Investigation of the physicochemical and textural properties of an iron-rich 3D-printed hybrid food

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Session 5 **《S5-O2》**

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Objectives: This study investigates the feasibility to formulate an iron-rich hybrid food product mixing vegetal and animal sources for iron deficient populations and its evolution over time to verify that it provides the necessary quantity and quality of iron. 3D-printing is used here as a tool to personalize nutritional intake, texture and aesthetics of products while valorizing valuable animal by-products. The main objective of this project is to follow during 21 days the physico-chemical and texture kinetics of a product manufactured with such a process. This work is also a first approach to determine the influence of the product shape and the most suitable packaging method by highlighting its impact on the end product in relation to its oxidation state and its texture properties.

Materials and Methods: The animal mixture was obtained by chopping raw pork liver (27.25% w/w) and raw poultry liver (70.25% w/w), then mixing with raspberry vinegar (2% w/w) and salt (0.5% w/w). To improve printability, the animal mixture was pre-cooked at 50°C for 15 min. Concerning the vegetal mixture, coral lentils (approx. 86% w/w) were cooked for 15 min from boiling in unsalted water in the ratio of 1:5 (w/w) before being sieved and mixed with lupine flour (10% w/w), peanut oil (3% w/w), curry powder (0.4% w/w) and salt (0.4% w/w). Food matrices were 3D-printed using the Foodini extrusion-based 3D-printer (Natural Machines, Spain) with a 4 mm nozzle diameter. Two shapes were selected corresponding to various ratios of animal and vegetable mixtures: shape (C), a filled cookie (ϕ 70 × 16 mm; 5 layers; 20:80) with the offal mixture hidden inside and shape (F), a flower (ϕ 86 × 9 mm; 3 layers; 15:85) whose petals were filled with the offal mixture. To reach 72 °C at the core of the product, 3D-printed products were baked at 180 °C with 70% steam, respectively 5 min for 'C' and 2 min 45 for 'F'. 3D-printed products were packaged under two modified atmosphere packaging (MAP): O_2-MAP with 70% oxygen [O_2] + 30% carbon dioxide [CO_2] or N_2-MAP with 70% nitrogen [N_2] + 30% [CO_2], then stored at 4°C during 21 days. Analyses were performed on the raw materials and on the product after printing and during storage on days 0, 7, 14 and 21. Water content, water activity (a_w) and pH were measured. Heme iron (HI) rate was determined by the Hornsey method's (Hornsey, 1956) and non-heme iron (NHI) was estimated according to Ahn *et al.* (1993). Lipid oxidation was measured by dosing thiobarbituric acid reactive substances (TBARs). Texture Profile Analysis (TPA) tests were performed on 'C' at 20°C using a texture analyser (EZ-Test LX, Shimadzu, France) with a cylinder probe diameter 50 mm and a double compression at 50% of the sample height. All analyses were carried out in triplicates. The results were statistically analyzed through Analysis of Variance (ANOVA) followed by Tukey *post-hoc* test.

Results and Discussion: Liver iron content was 20.51 ± 1.05 mg/100g and 22.95 ± 1.41 mg/100g for pork and poultry respectively. After 21 days of storage, the 3D-printed hybrid products displayed iron content of around 13 mg/100g, regardless the product shape ('C' or 'F') and packaging method (O_2-MAP or N_2-MAP). These products can be considered as sources of iron which especially fit with the daily needs for women and can thus reduce the incidence of iron deficiency. Most of the changes occurred from day 7 for products stored under O_2-MAP. Indeed, TPA tests indicated that hardness, gumminess and chewiness were higher with O_2-MAP from ($p < 0.05$) than N_2-MAP. In the same way, lipid oxidation was significantly greater in O_2-MAP conditions from day 7 ($p < 0.05$) while the TBARs values for products 'C' and 'F' remained constant until day 21 with N_2-MAP. HI content (5 to 10% of total iron) dropped more sharply when the products were stored under O_2-MAP with a decrease of 0.65 mg/100g compared to 0.06 mg/100g under N_2-MAP between days 0 and 7. A change in liver color was also visually perceived from day 7. On the contrary, MAP conditions had no impact on the NHI content which decreased over time (average loss of 13.5% for 'C' and 27% for 'F' between D0 and D21). In parallel, pH, a_w, water content, remained globally stable whatever MAP during the 21 days of storage with some variations between the two forms mainly due to their composition and not to their proper form.

