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UNITE UNIVERSITÀ DEGLI STUDI DI TERAMO

ERASMUS + PROJECT : AG-LAB

Use of vacuum impregnation for food stabilization and enrichment of functional compounds (plants food and meat): basic principles, concepts and techniques, comparison with traditional techniques

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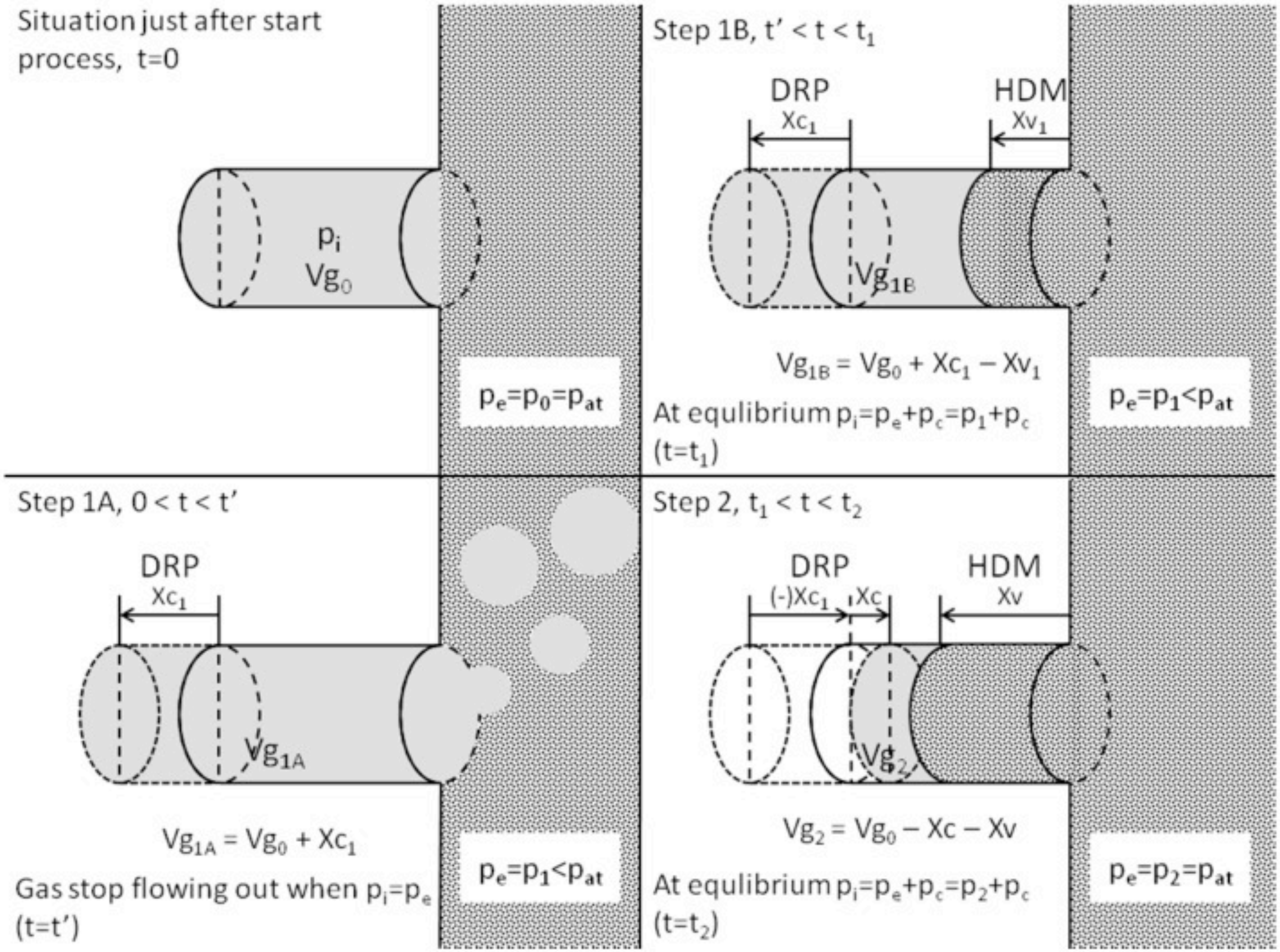
VACUUM IMPREGNATION: GENERAL ASPECTS

- Vacuum impregnation (VI) is a food processing technique based on diffusion.
- **Its main goal is the injection of the external solution into the food material**
- To achieve this, **the application of vacuum is the only necessity**, hence the solution may be isotonic.
- The process is notably faster and/or more efficient compared with traditional process (eg. dipping, salting, osmotic dehydration….)

BASIC PRINCIPLES: HDM and DRP phenomema

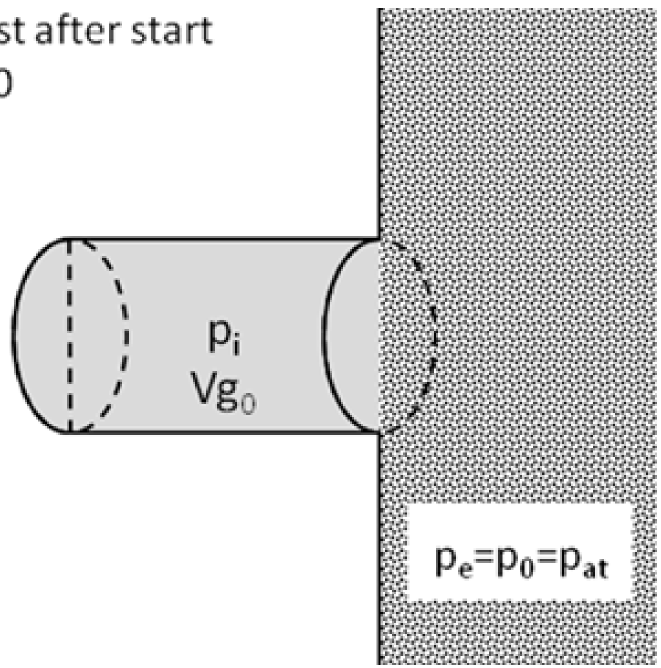


Int J Mol Sci. 2014 Sep; 15(9): 16577–16610.

BASIC PRINCIPLES: HDM and DRP phenomema

- Ideal cylindrical pore of constant diameter, from a porous solid food submerged in a liquid.

Situation just after start process, t=0



$$\varepsilon_e = \frac{V_{g0}}{V_0}$$

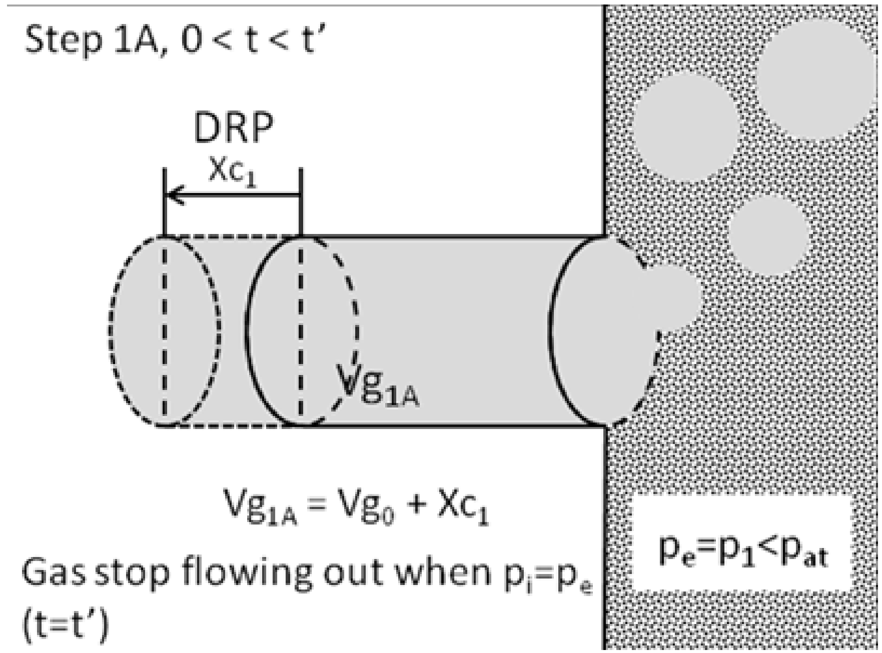
total gas volume

initial sample volume

Effective porosity

- After immersion of the material in solution (t_0), the pressure inside (p_i) and outside (p_e) the capillary are equal to atmospheric pressure ($p_i = p_e = p_{at}$).
- The initial volume of the capillary (Vg_0) is filled with gas.

