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# Semi-industrial scaling up of a membrane process to obtain a nutrient-dense citrus concentrate

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Citrus fruit juices are important sources of bioactive compounds such as provitamin A carotenoids ( $\beta$ -cryptoxanthin,  $\beta$ -carotene), and flavonoids (hesperidin, narirutin) that contribute along with vitamins, minerals and fibers to their beneficial health effects. Crossflow microfiltration is a non-thermal and low energy consuming process increasingly used for insoluble solid concentration in liquid foods. As previously demonstrated at laboratory scale (membrane area  $1.5 \cdot 10^{-2} \text{ cm}^2$ ), an optimized CMF process has allowed to obtain with satisfactory performances nutrient-dense foods, up to 8-10 folds enriched in carotenoids and flavonoids, from citrus juices without increasing sugar content.

The aim of this study was to evaluate and validate at semi-industrial scale an integrated process including enzymatic liquefaction (pectinolytic enzymes), in order to improve the filterability of the product, crossflow microfiltration with continuous retentate extraction (area  $0.2 \text{ m}^2$ ), and a final step of stabilization by pasteurization. The interest of the combination of these 3 successive unit operations was appreciated through the nutritional quality of the final citrus concentrate, focusing on carotenoid and phenolic compound contents.

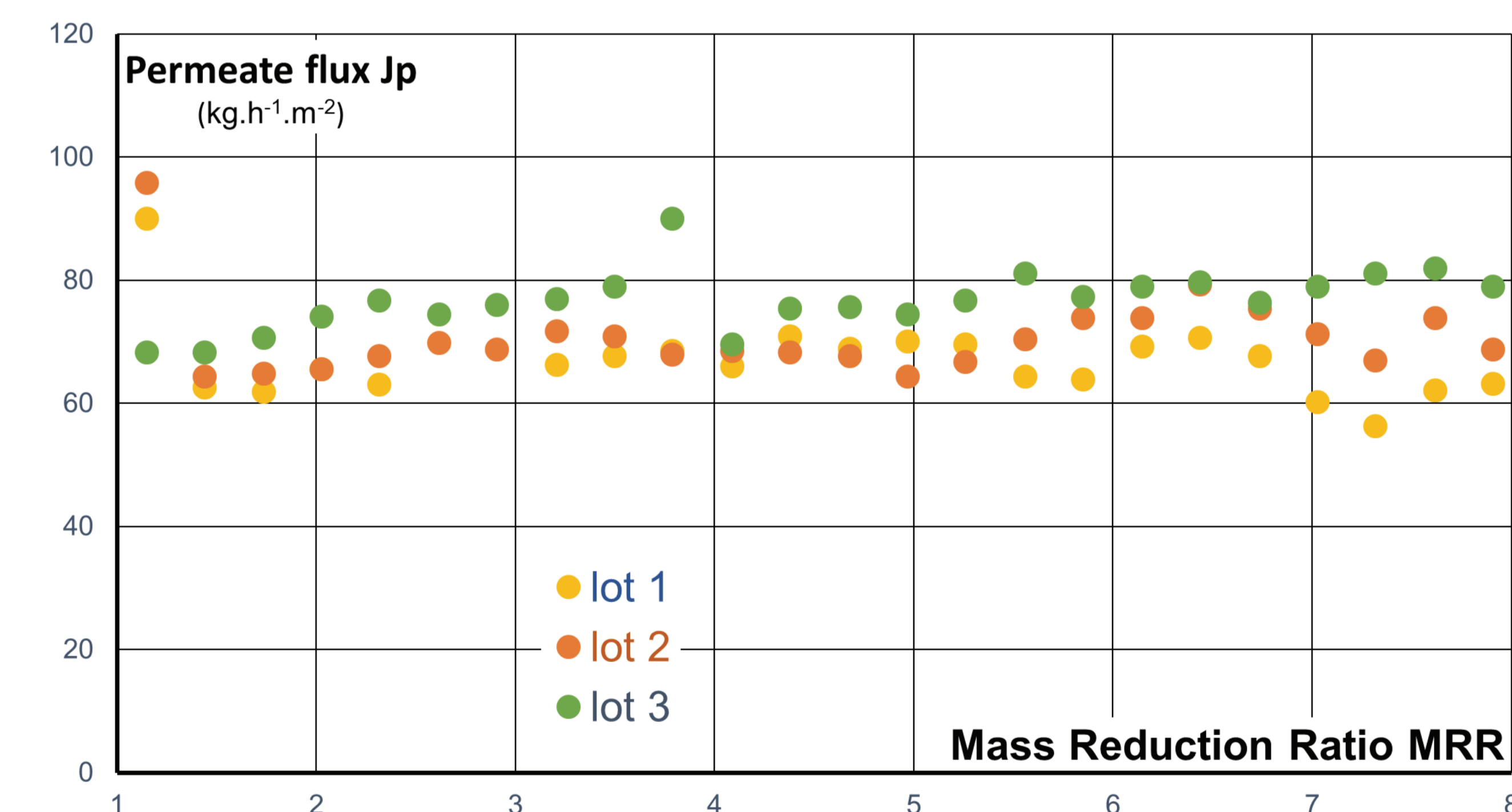
## Raw materials

Three lots of commercial pasteurized citrus juices		Lot 1	Lot 2	Lot 3
pH		3.90 (0.03)		
Titratable acidity TA ( $\text{g} \cdot \text{kg}^{-1}$ )		6.8 (0.2)	7.0 (1.0)	6.8 (0.1)
Total dry matter TDM ( $\text{g} \cdot \text{kg}^{-1}$ )		118 (2)	118 (2)	112 (3)
Total soluble solids TSS ( $\text{g} \cdot \text{kg}^{-1}$ )		117 (4)	117 (4)	116 (1)
Suspended insol. solids SIS ( $\text{g} \cdot \text{kg}^{-1}$ )		4	2	2
Carotenoids ( $\text{mg} \cdot \text{kg}^{-1}$ )	$\beta$ -cryptoxanthin	6.1 (0.1)	6.1 (0.1)	3.6 (0.2)
	$\beta$ -carotene	0.9 (0.1)	1.1 (0.1)	0.5 (1)
Flavonoids ( $\text{mg} \cdot \text{kg}^{-1}$ )	hesperidin	877 (19)	704 (3)	655 (3)
	narirutin	79 (7)	73 (2)	214 (2)