Conclusions: These iron-rich 3D-printed hybrid products open up prospects for preventing iron deficiency while valorizing animal by-products. 3D-printing demonstrated its potential for rapid prototyping to design functional foods. N_2-MAP seems to have limited the oxidation of the 3D-printed products and further stabilized their physicochemical characteristics and texture properties.

References: Hornsey (1956). Sci. Food Agric, 7 (8), 534-540. Ahn *et al.* (1993). J. Food Sci, 58 (2), 288-291.

Key words: 3D-food printing, Iron deficiency, By-products, Hybrid food, Personalized nutrition

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Kinetic monitoring of hybrid foods during storage

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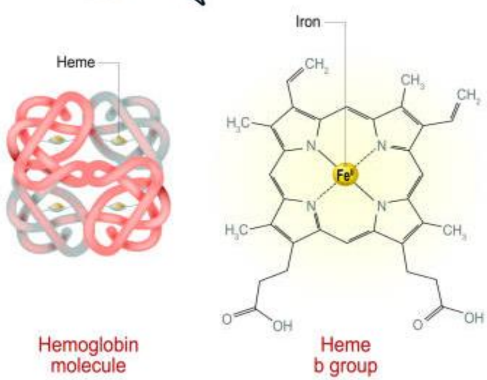
Iron deficiency

1st cause of anemia → 25 % of the world population affected (WHO, 2015)

Dietary iron

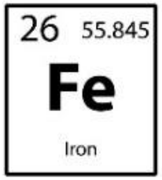
Heme iron (HI)



Non-heme iron (NHI)

Fe^{3+}

Fe^{2+}



How to develop a sustainable and appetizing iron-rich food?

Hybrid food design

Nutritional, functional and
organoleptic complementarity

Animal by-products valorization

High HI content

High protein content

Liver

Animal
+
Vegetal

Lentils

High fiber content

Organoleptic qualities

Functional properties

↓

Design and formulation
(prototypes)

↓

Kinetic monitoring
(physicochemistry, texture, ...)

↓

Ultrastructural and chemical analyses
(Autumn 2022)

Design by 3D-food printing

Foodini 3D-printer

by *Natural Machines*

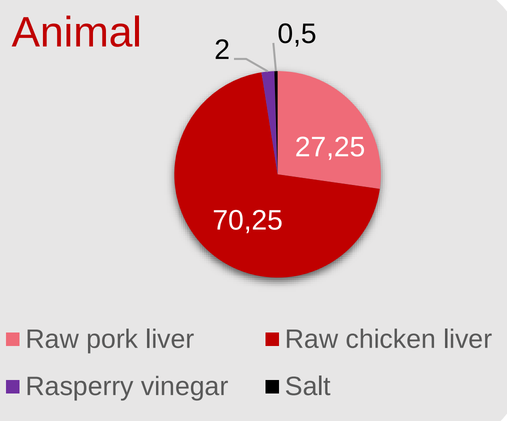
An innovative and suitable tool:

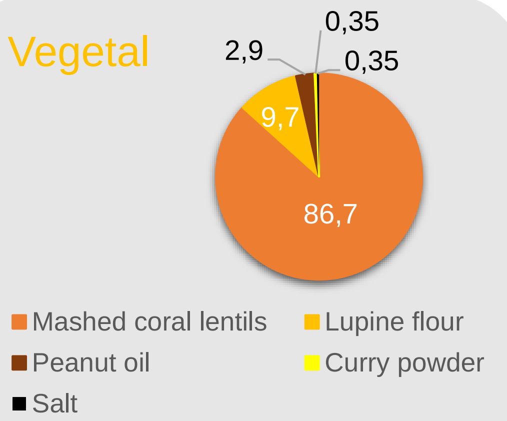
- Rapid prototyping
- Personalized nutrition
- By-products valorization
- Food design and texture
- Increase food acceptability