BASIC PRINCIPLES:



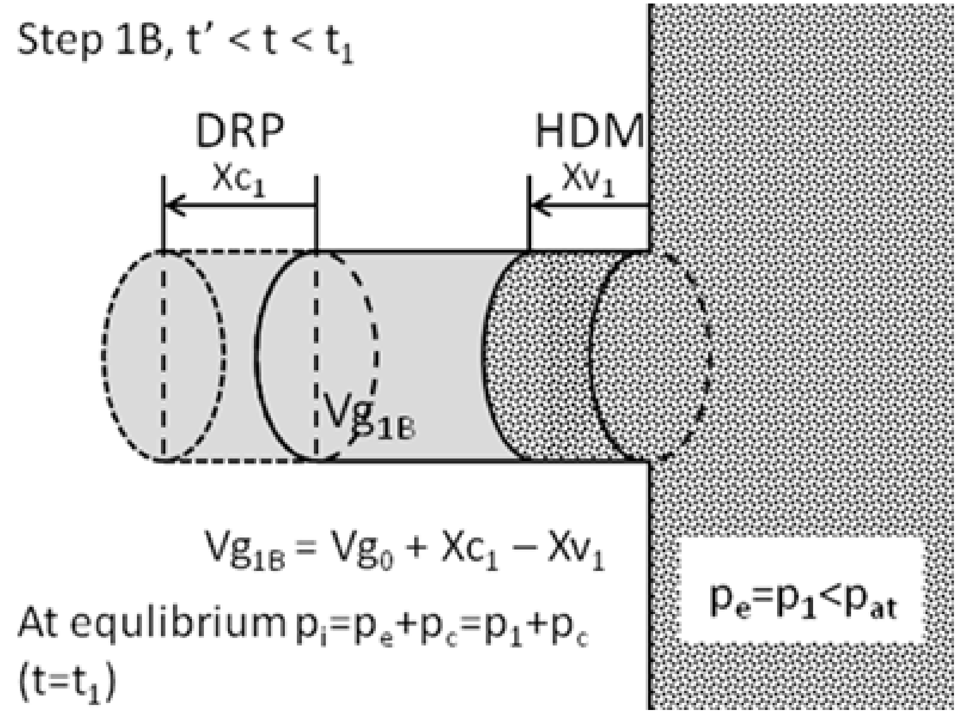
First phase of the VI process (t'): the pressure is reduced ($p_1 < p_{at}$)

As a result:

- the gas in the capillary expands partially flowing out and and the volume of gas into the pore has been increased by Xc_1 as a consequence of solid matrix deformation (DRP). The volume of the capillary becomes equal $Vg_{1A} = Vg_0 + Xc_1$
- This stage lasts until pressure equilibrium (pi = pe) is reached

Thus, **In the first phase of the process,** the reduced pressure acting from the outside causes the deformation and expansion of the capillary, which is the first part of the deformation–relaxation phenomenon (DRP).

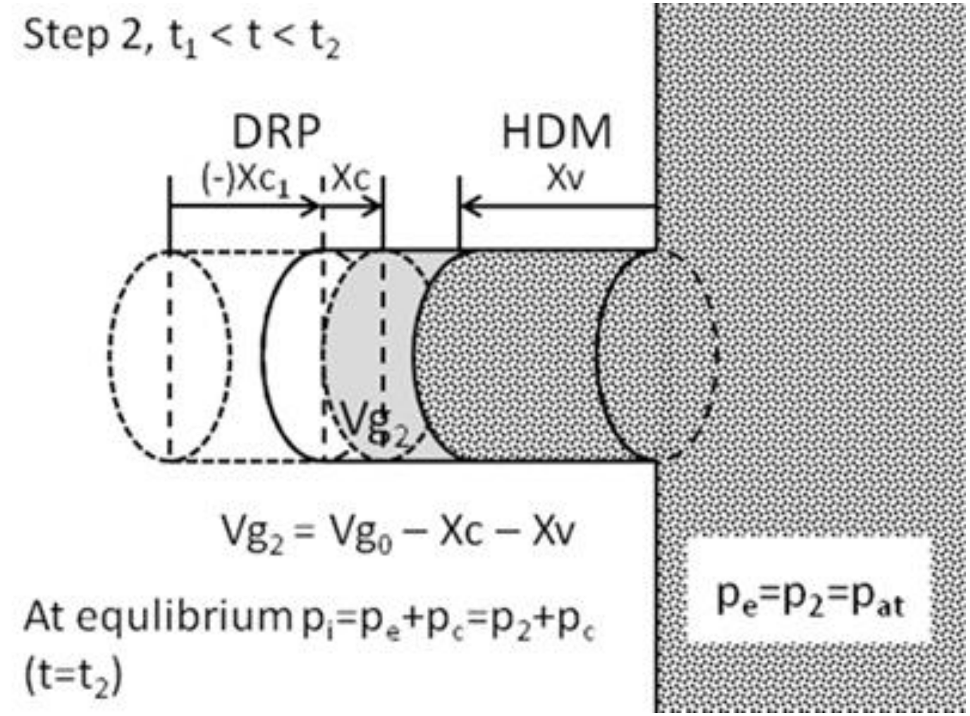
During the vacuum period (t_1), the HDM occurs



- External liquid begins to enter the pore as an effect of capillary pressure;
- The remaining gas compresses and an equilibrium situation is reached pi=p_1 +p_c ($t=t_1$).
- Non-occurrence of DRP is assumed between t' and t_1;
- At equilibrium, the gas volume has diminished and the same volume of liquid has been penetrated by HDM (Xv_1).

Thus, during t_1, the capillary starts to be partially filled with liquid, as a result of the HDM. The pressure inside the capillary increases slightly due to the compression of the gas still present in the pore, while the free volume inside the capillary decreases to the value Vg_{1B}

Second phase of vacuum impregnation: the pressure returns to the atmospheric value.



This causes:

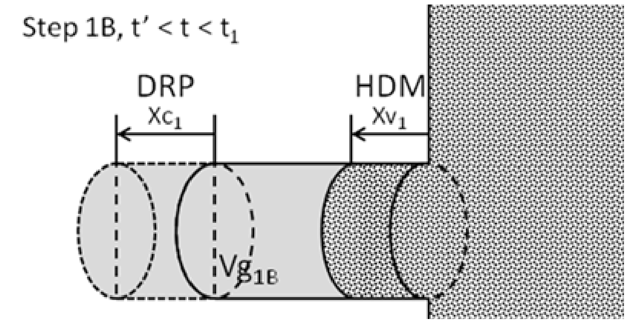
- relaxation phase (RP); the capillary shrinks to an even greater extent than before the start of the process and the volume of gas into the pore has been decreased by $Xc_1 + Xc$
- At the same time, as a result of the action of capillary pressure and decompression, an intensive inflow of liquid from the outside to the inside of the capillary is observed and the final gas volume decreases to Vg_2

Note: The relaxation phase is particularly important from the practical point of view, since tissue impregnation occurs at this stage.
Removal of vacuum should not be too rapid, since the excessively fast pressure equalization may lead to closure of the capillary vessels and HDM inhibition.

The value of each variable, referring to the ideal pore, may be extended to overall sample volume by multiplying by the effective porosity ε_e

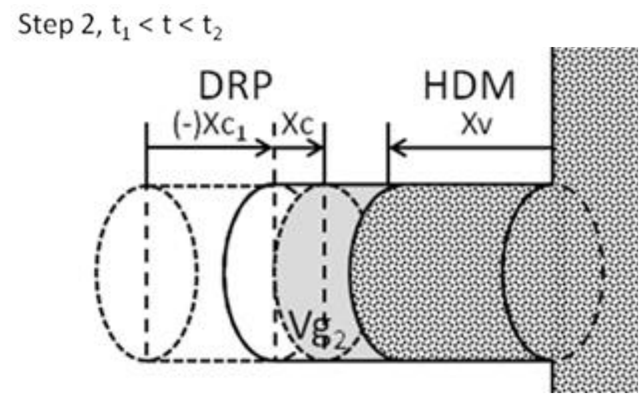
Impregnation at t_1 ← $X_1=\varepsilon_e Xv_1$

Deformation at t_1 ← $\gamma_1=\varepsilon_e Xc_1$



Impregnation at t_2 ← $X=\varepsilon_e Xv$

Deformation at t_2 ← $\gamma=\varepsilon_e Xc$



MEAN VALUES OF IMPREGNATION, DEFORMATION AND EFFECTIVE POROSITY

Product	X_1	se	γ_1	se	X	se	γ	se	ε_e	se
G.Smith apple	-4.2	0.3	1.7	0.3	15	2	-0.6	1.2	16.6	1.2
Red Chief apple	-5.0	0.4	2.1	0.4	12.2	1.8	-2.4	1.0	15.5	1.1
Golden apple	-2.7	0.3	2.8	0.2	8.9	1.2	-6.0	0.5	15.9	1.2
Mango	0.9	0.2	5.4	0.5	14.2	0.5	8.9	0.4	5.9	0.4
Strawberry	-2.1	0.2	2.9	0.4	0.2	0.7	-4.0	0.6	4.8	0.3
Kiwi	-0.2	0.2	6.8	0.6	0.89	0.14	0.8	0.5	0.5	0.5
Peach	-2.29	0.13	2.0	0.3	5.6	1.0	2.1	0.4	2.9	0.4

X_1 negative value: loss of native liquid from intercellular spaces

Journal of Food Process Engineering 21 (1998) 59-73.

FACTORS AFFECTING VI

During VI, the porous fraction composed by the intercellular spaces, is filled by an external solution to a degree that depends on various factors including:

- **PROCESS CONDITIONS:**
  - sub-atmospheric pressure level (it needs to be optimised as a function of the processed product)
  - process duration (vacuum time; post vacuum time)
  - Temperature, solution amount to product ratio, mixing
- **OSMOTIC PRESSURE, INTERFACIAL TENSION AND VISCOSITY OF THE IMPREGNATION FLUID**
- **SIZE, SHAPE AND EFFECTIVE POROSITY OF THE SAMPLES**

affects the capillary pressure of fluids within the porous and its response to mechanical stress

FACTORS AFFECTING VI

- **TISSUE STRUCTURE** (pore size and distribution)

It plays a very important role, not only due to the total porosity, but also regarding the size and shape distribution of pores as well as their communications between themselves and with the external liquid.

e.g. strawberries exhibit a negligible impregnation level, even though this type of fruit showed greater porosity fraction in comparison with kiwi fruit and peach (Salvatori *et al.*, 1998)

These differences can be attributed to the microscopic properties of the strawberry tissues, such as high tortuosity of the internal pathways and/or size and shape of pores which hindered the influx of the external solution.

