d. drying or frying the slices
  - e. finishing the slices by a coating- or layering step,

whereby the steps d and e are optional. The infusion step may be a vacuum infusion or atmospheric infusion.
2. Process of infusion with a water-based solution into slices of plant-based material using claim 1
3. Infusion with an oil-based liquid into slices of plant-based material using claim 1
4. Infusion with a water-based solution into slices of tubers or roots using claim 1
5. Infusion with an oil-based liquid into tuber slices using claim 1
6. Infusion based on claim 1 with a water-based liquid containing a water-soluble mineral or a combination of water-soluble minerals
7. Infusion based on claim 1 with a water-based suspension containing a non-water-soluble mineral or a combination of non-water-soluble minerals
8. Infusion based on claim 1 with a water-based liquid containing vitamins, i.e. either a single vitamin or a combination thereof, where the vitamin is water soluble
9. Infusion based on claim 1 with a water-based liquid containing vitamins, i.e. either a single vitamin or a combination thereof, where the vitamin is not water soluble
10. Infusion based on claim 1 of a liquid fat containing a mineral or a combination of minerals
11. Infusion based on claim 1 of a liquid fat containing an oil-soluble vitamin or a combination of oil-soluble vitamins
12. Infusion based on claim 1 of a liquid fat containing a combination of one or more of the following components: oil-soluble vitamins, non-oil-soluble vitamins and minerals
13. Infusion based on claim 1 of an oil-in-water emulsion containing any combination of the following components: an oil-soluble vitamin, non-oil-soluble vitamins, water-soluble mineral or non-water-soluble mineral in either of the phases.
14. Any combination of claim 5 to 12.
15. Any combination of claim 2 and 3.



Ca Concentration

Ca-solution:

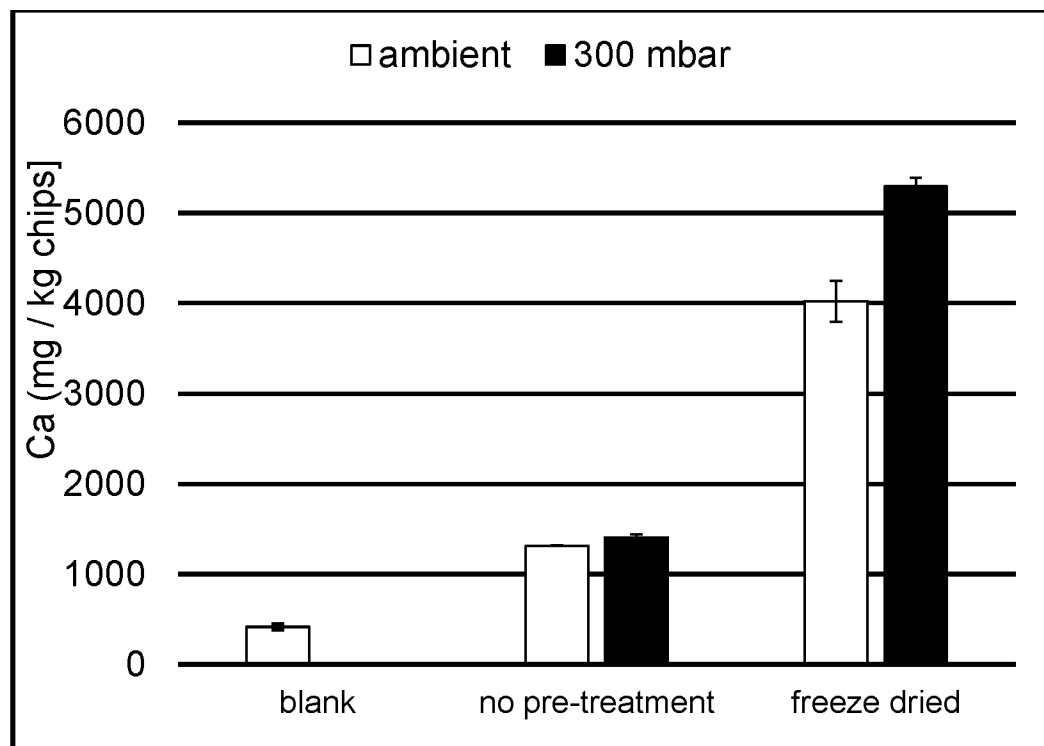


Figure 1: Calcium concentration in mg /kg dried potato chips infused under ambient (grey) and vacuum (blue) conditions for untreated and freeze dried slices prior to infusion; for the blank water was used as infusion liquid

Fe/Zn Concentration Fe/Zn-solution:

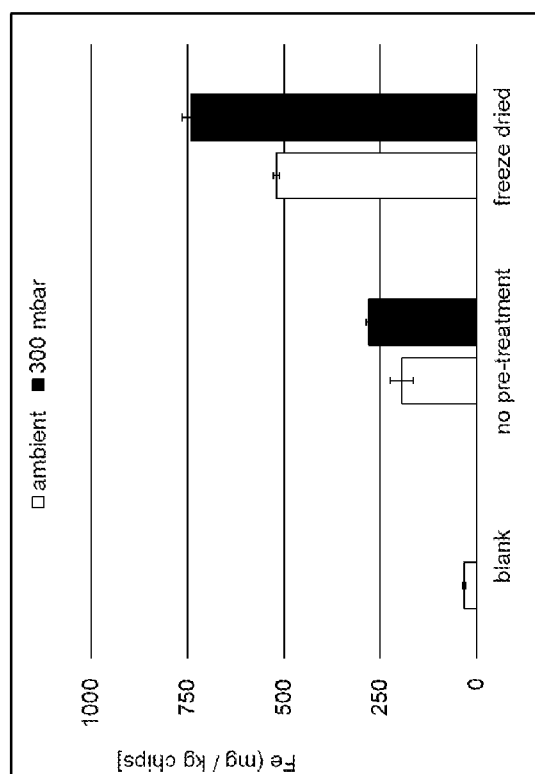
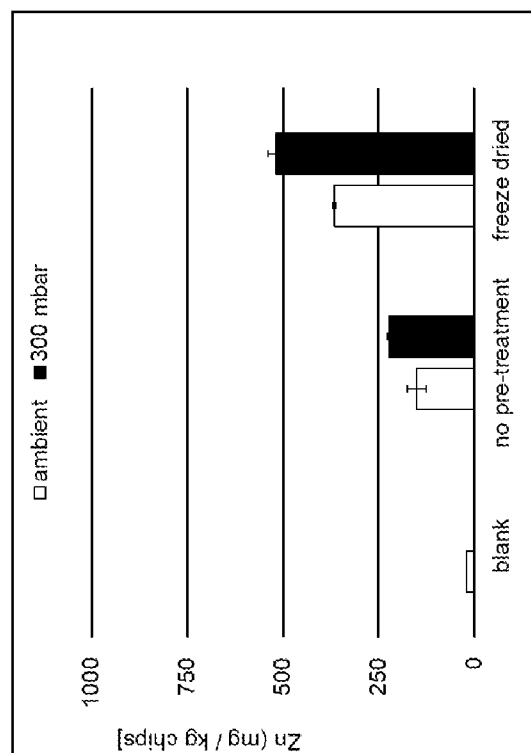


Figure 2: Iron (left) and Zinc (right) concentration in mg /kg dried potato chips infused under ambient (grey) and vacuum (blue) conditions for untreated and freeze dried slices prior to infusion; for the blank water was used as infusion liquid

## Oil concentration

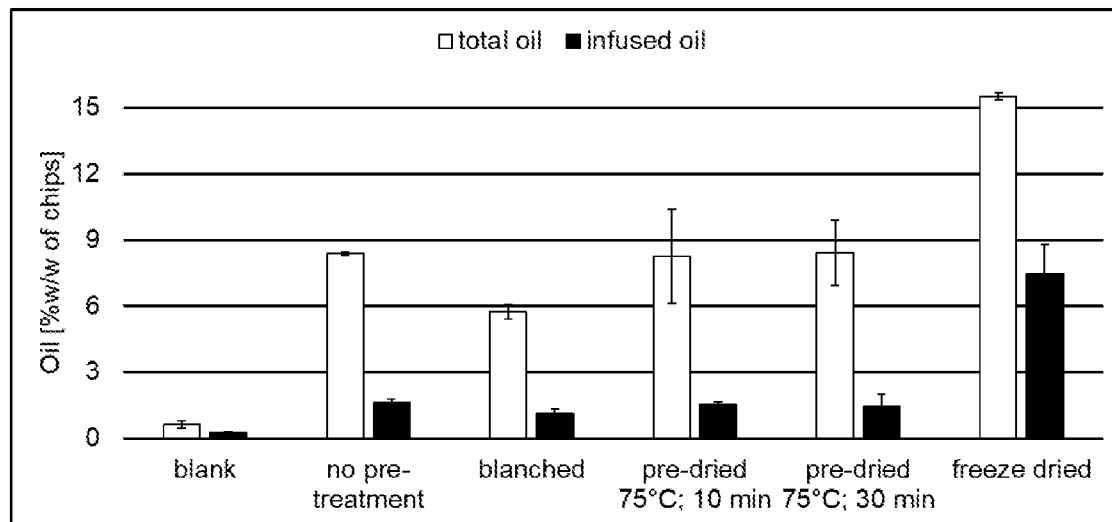


Figure 3: Amount of total and infused oil in dried potato chips differing in the pre-treatment of slices prior to infusion; for the blank water was used as infusion liquid

## INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2021/051935

## A. CLASSIFICATION OF SUBJECT MATTER

INV. A23L19/10 A23L19/12 A23L19/18 A23L33/15 A23L33/155  
A23L33/16

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 440 449 B1 (HIRSCHBERG EDWARD [US]) 27 August 2002 (2002-08-27) column 7, lines 52-55; claim 26 column 6, line 43 column 9, lines 13-20	1-15
X	----- US 2016/095329 A1 (ROOS YRJO [IE]) 7 April 2016 (2016-04-07) paragraphs [0036], [0039], [0044]; claims 1-6	1-15
A	----- US 2009/297671 A1 (BASKER VARADHARAJAN RADHAMANI [US] ET AL) 3 December 2009 (2009-12-03) the whole document ----- -/-	1-15



Further documents are listed in the continuation of Box C.



See patent family annex.

\* Special categories of cited documents :

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Date of the actual completion of the international search

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Name and mailing address of the ISA/

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## INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2021/051935

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>TIWARI PRATIBHA ET AL: "Process standardization and storability of calcium fortified potato chips through vacuum impregnation", JOURNAL OF FOOD SCIENCE AND TECHNOLOGY, SPRINGER (INDIA) PRIVATE LTD, INDIA, vol. 55, no. 8, 10 July 2018 (2018-07-10), pages 3221-3231, XP036547856, ISSN: 0022-1155, DOI: 10.1007/S13197-018-3254-3 [retrieved on 2018-07-10] cited in the application the whole document -----</p>	1-15

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2021/051935

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