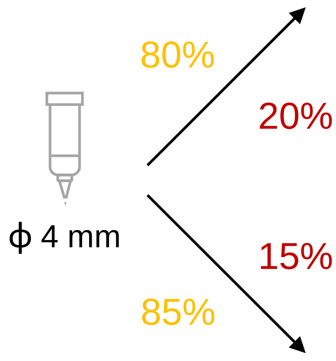
…

Printable recipes and printed shapes

Printable recipes

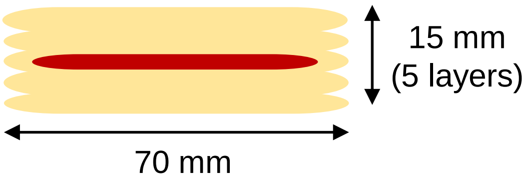




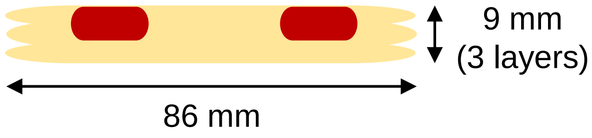


2 shapes selected

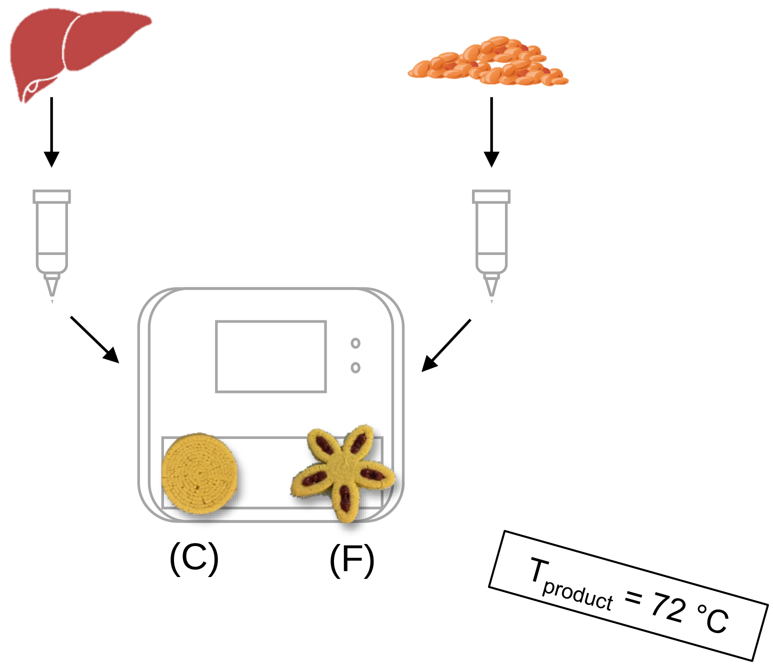
"Cookie" (C)



"Flower" (F)



Experimental approach



Kinetic monitoring of 3D-printed products:

- Physico-chemistry:
  - pH
  - Water activity (a_w)
  - Water content
- Iron content:
  - Heme iron (HI)
  - Non-heme iron (NHI)
- Lipids oxidation:
  - ThioBarbituric Acid Reacting substances (TBARs)
- Texture Profile Analysis (TPA):
  - Hardness
  - Springiness
  - Cohesiveness
  - Gumminess
  - Chewiness

Baking conditions according to shape:
- (C): 180 °C / 5 min; 70% steam
- (F): 180 °C / 2 min 45 s; 70% steam