FACTORS AFFECTING VI

- **TISSUE MECHANICAL PROPERTIES** → deformation-relaxation phenomenon may be reduced by tissue rigidity

N.B. For very rigid tissues vacuum impregnation process can negatively modified the quality of the product.

VI: MAIN APPLICATIONS

1. FOOD FORTIFICATION: enrichment with nutrients and bioactive compound (e.g., polyphenols, probiotics, micronutrients)

2. STABILIZATION and FOOD SHELF LIFE EXTENSION: enrichment with substances capable to reduce the pH and a_w , inhibitors of enzymatic browning, enzymes

3. MODIFICATION OF SENSORY ATTRIBUTES: introduction of sugar, flavours, acids, salt

4. SALTING, OSMOTIC DEHYDRATION

5. ADDITION OF THERMO AND CRYOPROTECTANTS

6. MODIFICATION OF THE THERMAL and MECHANICAL PROPERTIES: introduction of firming agents or compounds for softening inhibition)

Use of vacuum impregnation for vegetable and fruit products

APP: Acidification/pH reduction

- A major factor influencng microbiological safety of products is connected with pH.
- Lowering of pH value reduces thermal resistance of microorganisms and their growth rate, while it also prevents out-growth of spores.
- Traditional acidification: blanching in water or soaking using the difference in the concentration of hydrogen ions between the solution and raw material as the driving force of this process. It is frequently a long-term process, particularly in the case of soaking.
- Vacuum impregnation: the tissue porosity is enhanced through expansion of gas trapped in pores thus a greater volume of raw material is available in the impregnation process during the restoration of atmospheric pressure.

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Acidification/ pH reduction

Reduction in the pH of vegetables by vacuum impregnation: A study on pepper

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Table 1
Experimental design.

Vacuum period t_1 (min)[a]	Relaxation period t_2 (min)		
2	10	15	30
5	10	15	30

[a] The vacuum period was applied at both 200 and 400 mbar.

Main results:

- VI with lactic acid solution increased the acidification degree to a greater extent than processing carried out at atmospheric pressure.
- Vacuum level was found to be the most important variable influencing the variations in total mass and pH.
- Vacuum impregnation led to an increase in diffusion rate of hydrogen ions into the vegetable tissue due to the increase in contact area between acid solution and cells.
- Direct correlation between pH reduction and vacuum and relaxation times
- Results proved that vacuum impregnation process is a useful technique to improve acidification treatments of vegetables.

APP: pH reduction (other cases of study)

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
mushrooms (*Agaricus bisporus*) (cut in half)	lactic acid solution (pH 3.05)	solution:product ratio of 8:1; p_1 20 and 40 kPa t_1 2 min t_2 20, 40, 60, 120, 240, 300, 360 and 720 min	increase of the acidification degree in mushrooms
zucchini (slices of 1.5 cm in thickness and a diameter of 2.0 cm; the average weight of each slice was 5 g)	lactic acid solution (pH 2.70)	solution:product mass ratio of 8:1; p_1 20 and 40 kPa t_1 2 min t_2 20, 40, 60, 120, 240, 300, 360 and 720 min	increase of the acidification degree in zucchini slices

Derossi, A.; de Pilli, T.; Severini, C. Application of pulsed vacuum acidification for the pH reduction of mushrooms. *LWT Food Sci. Technol.* **2013**, *54*, 585–591.

Derossi, A.; de Pilli, T.; la Penna, M.P.; Severini, C. pH reduction and vegetable tissue structure changes of zucchini slices during pulsed vacuum acidification. *LWT Food Sci. Technol.* **2011**, *44*, 1901–1907.

APP: Reduction of Water Activity (aw)

Vacuum impregnation may be used to reduce water activity by the addition of water binding molecules (sugars, salts,…)

EFFECTS:
FOOD STABILIZATION
(decrease of reaction rates)

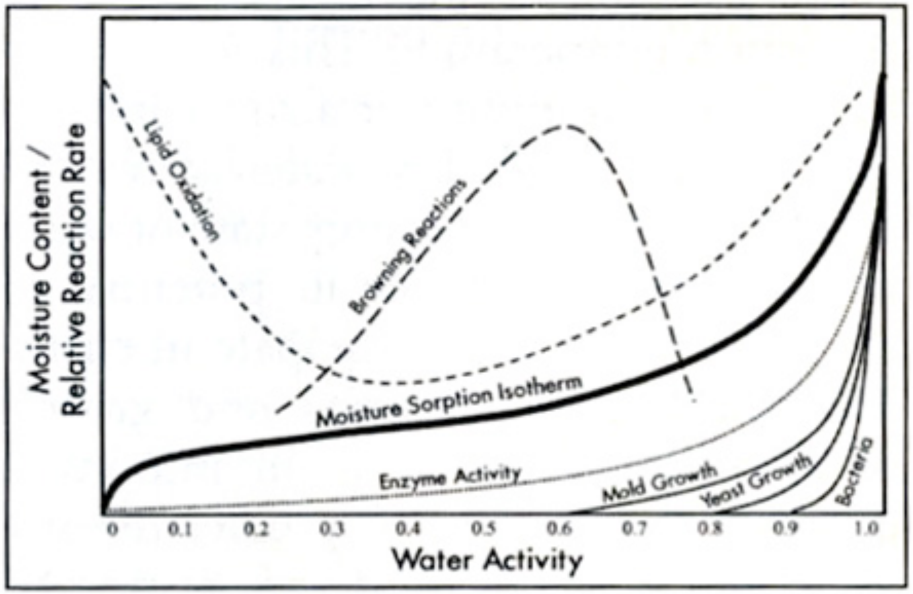

Figure 1. Water activity stability map. (Adapted from Labuza).[8]

<u>Impregnation agents:</u>
hypertonic sugars solutions (55° or 65° Brix), hypertonic high-fructose corn syrup (50%), salts (mostly for meat products)

APP: Reduction of Water Activity (aw)

OTHER APPLICATIONS: **OSMOTIC DEHYDRATION.**

Vacuum osmotic dehydration(VOD) or Pulsed vacuum osmotic dehydration (PVOD) improve capillary flow and mass exchange and allow to fill intracellular spaces with osmotic solution initiating osmotic dehydration.

Note I:

- The solid content increase in tissue is inversely proportional to the concentration of osmotic solution ➔high viscosity hinders penetration of the solution into tissues resulting in water loss.

APP: Reduction of Water Activity (aw)

NOTE II: VOD and PVOD are usefull drying and freezing pretreatments

- ➢IN DRYING: moisture content reduction by VOD and PVOD allow the shortening of drying time➔improvement of dried material quality

- ➢IN FREEZING, moisture content reduction by VOD and PVOD reduce the content of freezable water and provide a more stable product as a result of an increase in glass transition temperature at maximum cryoconcentration of the liquid phase of the product ➔lower drip loss at thawing and improvement of texture in frozen fruit and vegetables.

Examples of VI applications to modify physico-chemical and sensory attributes

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
peppers (slices of 15 cm in length and 1 cm in width)	lactic acid solution (pH 2.70)	solution:sample mass ratio of 5:1; p_1 20 or 40 kPa t_1 2 or 5 min t_2 10, 15 and 30 min	increase of the acidification degree in peppers
mushrooms (*Agaricus bisporus*) (cut in half)	lactic acid solution (pH 3.05)	solution:product ratio of 8:1; p_1 20 and 40 kPa t_1 2 min t_2 20, 40, 60, 120, 240, 300, 360 and 720 min	increase of the acidification degree in mushrooms
zucchini (slices of 1.5 cm in thickness and a diameter of 2.0 cm; the average weight of each slice was 5 g)	lactic acid solution (pH 2.70)	solution:product mass ratio of 8:1; p_1 20 and 40 kPa t_1 2 min t_2 20, 40, 60, 120, 240, 300, 360 and 720 min	increase of the acidification degree in zucchini slices
papayas (cut into 4 × 2.5 × 0.5 cm pieces (length × width × thickness))	55% and 65% (*w*/*w*) sucrose solution	p_1 5 kPa t_1 10 min at 30 °C	decrease of a_w
strawberry	65% (*w*/*w*) sucrose solution	steam blanching or microwave and osmotic dehydration at atmospheric pressure or pulsed vacuum treatments p_1 5 kPa t_1 5 min at 30 °C	decrease of a_w
rabbiteye blueberries	aqueous sucrose solutions (600 g/kg)	solution:product ratio of 1:1; p_1 88 kPa	shortenning of dehydratation time in comparison with soaking at atmospheric pressure
plum (cut in slices of 4 × 1 × 1 cm, weighting approximately 10 g)	40°, 50° and 60° Brix sucrose solution	solution:product ratio of 10:1; t_1 10 min	new product with good visual quality and satisfactory shrinkage

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APP: CHANGES IN THERMAL PROPERTIES

- Vacuum impregnation seems to be a suitable technique for modifying the thermal properties of fruit and vegetables

- The replacement of gas in intracellular spaces with the impregnant solution, the slight increase in density and the structure modifications can increase the thermal conductivity and thermal diffusion coefficient of the matrices thus improving the efficiency of heat conduction

<u>Thermal treatment shortening (➔freezing, blanching, pasteurization or sterilization)</u>

<u>Quality enhancemnet of the final products. *i.e.*, dried or frozen material, and fruit and vegetable preserves.</u>

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JOURNAL OF FOOD ENGINEERING

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Changes in thermal properties of apple due to vacuum impregnation

J. Martínez-Monzó, J.M. Barat, C. González-Martínez, A. Chiralt, P. Fito *

EXPERIMENTAL PLAN:
Vacuum impregnation (VI) in isotonic sucrose solution; 50 mbar for 10 min; 20 min post vacuum time; T=30, 40, 50°C

Thermal properties of impregnated (VI) and non-impregnated (NVI) samples[a]

	$\alpha \times 10^7$ (m^2/s)	K (W/m K)	$c_P \times 10^{-3}$ (J/kg K)
NVI			
30°C	1.36 ± 0.05	0.42 ± 0.03	3.81 ± 0.02
40°C	1.51 ± 0.14	0.48 ± 0.06	3.86 ± 0.03
50°C	1.72 ± 0.05	0.54 ± 0.02	3.85 ± 0.03
VI			
30°C	1.40 ± 0.06	0.57 ± 0.02	3.81 ± 0.02
40°C	1.56 ± 0.04	0.64 ± 0.02	3.86 ± 0.03
50°C	1.65 ± 0.03	0.67 ± 0.01	3.85 ± 0.03

[a] At 25°C: ρ_b(NVI) = 813 kg/m^3; ρ_b(VI) = 986 kg/m^3.