## Analysis methods

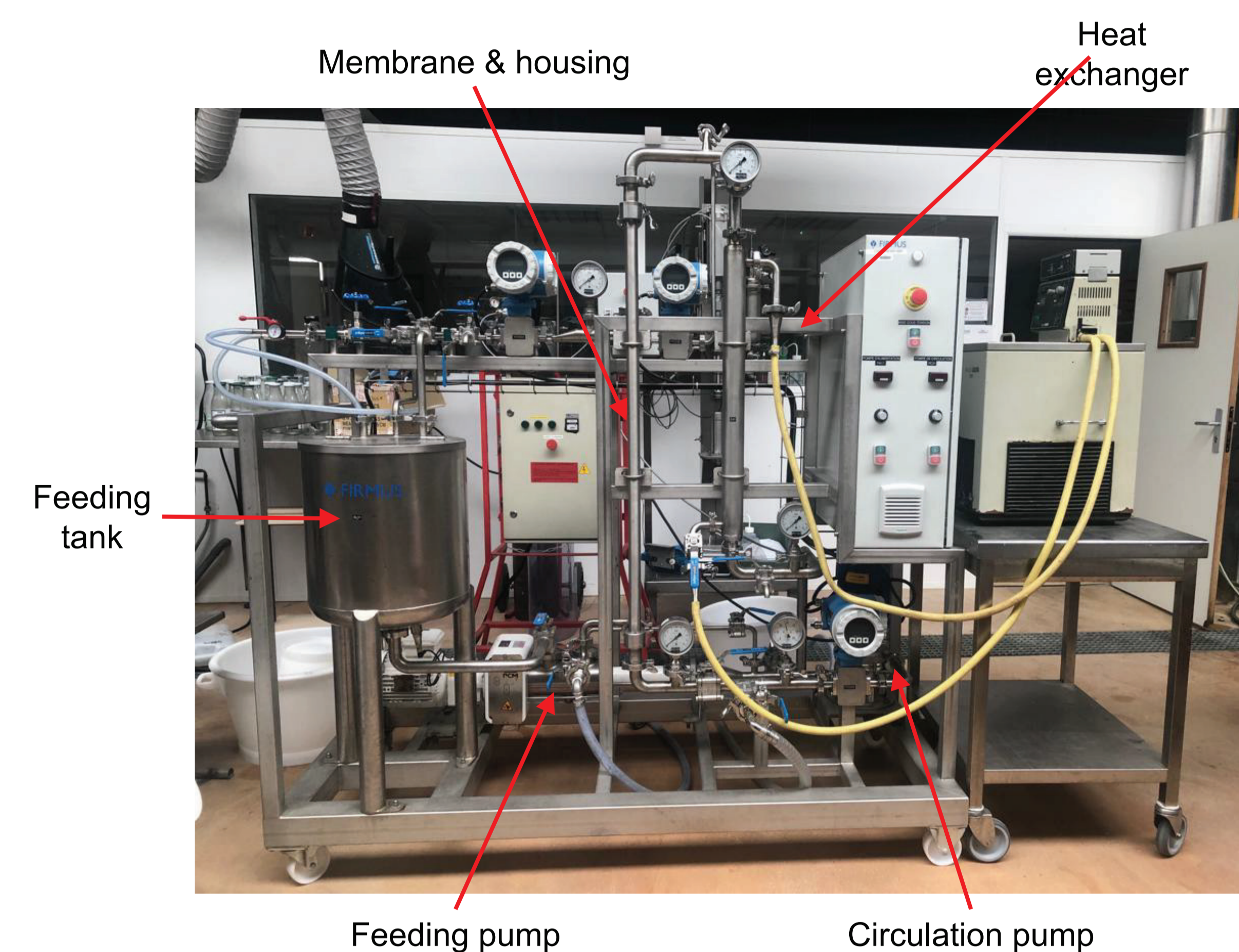
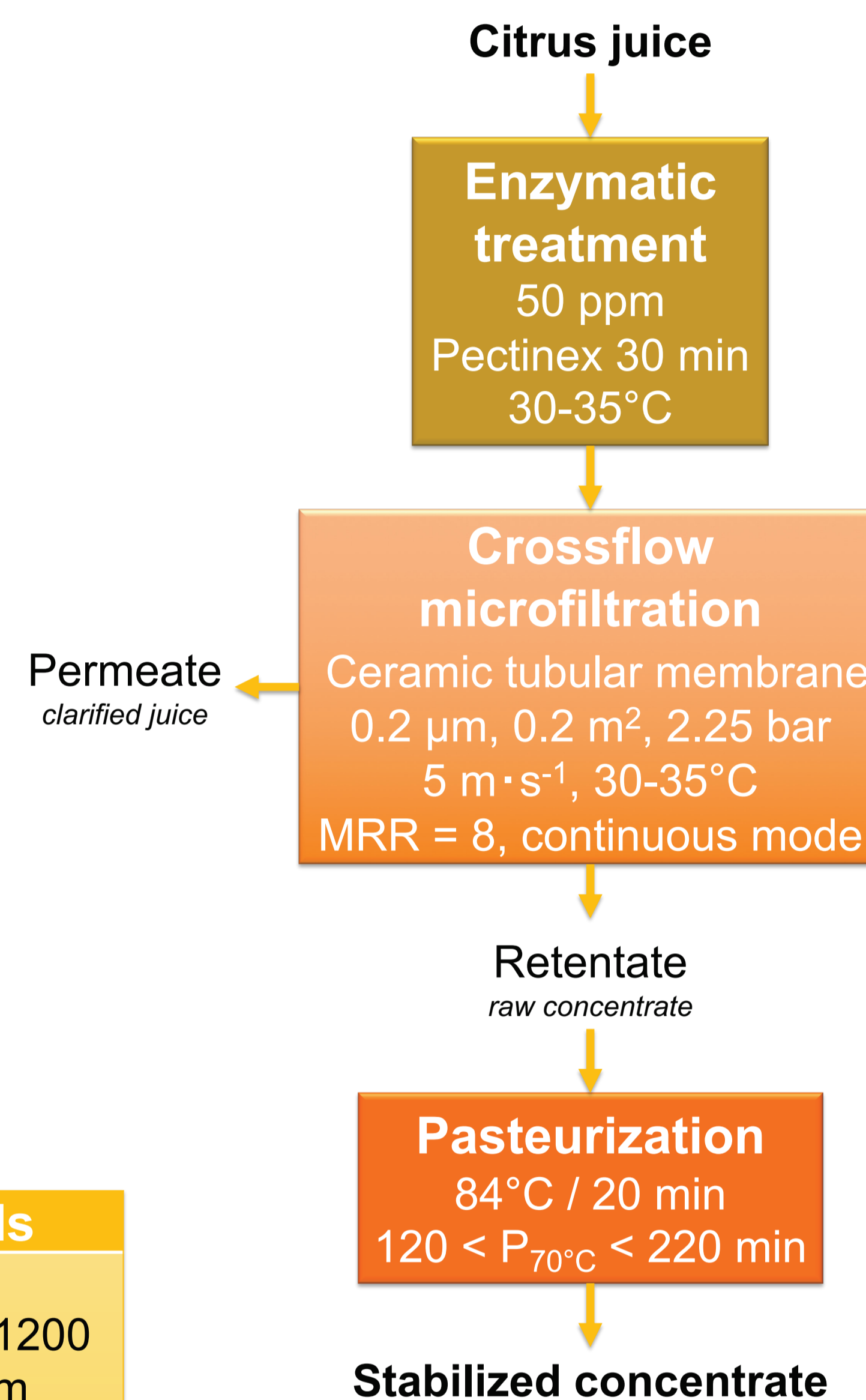
pH, TA	TDM, TSS, SIS	Color	Carotenoids	Flavonoids
pHmeter Tritoline Schott 0.1 N NaOH pH 8.3	TDM: Vacuum drying $70^\circ\text{C}$ TSS: Refractom. Pal3 Pocket Atago SIS: 3000g centrif., vacuum drying $70^\circ\text{C}$	Chrom. CR-400 Konica Minolta CIE Lab	Saponification Hexane extraction HPLC Agilent 1100 DAD 450 nm $\text{C}_{30}$ YMC Gradient water/methanol/MTBE	Extraction HPLC Agilent 1200 DAD 280 nm $\text{C}_{18}$ YMC Isocratic water/ $\text{CH}_3\text{CN}$ /tetrahydrofuran/acetic acid