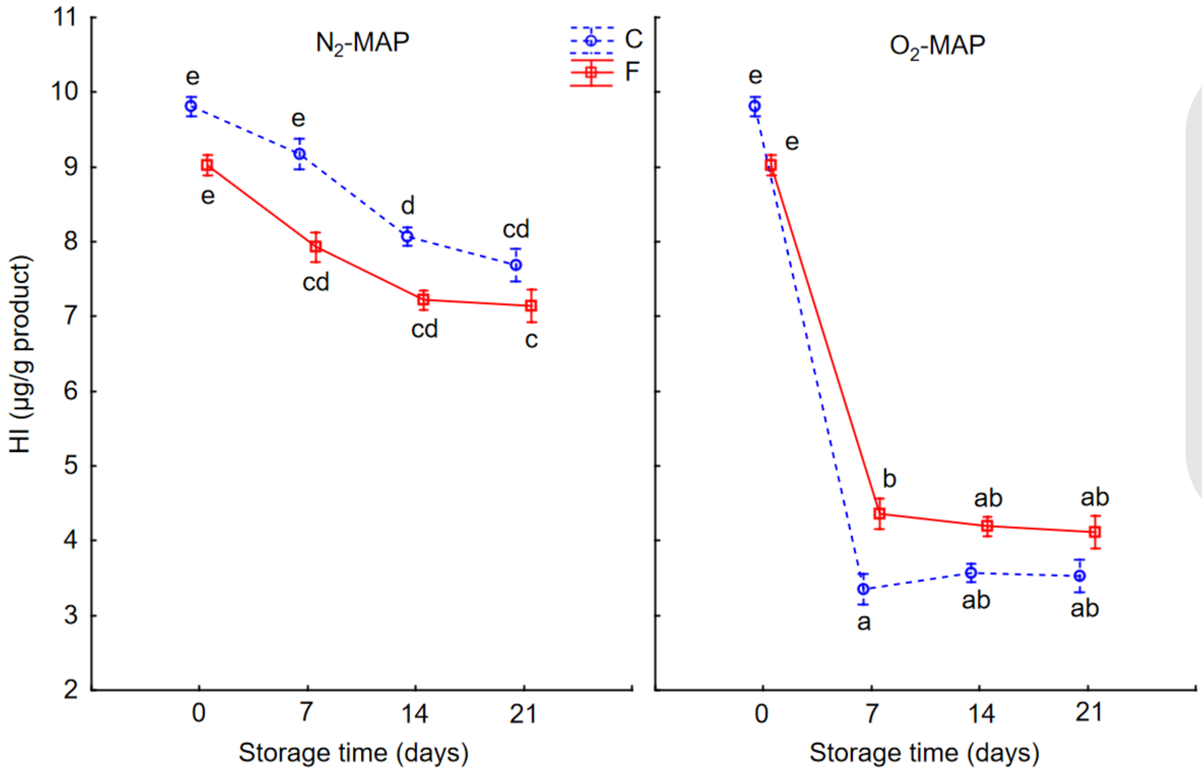
2 modified atmosphere packaging (MAP):
- O_2-MAP: 70% O_2 / 30% CO_2
- N_2-MAP: 70% N_2 / 30% CO_2

→ Analyses on days 0, 7, 14 and 21 of storage at 4 °C

→ Triplicates

Heme iron (HI)

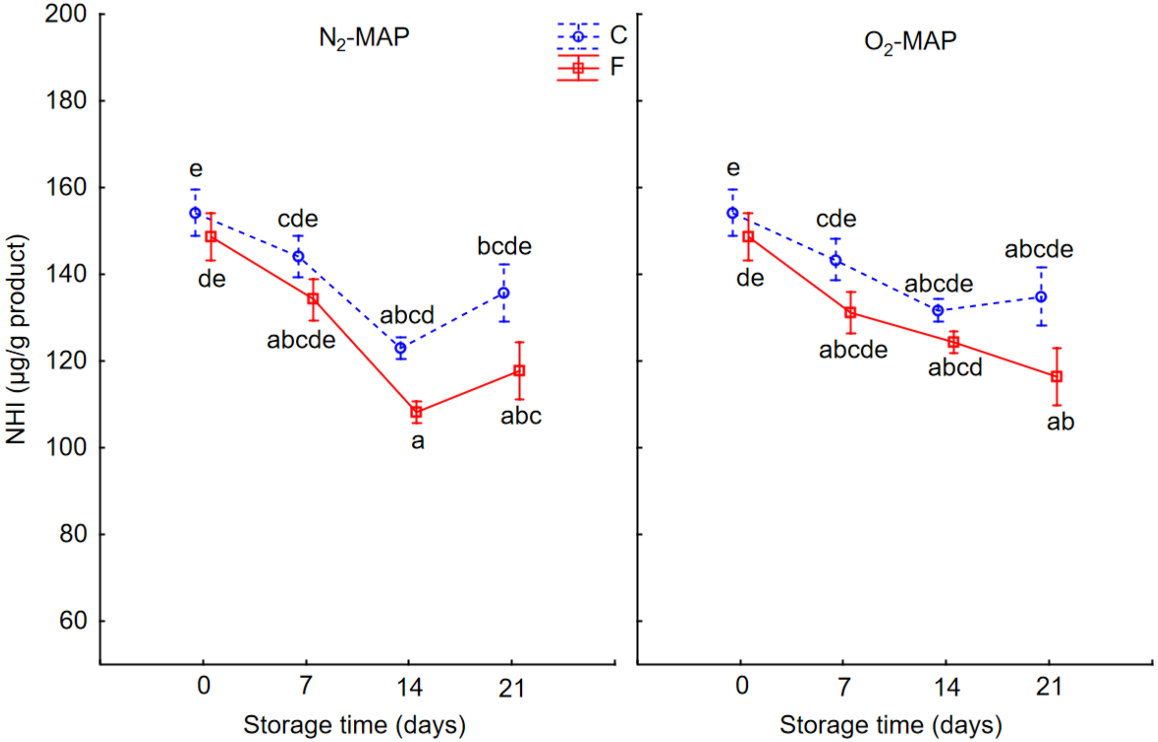
Effect of MAP, storage time and shape on HI content