$\alpha = K/\rho c_p$

MAIN RESULTS:
- Thermal properties were measured in non-impregnated (NVI) and impregnated samples
- Thermal diffusivity (α) changes due to VI were relatively low (2-4%)
- Thermal conductivity significantly increased (15-24%) due to change in fruit porosity, density and composition.

APP: STRUCTURE IMPROVEMENT

Vacuum-impregnation is faster and more homogeneous than soaking and allow to improve the firmness of thermally treated fruit.

STRATEGIES:

1) Introduction of calcium ions inside the tissue. The mechanism of this process is explained by binding of calcium ions by carboxyl groups of pectin which leads to gel formation at low pH

VI➔more effective impregnation of tissue with calcium ions than blanching or soaking

2) Addition of exogenous PME in combination with calcium.

PME catalyzes the hydrolysis of methyl groups esterifying cell wall pectins➔ release of free carboxylic acids➔interaction with calcium ions➔ strengthening the cell wall structure and firmness increase.

3) Addition of structuring and firming molecules: maltodextrines, HM pectines

APP: COLOUR PRESERVATION

One of the phenomenon limiting the quality of plant products is enzymatic browning which is caused by the activity of polyphenol oxidases enzymes which catalyze the oxidation of phenolic compounds with oxygen.

<u>Traditional inhibition method for colour changes:</u> blanching or soaking of raw material in a solution of inhibitors.

<u>Innovative inhibition strategy:</u> VI of the tissue with a solution of enzymatic browning inhibitors and removal of oxygen from the intracellular space.

The effectiveness of VI is connected with the tissue structure, the type of inhibitors used and the conditions of vacuum impregnation.

Inhibitors: citric or ascorbic acids, sulfur compounds (allergenic), EDTA, calcium lactate, cysteine, 4-hexylresorcinol, chitosan

APP: Probiotics introduction

Probiotics: selected strains of living bacteria, which exhibit an advantageous effect on human health after consumption.

Well-known probiotics: **lactic acid bacteria belonging to the genera *Bifidobacterium* and *Lactobacillus***.

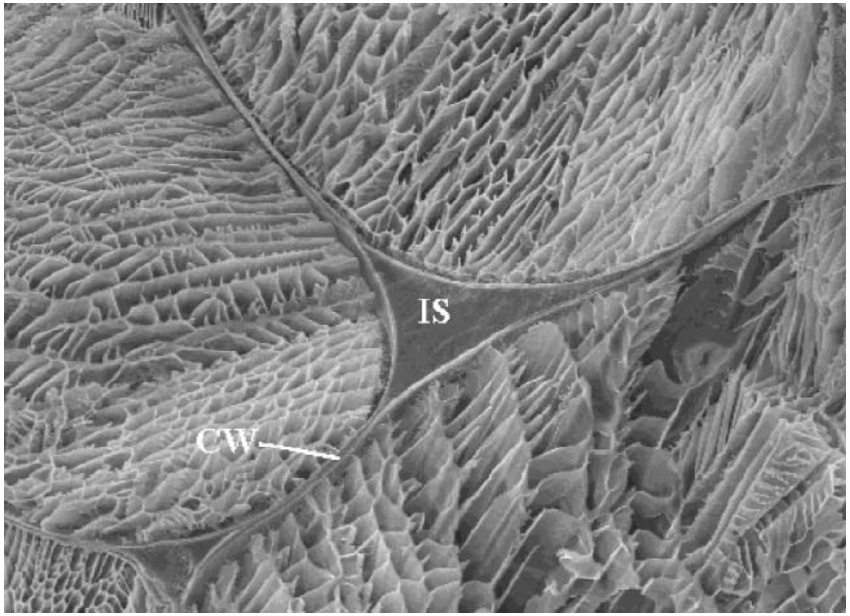
N.B. Metabolites of probiotic bacteria stimulate the immune system, promote peristalsis and intestinal secretion, while also exhibiting antibacterial and antiviral action, thus inhibiting the development of pathogenic bacteria and reducing the amount of produced toxins.

Attempts to apply vacuum impregnation in order to introduce probiotics to the fruit and vegetable matrix were associated with the extension of the range of probiotic foodstuffs.

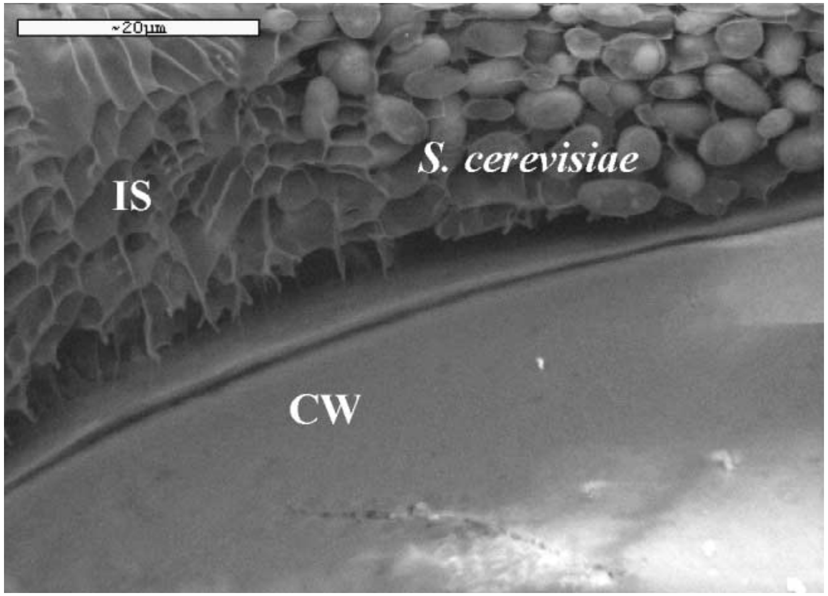
- addition of probiotics to dairy products → two basic disadvantages → lactose intolerance and high cholesterol content.

reason for the search for new possibilities to add probiotics, e.g., to raw materials, such as fruit and vegetables

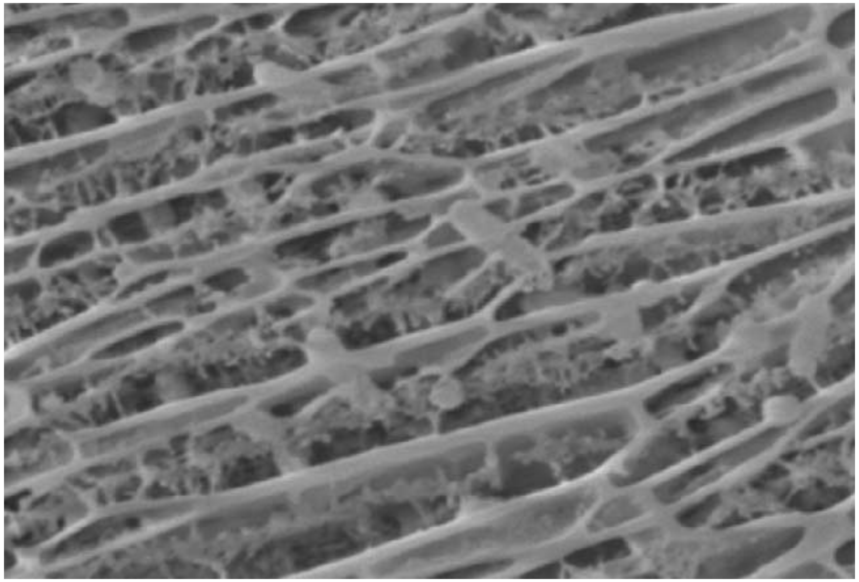
Development of probiotic-enriched dried fruits by vacuum impregnation, Betoret et al., J. Food Eng. 2003, 56, 273–277.