## Process performance



- ✓ stable and repeatable flux  $\Rightarrow$  process robustness
- ✓  $J_p = 70 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2} \Rightarrow$  compatible with industrial-scale implementation of the process
- ✓ results in accordance with those obtained at laboratory scale

## Process & microfiltration device semi-industrial & continuous



## Concentrate quality

	pH	Titratable acidity ( $\text{g} \cdot \text{kg}^{-1}$ )	Total dry matter ( $\text{g} \cdot \text{kg}^{-1}$ )	Total soluble solids ( $\text{g} \cdot \text{kg}^{-1}$ )	Suspended insol. solids ( $\text{g} \cdot \text{kg}^{-1}$ )
Raw concentrate	3.80 (0.04)	7.6 (0.2)	135 (3)	138 (1)	24.5 (0.2)
Pasteurized concentrate	3.80 (0.02)	7.4 (0.2)	134 (4)	130 (1)	25.5 (0.2)

- ✓ no significant difference between the 3 different lots
- ✓ soluble fraction not modified by the process
- ✓ total dry matter increased, suspended insoluble solids multiplied by 8

Content ( $\text{mg} \cdot \text{kg}^{-1}$ )	$\beta$ -cryptoxanthin	$\beta$ -carotene	Hesperidin	Narirutin
Raw concentrate	39 (4)	6.0 (0.9)	4065 (64)	233 (60)
Pasteurized concentrate	35 (7)	5.2 (1.3)	4145 (134)	241 (69)

- ✓ concentration ratio of 7 for carotenoids and 6 for hesperidin. Consistent but lower than MRR  $\Rightarrow$  losses probably through oxidations
- ✓ narirutin not completely retained by the membrane (retention  $< 50\%$ ) in spite of a very similar molecular structure compared to hesperidin
- ✓ no significant effect of pasteurization on the concentrate composition

## Conclusions

- ✓ The process allowed to obtain a product enriched 8 fold in carotenoids and hesperidin at semi-industrial scale
- ✓ The results are consistent with those obtained at laboratory scale
- ✓ The industrial feasibility of the process is clearly demonstrated
- ✓ Preliminary tests have shown that the product has a shelf-life of at least 11 months at room temperature

## Before considering industrial transfer

- ✓ Improve the process to reduce phytomicronutrient losses
- ✓ Assess production costs

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