- Sharp drop in HI from day 7 under O_2-MAP
- Degradation of heme molecule (oxidation)
- No difference between the two shapes

Non-heme iron (NHI)

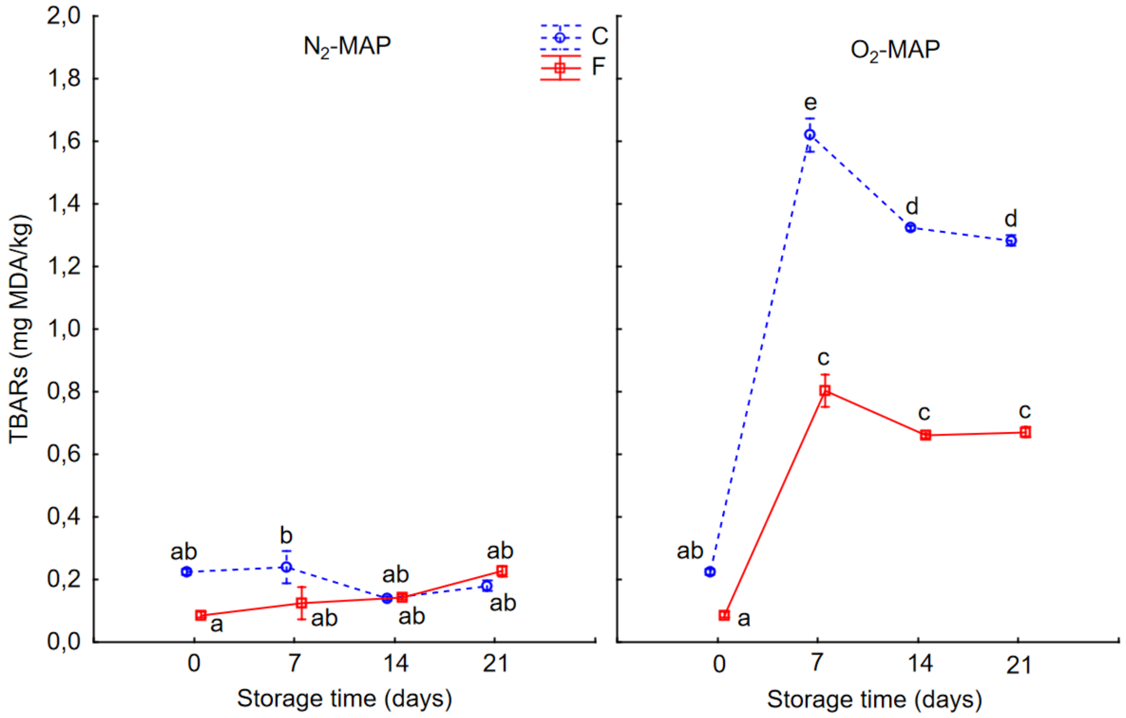
Effect of MAP, storage time and shape on NHI content



- No significant impact of packaging on NHI content
- HI/NHI conversion not seen at this stage
- No difference between the two shapes