Fresh apples

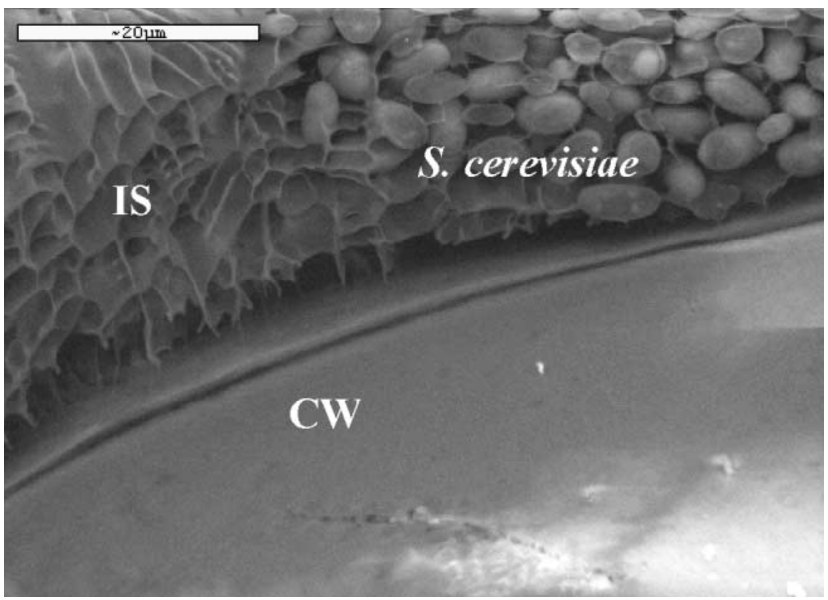


S. Cerevisiae in intercellular spaces of vacuum impregnated apples

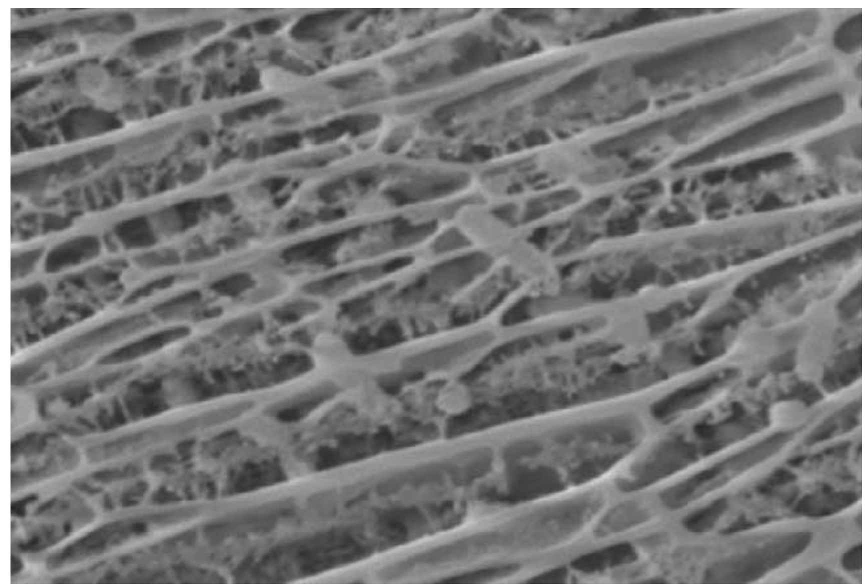


L. Casei in intercellular spaces of vacuum impregnated apples

MAIN RESULT: Impregnation facilitated the effective introduction of probiotics to apple tissue, providing the content of microorganisms in the product after convection drying (air drying) at 106–107 CFU/g.
This is equivalent to the level of bacteria in dairy products.



S. Cerevisiae in intercellular spaces of vacuum impregnated apples



L. Casei in intercellular spaces of vacuum impregnated apples

APP: Probiotics introduction

Raw Material	Vacuum Impregnation Solutions Composition	Process Parameters	Effect	References
apple cylinders	apple juice with an addition of microorganisms *Saccharomyces cerevisiae*, milk with an addition of *Saccharomyces cerevisiae* and *Lactobacillus casei*	p_1 5 kPa t_1 10 min t_2 10 min	over 10^6 CFU/g *Lactobacillus casei* in air dried (40 °C) product	[4]
pieces of guava and papaya	papaya and guava fruit juices (1—Extracted by blending with water, ratio 1:1; 2, 3—Extracted fruit juices containing 15° and 30° Brix, respectively) with an addition of *Lactobacillus casei* microorganisms	p_1 5 kPa t_1 5, 10, 15 min t_2 10 min	after impregnation: 10^8 to 10^9 CFU/g *Lactobacillus casei*, after drying at 40 °C for 36 h: 10^7 CFU/g *Lactobacillus casei* in impregnated fruits	[69]
apple	isotonic sucrose solution containing 10^8 CFU/g *Bifidobacterium* ssp.	p_1 14, 17, 30, 43, 57 kPa	greater incorporation at pressures of 14 and 17 kPa, levels of microorganisms over 10^7 CFU/g	[70]
apple cylinders cv. Granny Smith	sucrose isotonic solution containing microorganisms *Saccharomyces cerevisiae*, *Lactobacillus acidophilus* and *Phoma glomerata*	p_1 10, 17, 30, 43, 57 kPa (one vacuum pulse of 2 min)	increase by 0.36 log for *Saccharomyces cerevisiae*, 0.73 log for *Lactobacillus acidophilus* and 1.07 log for *Phoma glomerata* for vacuum impregnated sample in comparison to soaking sample	[71]
apple slices (cv. Fuji)	apple juice diluted with pre-sterilized distilled water (1:1, *v*/*v*, pH 5–5.2) with an addition of *Lactobacillus rhamnosus* (ATCC 7469, in 1:1 (*v*/*v*) glycerol frozen cultures)	p_1 20 kPa t_1 15 min t_2 15 min apple slices were dried by air drying, freeze drying, and a combination of air drying + REV drying	after vacuum impregnation: 10^9 CFU/gof tissue	[72]
apples cv. Granny Smith (disk-shaped samples)	mandarin juice (pH 5, 8–6, 0) with an addition of *Lactobacillus salivarius* (*Salivarius* spp.)	p_1 5 kPa t_1 10 min t_2 10 min	after vacuum impregnation: $1.51 \cdot 10^8$ CFU/g *Lactobacillus salivarius* spp. *Salivarius*; the highest microbial content: after 24 h incubation period, pH 6	[73]

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Innovative Food Science and Emerging Technologies

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PRODUCTION OF FUNCTIONAL PRODUCT

Technological development and functional properties of an apple snack rich in flavonoid from mandarin juice

E. Betoret [a], E. Sentandreu [b], N. Betoret [a], P. Codoñer-Franch [c], V. Valls-Bellés [c], P. Fito [a,*]

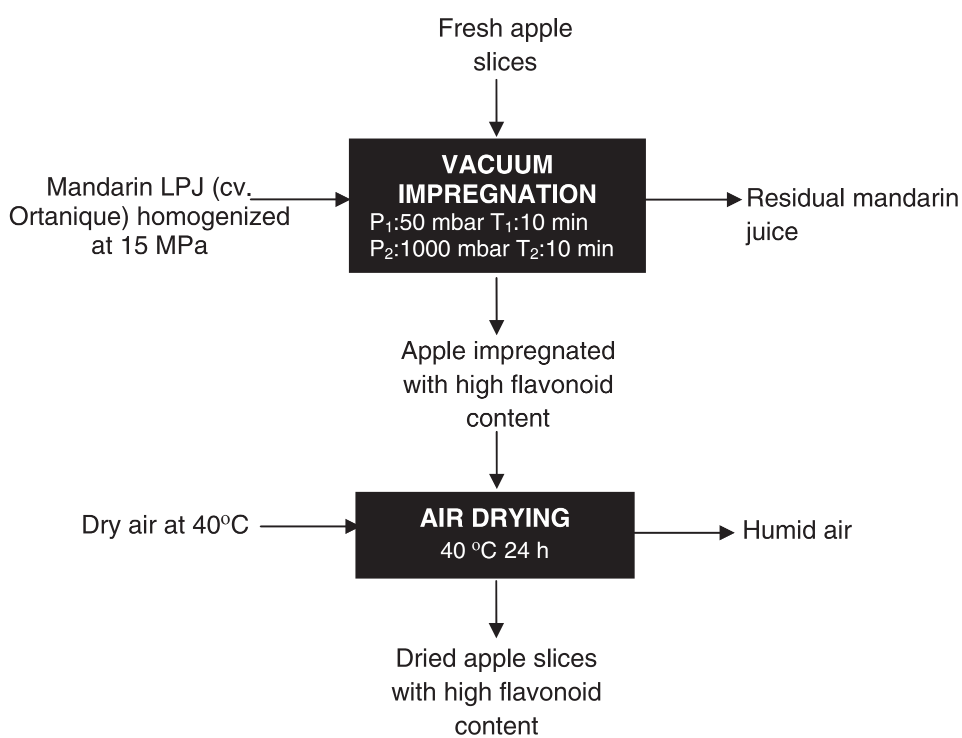

Fig. 1. Production of flavonoid-enriched, dried apple slices.

MAIN RESULTS:

- Forty grams of the final product made using mandarin juice homogenized at 15 MPa could provide the same quantity of hesperidin as 250 mL of fresh mandarin juice.
- The bioactive compounds of mandarin LPJ was successfully incorporated into the structural matrix of fresh apple slices with no negative effects on their antiradical capacities following impregnation and air drying.

ADDITION OF CRYOPROTECTANTS

Available online at www.sciencedirect.com
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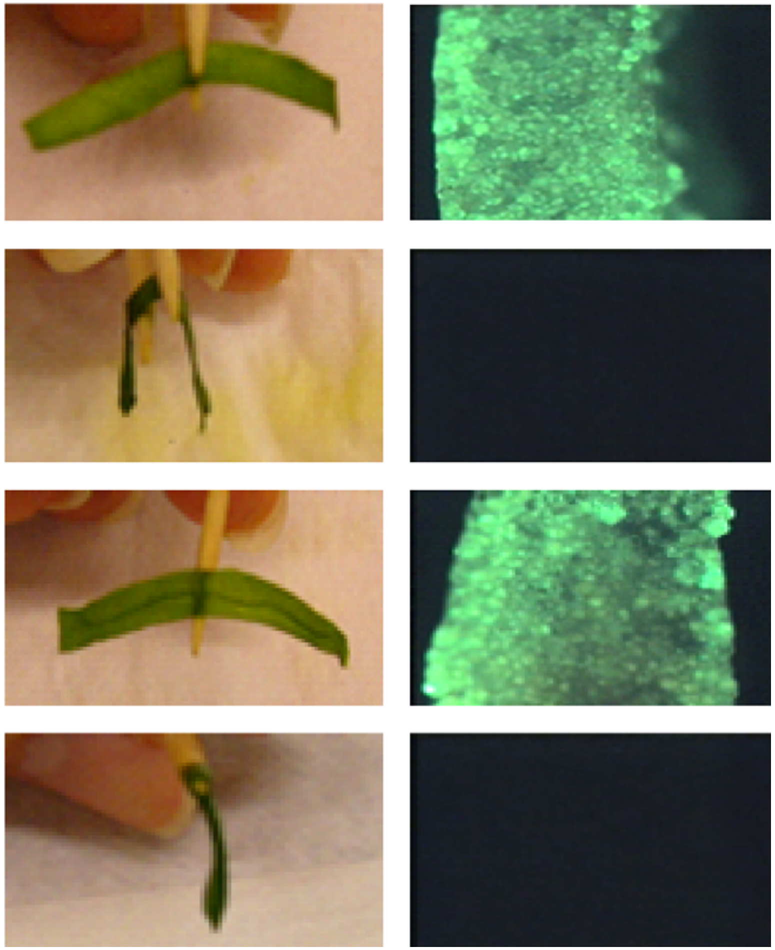
Research note

Pulsed electric field in combination with vacuum impregnation with trehalose improves the freezing tolerance of spinach leaves

Pui Yeu Phoon [a,1], Federico Gómez Galindo [b,*], António Vicente [b], Petr Dejmek [a]

EXPERIMENTAL PLAN:

1. Spinach samples were treated with ten trains of bi-polar, rectangular electric field pulses with a electric field strength of 580 V/cm;
2. immersion in 40% trehalose solution under vacuum for 20 min+ 2.5 h at atmospheric pressure;
3. immersion in deionised water at 4 °C overnight;
4. frozen in liquid nitrogen 5)thawed in water at room temperature.



Fresh spinach

Fresh spinach after freezing and thawing

PEF + VI spinach after freezing and thawing

VI spinach after freezing and thawing

Wilting test: samples turgidity

microscopic observations: fluorescein diacetate used to identify viable cells (bright fluorescence).

MAIN RESULTS:
impregnation with trehalose by the combined actions of electric fields and vacuum impregnation drastically improved the freezing tolerance of the spinach PEF +VI probably allowed trehalose uptake in extracellular and intercellular spaces.

Examples of VI applications to modify physico-chemical and sensory attributes

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
peppers (slices of 15 cm in length and 1 cm in width)	lactic acid solution (pH 2.70)	solution:sample mass ratio of 5:1; p_1 20 or 40 kPa t_1 2 or 5 min t_2 10, 15 and 30 min	increase of the acidification degree in peppers
mushrooms (*Agaricus bisporus*) (cut in half)	lactic acid solution (pH 3.05)	solution:product ratio of 8:1; p_1 20 and 40 kPa t_1 2 min t_2 20, 40, 60, 120, 240, 300, 360 and 720 min	increase of the acidification degree in mushrooms
zucchini (slices of 1.5 cm in thickness and a diameter of 2.0 cm; the average weight of each slice was 5 g)	lactic acid solution (pH 2.70)	solution:product mass ratio of 8:1; p_1 20 and 40 kPa t_1 2 min t_2 20, 40, 60, 120, 240, 300, 360 and 720 min	increase of the acidification degree in zucchini slices
papayas (cut into 4 × 2.5 × 0.5 cm pieces (length × width × thickness))	55% and 65% (*w*/*w*) sucrose solution	p_1 5 kPa t_1 10 min at 30 °C	decrease of a_w
strawberry	65% (*w*/*w*) sucrose solution	steam blanching or microwave and osmotic dehydration at atmospheric pressure or pulsed vacuum treatments p_1 5 kPa t_1 5 min at 30 °C	decrease of a_w
rabbiteye blueberries	aqueous sucrose solutions (600 g/kg)	solution:product ratio of 1:1; p_1 88 kPa	shortenning of dehydratation time in comparison with soaking at atmospheric pressure
plum (cut in slices of 4 × 1 × 1 cm, weighting approximately 10 g)	40°, 50° and 60° Brix sucrose solution	solution:product ratio of 10:1; t_1 10 min	new product with good visual quality and satisfactory shrinkage

Int J Mol Sci. 2014 Sep; 15(9): 16577–16610.

Examples of VI applications to modify physico-chemical and sensory attributes

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
apples cv. (cultivar). Granny Smith (cylindrical samples (2 cm height and diameter))	rectified grape must (hypertonic solutions: 65°, 50° and 30° Brix) and 3% (*w*/*w*) high methoxyl pectin solutions	p_1 5 kPa t_1 5 min t_2 25 min in higher solution viscosity t_2 55 min	improvement of mechanical and structural properties of tissue, notable reduction of freezable water which could improve fruit resistance to freezing damage
strawberry (10 mm slices)	50% (*w*/*w*) high fructose corn syrup or 3% (*w*/*w*) high methoxyl pectin solution containing calcium and/or zinc	p_1 7 kPa t_1 15 min t_2 30 min	improvement of textural quality and reduced drip loss of frozen-thawed strawberries
spinach (rectangular 3.0 cm long, 0.5 cm wide and 0.06 cm thick)	40% (*w*/*w*) trehalose solution	pulsed electric fields (580 V/cm) in combination with vacuum impregnation p_1 86 kPa t_1 20 min t_2 150 min	improvement of freezing tolerance of spinach leaves
apple samples cv. Granny Smith (cylindrical samples (8 cm height and 2 cm diameter))	sucrose isotonic solution	p_1 50 kPa t_1 10 min t_2 20 min	increase of thermal conductivity
zucchini (slices 0.5-cm thick)	maltodextrine solution (7.5%–9%, 10%), NaCl (0%–5%) and $CaCl_2$ (0–1000 mM)	product:solution ratio of 1:3.3; p_1 2.5 kPa t_1 10 min t_2 30 min	improvement of solute and water gain and limitation of textural and microstructural changes
eggplant, carrot, oyster mushroom	33 g sucrose and 20 g calcium lactate solution in isotonic solution	p_1 5 kPa t_1 10 min t_2 10 min	notable impact on mechanical behaviour of eggplant and carrot, no effects in oyster mushroom

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Examples of VI applications to modify physico-chemical and sensory attributes

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
apple samples cv. Granny Smith	$CaCl_2$ solution (0.6%, 2.0% or 4.0% (*w/w*))	p_1 9.3 and 59.9 kPa t_1 4 min t_2 5 min	improvement of texture
apple cv. Jonagold (cut into 1 cm thick slices)	10 mg/L ascorbic acid, 0.05 mg/L 4-hexylresorcinol, 5 mg/L calcium chloride and 200 mg/L sucrose	p_1 8 kPa t_1 5 min t_2 5 min	the same effect of dipping and vacuum impregnation regarding hardness
apples cv. Granny Smith (1 cm cubes) strawberries (cut in halves) and raspberries	high methylated pectin solution preparation up to 3% (*w/w*) and/or $CaCl_2$, up to 6.5% (*w/w*)	p_1 6.6 kPa t_1 2 min	limitation of loss in fruit firmness following pasteurization
strawberries (cv. Elsanta and Darselect) (cut in halves)	high methylated pectin (from *Aspergillus aceleatus*) containing 100 U/mL, 0.5% (*w/w*) $CaCl_2 \cdot 2H_2O$ 1% and 3% (*w/w*) of apple pectin	p_1 1 kPa t_1 5 min	limitation of structural damage during subsequent rapid freezing processes
peaches (cut in halves)	pectin methylesterase together with $CaCl_2$ (100 mg/L)	p_1 85 kPa t_1 30, 60, 90, 120 min	increase of firmness in canned peaches
eggplants (slices of 1 cm trick)	pectinmethyl-esterase derived from *Aspergillus niger* and extracted from orange and grapefruit and 4000 ppm $CaCl_2 \cdot 2H_2O$	1st method: p_1 68 kPa t_1 15 min at 30 °C 2nd method: pulsed vacuum impregnation p_1 85 kPa t_1 5 min release vacuum to atmospheric pressure for 1 min reapply vacuum for 5 min and release for 5 min	increase of firmness in impregnated eggplants

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Examples of VI applications to modify physico-chemical and sensory attributes