Lipid oxidation

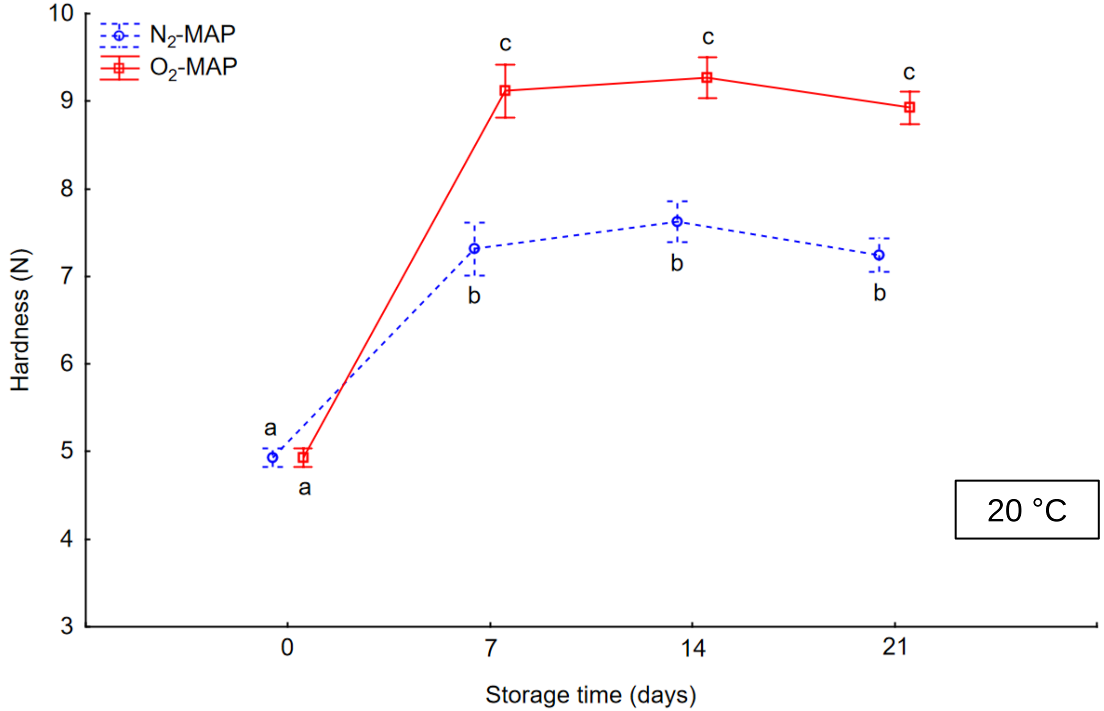
Effect of MAP, storage time and shape on lipid oxidation



- High lipid oxidation from day 7 then plateau
- Difference between the two shapes due to their composition (O_2-MAP)

Texture Profil Analysis (TPA)