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
watercress (leaves were selected diameter 1.4 cm)	winter flounder antifreeze protein type I solution (1 mg/100 mL AFP-I ultra pure water)	p_1 51, 58, 68, 85 and 101 kPa t_1 5 min	smaller ice crystals in AFP-I impregnated (58 kPa, for 5 min) frozen samples
strawberry	12 g/100 g trehalose solution; 0.2 g/100 g solution unpasteurized cold acclimated winter wheat grass extract as a source of AFP and 12 g/100 g trehalose and 0.2 g/100 g unpasteurized cold acclimated winter wheat grass extract	p_1 86 kPa t_1 5 min	improvement of freezing tolerance of strawberry
strawberry slices	$CaCl_2$ solution (1, 10, 100 mM); spermine solution (1, 10, 100 mM); spermidine (1, 10, 100 mM); putrescine (1, 10, 100 mM);	p_1 16.9 kPa t_1 8 min	effect of spermine and spermidine on the increase of firmness, whereas putrescine was not as effective
carrots (cv. Nantesa) slices (20-mm diameter, 10 mm thick)	chitosan (1%, *w*/*v*) dispersed in aqueous solution of glacial acetic acid (1%, *w*/*v*), at 40 °C	p_1 5 kPa t_1 4 min t_2 2 min	improvement of sample resistance to water vapor transmission, better preservation of color and mechanical response during cold storage
pineapple (slices 1 cm thickness)	chitosan- or casinate-based film-forming emulsions	ratio of the weight of coating solution:sample: 20:1; p_1 5 kPa t_1 3 min t_2 2 min	extension of shelf-life in pineapple-cereal system for caseinate based coating
pear (*Pirus communis* cv. *Blanquilla*) (cylinders 2 cm height, 2 cm diameter)	isotonic sucrose solution (14° Brix) containing trisodium citrate 2-hydrate, sodium L-ascorbate, ethylenediamine tetraacetic acid 2-hydrate disodium salt and calcium lactate 5-hydrate and 4-hexylresorcinol	solution: fruit ratio of 20:1; p_1 5 kPa t_1 5 min t_2 10 min	ascorbate and calcium lactate in impregnated solution were the most effective for extending the shelf life of pear

Examples of VI applications to modify physico-chemical and sensory attributes

Raw Material	Composition of Vacuum Impregnation Solutions	Process Parameters	Effect
apple cv. Jonagold (1-cm thick slices)	ascorbic acid, citric acid, 4-hexylresorcinol, sodium chloride, calcium chloride, sodium lactate, calcium lactate and sucrose solutions	p_1 7 kPa t_1 5 min t_2 5 min	effective inhibition of browning and softening of apple slices during storage by 1% ascorbic acid, 0.005% 4-hexylresorcinol, 0.5% calcium chloride, 20% sucrose in impregnated solution
button mushrooms (slice thickness was 6.5 mm with a 3 to 5 mm cap length)	2 g/100 g ascorbic acid + 1 g/100 g calcium lactate solution; 2 g/100 g citric acid + 1 g/100 g calcium lactate; 1 g/100 g chitosan + 1 g/100 g calcium lactate solution; and 1 g/100 g calcium lactate solution	p_1 6.7, 10.0, 13.3, 16.7 kPa t_1 5 and 10 min t_2 5 and 10 min	vacuum impregnation with ascorbic acid and calcium lactate at 6.7 kPa for 5 min and atmospheric restoration time of 5 min was the most effective to limit adverse changes of color in sliced button mushrooms
litchi cv. Rose	502 g/kg sucrose solution containing 4.9 g/kg cysteine + 20 g/kg ascorbic acid + 0.134 g/kg 4-hexyl resorcinol and 502 g/kg sucrose solutions also contained 20 g/kg calcium lactate and 1 g/kg potassium sorbate	p_1 76 kPa t_1 10 min t_2 10 min	samples were sensory acceptable up to 24 days
apple sticks	mass ratio of fruit:syrup was 1:17; fructose isotonic solution (14.0°–15° Brix) containing ascorbic acid (0.5% w_t/w_t) and dry, food-grade green apple flavoring (0.5% w_t/w_t)	p_1 28 kPa t_1 5 min t_2 2.5, 5, 12.5 min	aroma enrichment
olive fruits cv. Domat	NaCl (3%), NaOH (1.5%) and NaOH (1.5%) + NaCl (3%) solutions	p_1 68 kPa	shortening the duration of debittering process
apples cv. Granny Smith and Stark Delicious	higher values of hardness, crispness, juiciness and sourness in vacuum impregnated Granny Smith apples	the solution:fruit ratio was 11:1; p_1 10 kPa t_1 30 min t_2 5 min	higher values of hardness, crispness, juiciness and sourness in vacuum impregnated Granny Smith apples

p_1—vacuum pressure in the Vacuum impregnation (VI) process; t_1—time in reduced pressure; t_2—time in atmospheric pressure.

Use of vacuum impregnation
for meat, fish and dairy products

APP: SALTING

- Salting is a commonly used food preservation operation in meat, fish, dairy and some plant products, such as pickles and olives often coupled with fermentation processes.

- During salting, two main phenomena occur:

- uptake of NaCl and other possible curing compounds
- loss of water and some internal soluble solids.

- The aim of this operation is:

- To reduce product water activity a_w in order to improve its microbial, chemical and biochemical stability;
- To develop desirable flavour characteristics.

APP: SALTING

TRADITIONAL PROCESS:

- dry salting
- brining ➔also combined with other operations such as smoking, acidification, air drying, in order to obtain stable products➔product stability is promoted by aw and pH reduction and by the action of competitive microorganisms.

LIMITATIONS:

- long time processing (several days for big peaces) due to the low values of salt diffusivity at the low temperatures required to assure food safety during the operation.
- management and waste recycling of great amounts of brine
- requirement of very big salting plants to achieve a reasonable production.

APP: SALTING

SALTING by BRINE VACUUM IMPREGNATION (BVI)

EFFECTS:

- reduce salting time:
- flatter salt distribution in the product
- higher process yields (lower weight loss)
- juicier product at equal salt content.
- In cheese (e.g. cheddar) better elimination of entrapped gas and whey, thus favouring pressing and elimination of mechanical openings → close structure, mechanical eyes disappear and texture becomes smooth → lighter appearance of the cut surface.
- elimination of blood in meat products and whey in cheese obtaining a safer product due to the reduction of microbial growth.
- Reduced oxidation in fatty fish (BVI reduces the accessibility of oxygen to sample active points)

APP: SALTING

SALTING by BVI

LIMITATIONS:

- In meat, fish or cheese products, pores contain small gas phase volume entrapped in a free liquid phase ➔it can impede the gas out flow (mainly in big pieces) and hinder the mechanical equilibration process in the vacuum period.

- solid matrix more viscous than elastic➔ pores collapse during the compression step ➔ no notable impregnation occur during step 2

Mass transport phenomena and structural changes occurring throughout food brining at the different steps in vacuum impregnation processes

Process step	Mass transport phenomena	Structural changes
Product immersion at atmospheric pressure	Capillary penetration of brine (brine penetration front: BPF) Salt and water diffusion in the product liquid phase near the sample surface	Changes in aqueous environment of product components (e.g. proteins) near the sample surface: conformational changes of biopolymers
Period at vacuum	Gas and free internal liquid flow out Advance of the BPF due to the more intense capillary effects Development of salt-water concentration profiles due to diffusion phenomena coupled with brine penetration	Expansion of the pores occupied by gas Progression of conformational changes of biopolymers and changes in their water bonding capacity (WBC), according to the developed salt concentration profile
Period at atmospheric pressure	Advance of brine penetration front due to compression Coupling with the diffusional transport of water and salt	Volume reduction of matrix pores: expulsion of free liquid phase can occur Progression of conformational changes of biopolymers and changes in their WBC, according to the developed salt concentration profiles

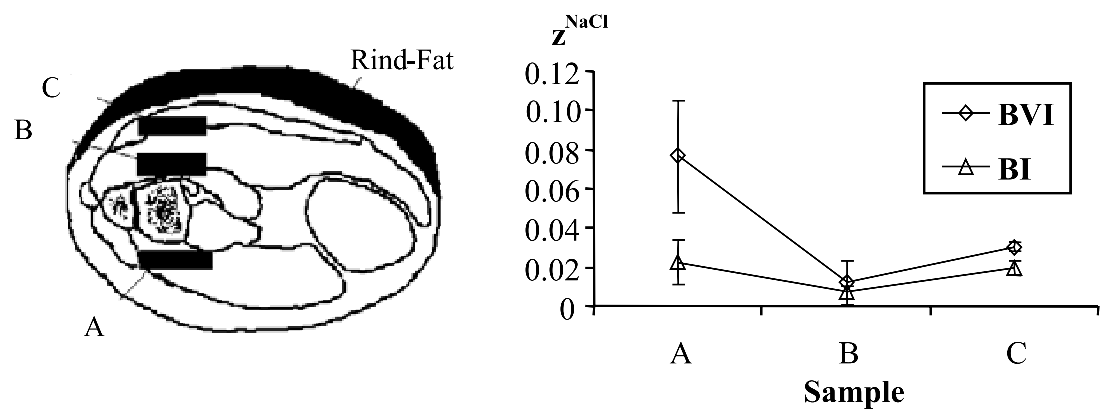

Fig. 6. Salt distribution in the internal parts (zones A–C in the widest ham cross-section) immediately after salting at 3°C for BVI (9 days at 50 mbar plus 1 day at atmospheric pressure) and for BI (15 days).

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Free fatty acid content of Manchego-type cheese salted by brine vacuum impregnation☆

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Abstract

A new salting method based on the brine vacuum impregnation of porous products was tested in Manchego-type cheese in order to assess its effect on cheese lipolysis during ripening. This new salting method would allow a faster salt diffusion and a more homogeneous initial salt distribution, and would reduce the disposal of brine. Salt-in-moisture content was evaluated in three different cheese zones during a 90-day ripening period in order to monitor salt penetration in the cheese. Lipolysis was evaluated by means of gas chromatography of individual free fatty acids in the medium and internal zones of both cheeses salted by the conventional and the new salting procedures. Free fatty acid concentration regularly increased during ripening. Short-chain free fatty acid content was higher in the internal zone of conventionally salted cheeses than in the internal and medium zones of vacuum impregnated cheeses from the first month after manufacturing, probably due to the low initial salt concentration achieved in the inner zone of conventionally salted cheeses, which can enhance both bacterial and indigenous lipase activity. Panelists considered that conventionally salted cheeses presented a more intense aroma than vacuum impregnated cheeses, though no differences in global flavor intensity were observed.

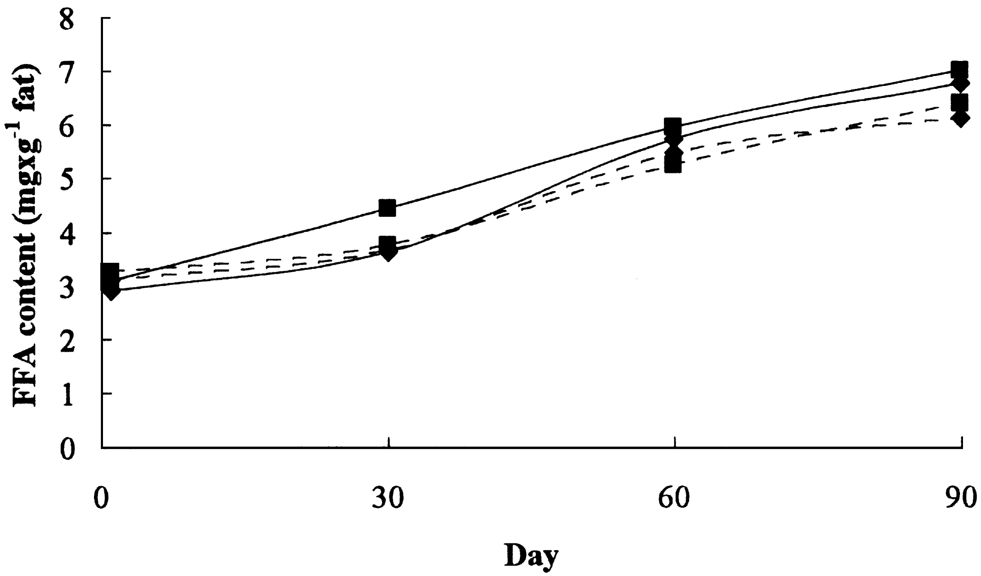

Fig. 2. Total free fatty acid evolution throughout ripening in the medium (◆) and the internal (■) zones of conventional salted (continuous line) and vacuum impregnated (discontinuous line) cheeses.

<u>MAIN RESULTS:</u>

- FFA: Internal zone of CS cheeses presents a content of short-chain FFA higher than the medium zone from the first month after manufacturing, whereas no differences are observed due to cheese zone in VI cheeses Salting procedure affects short-chain FFA release: slightly higher in the internal zone of CS cheeses than in VI cheeses (CS ➔lower inner salt concentration➔higher bacterial and endogenous lipase activity)
- SENSORY PANEL RESULTS: more intense aroma in CS cheeses than in VI cheeses; no differences in global flavour intensity.

OTHER APPLICATIONS: Salting and acidification

LWT - Food Science and Technology 74 (2016) 26–33

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Influence of vacuum application, acid addition and partial replacement of NaCl by KCl on the mass transfer during salting of beef cuts

CrossMark

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A B S T R A C T

The objective of this work was to study the influence of different salting procedures on the water loss (*WL*) and salt gain (*StG*) kinetics of beef cuts. The following procedures were evaluated: wet salting at atmospheric pressure; pulsed-vacuum wet salting (with application of one or three vacuum pulses); dry salting; pulsed-vacuum marination in brines with acetic, citric or lactic acid; and pulsed-vacuum salting in brines with partial replacement of NaCl by KCl. From the obtained experimental results, we verified that the application of three vacuum pulses during wet salting increased the *WL* by 20% and the *StG* by 15% in comparison with the wet salting at atmospheric pressure. In contrast, addition of different acids in the brine decreased the *StG* by 13–24% after 6 h of immersion. Moreover, results obtained with partial replacement of NaCl by KCl revealed that the diffusion of K^+ is faster than of Na^+ and also that KCl has a smaller capability of reducing water activity. This demonstrates the importance of adjusting salting time when using mixtures of NaCl and KCl for elaborating products with reduced Na^+ levels.

OTHER APPLICATIONS:
Incorporation of antimicrobial bacteriocines in sausage casing

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Food Control

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Antimicrobial activity of collagen casing impregnated with nisin against foodborne microorganisms associated with ready-to-eat sausage

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<u>EXPERIMENTAL PLAN:</u>

Collagen casing was impregnated with 10,000 ppm of nisin solution at vacuum pressure of -680 mmHg ; t1 (vacuum time) from 150 to 600 s and t2 (atmospheric time) from 30 to 90 s.

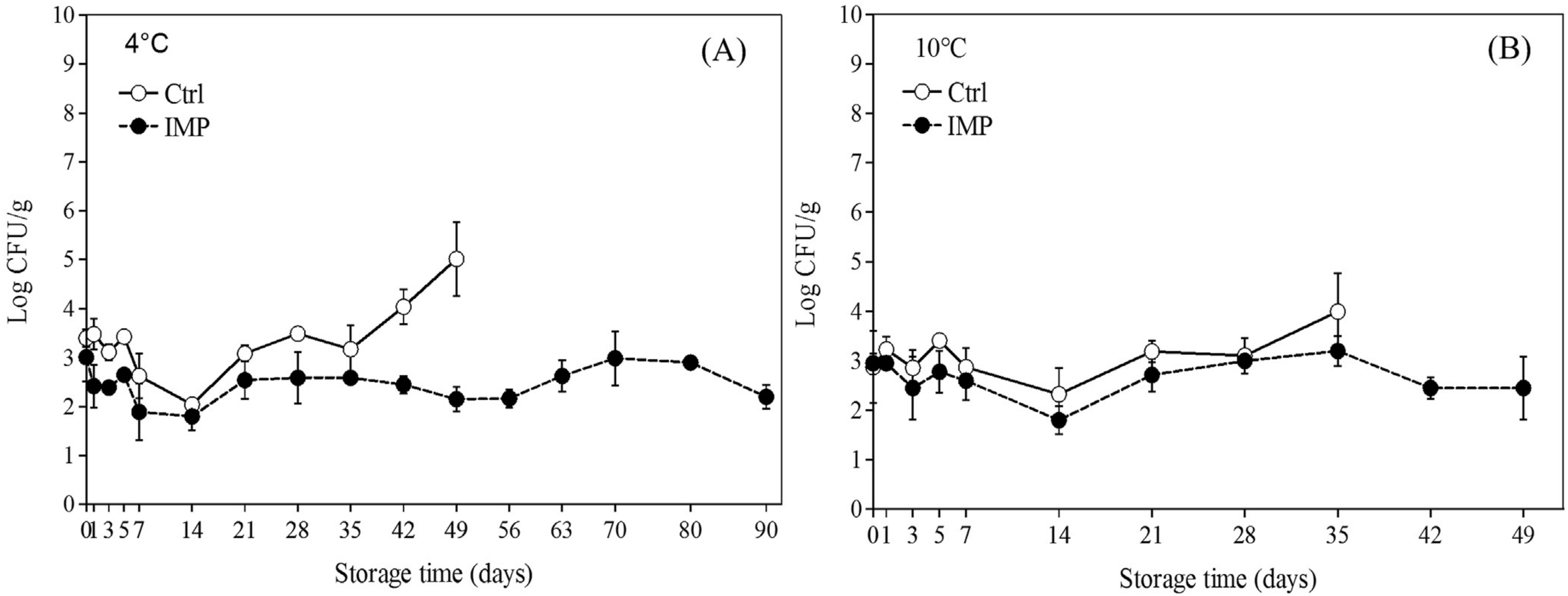

Fig. 4. Number of *L. monocytogenes* Scott A of sausage storaged at 4 and 10 °C for 90 days.

<u>MAIN RESULTS:</u>

- ON SAUSAGE: the antimicrobial casing extended shelf-life of the sausage at least 90 days at 4 °C and 49 days at 10 °C respectively; it reduced artificially inoculated L. monocytogenes by 1.1 log CFU/g (>90% reduction) during storage at 4 C.
- Physical properties of the antimicrobial casing and CTRL were not significantly different in color, thickness and percent elongation (%E) but the tensile strength of IMP was higher than CTRL.
- **Antimicrobial collagen casing impregnated with nisin has the potential to control foodborne microorganisms in order to enhance the microbial food safety and shelf life of sausage.**

OTHER APPLICATIONS:
Incorporation of antimicrobial bacteriocines and LAB

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Vacuum impregnation as a tool to introduce biopreservatives in gilthead sea bream fillets (*Sparus aurata*)

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⊞ **Show more**

https://doi.org/10.1016/j.lwt.2014.09.063

Highlights

- Vacuum impregnation can be used in food preservation with biopreservants.
- Vacuum impregnation implies a reduction in process time to introduce biopreservants.
- No relevant changes in physico-chemical properties were detected in fish fillets.
- Concentrations of LAB and nisin had a significant effect on fillet shelf life.
- Delay in the growth of microorganisms was achieved.