Effect of MAP and storage time on texture



Compact

Brittle

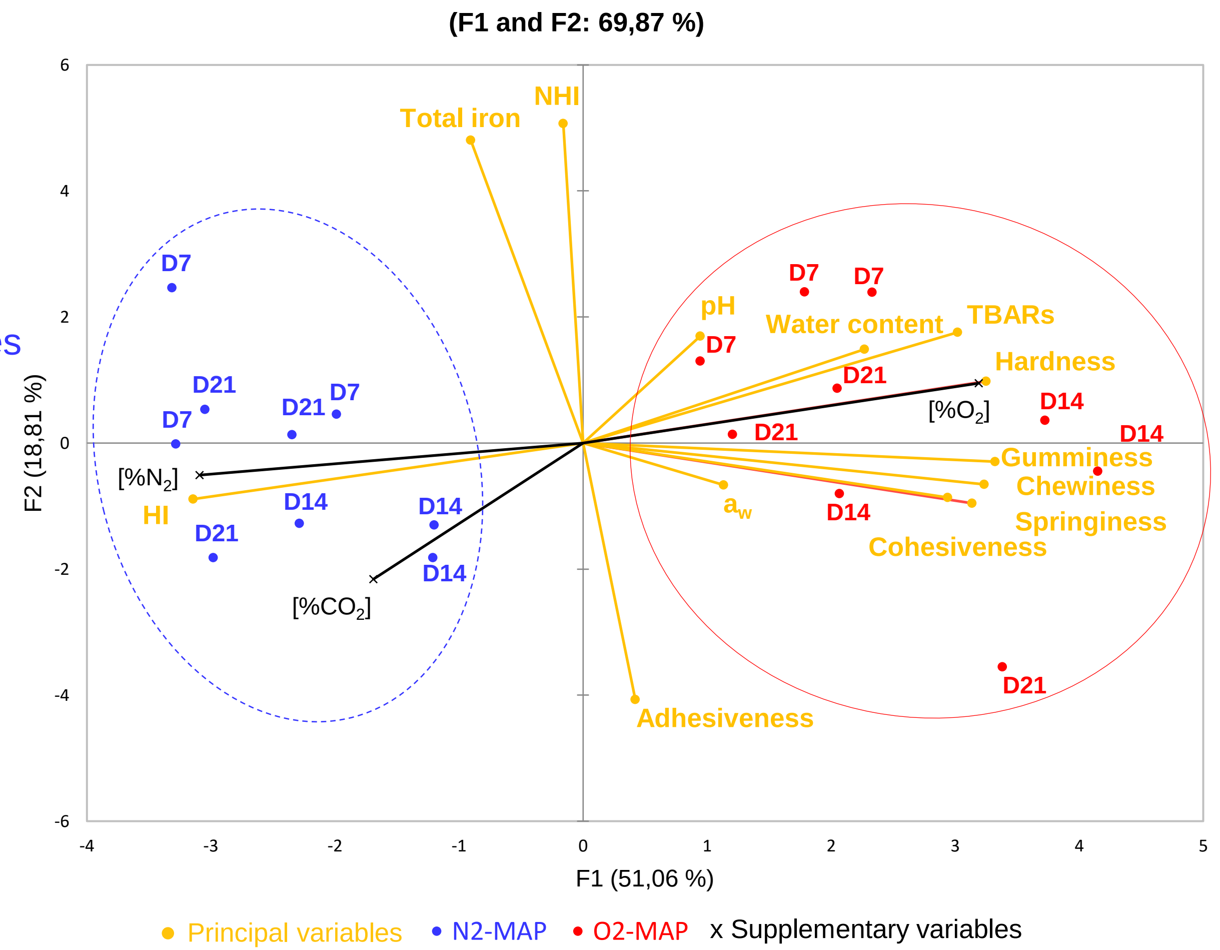
- Change in product structure/texture during storage
- Products significantly harder with O_2-MAP from day 7

Principal Component Analysis (PCA)

High HI content

Low TBARs values

Softer products



Low HI content

High TBARs values

Harder products

Conclusions

- Effect of MAP (O_2/N_2):
  - Storage under O_2-MAP caused oxidation of lipids, a drop in heme iron and product hardening

- Effect of storage time (4 °C):
  - Biochemical changes occurred mainly between D0 and D7 then a plateau seemed to set in

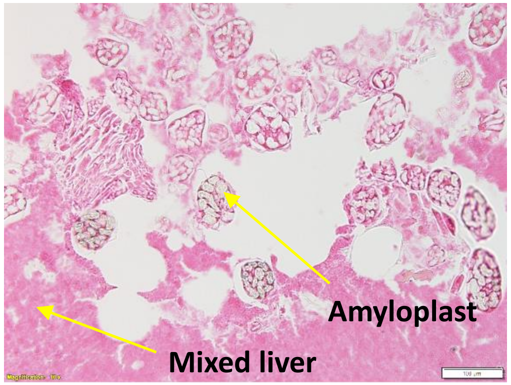
- Effect of product shape:
  - Significant differences mainly due to the product composition and not the shape

Iron investigations at the microscopic scale

Further work at the microscopy scale thanks to the Synchrotron radiation (planned in September):

- Determination of iron distribution and localization (XRF maps)
- Identification of iron oxidation state and its interactions with other elements (XANES and µ-XANES at Fe K-edge)

in 3 areas of interest: animal part, plant part and at the interface.



Interface between the liver (bottom) and the mashed pulses (top). 10 µm cut. Eosin staining to highlight proteins (pink).