



HAL
open science

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

Awa Ba Sow, Adrien Servent, Claudie Dhuique-Mayer, Manuel Dornier

► To cite this version:

Awa Ba Sow, Adrien Servent, Claudie Dhuique-Mayer, Manuel Dornier. Semi-industrial scaling up of a membrane process to obtain a nutrient-dense citrus concentrate. ICEF14: International Congress of Engineering and Food, Jun 2023, Nantes (44000), France. hal-04210219

HAL Id: hal-04210219

<https://institut-agro-montpellier.hal.science/hal-04210219>

Submitted on 18 Sep 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

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

¹ QualiSud, Univ. Montpellier, Cirad, Institut Agro, Univ. Avignon, Univ. La Réunion, Montpellier, France
² CIRAD, UMR QualiSud, F-34398 Montpellier, France

Awa Ba Sow^{1,2}, Adrien Servent^{1,2},
 Claudie Dhuique-Mayer^{1,2}, Manuel Dornier¹
 contact: manuel.dornier@institut-agro.fr

Citrus fruit juices are important sources of bioactive compounds such as provitamin A carotenoids (β -cryptoxanthin, β -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 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.

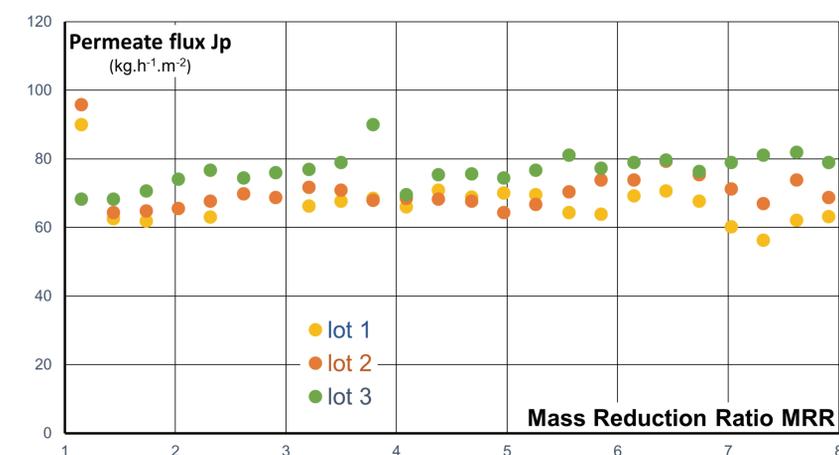
Raw materials

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 ($\text{mg} \cdot \text{kg}^{-1}$)	β -cryptoxanthin	6.1 (0.1)	6.1 (0.1)	3.6 (0.2)
	β -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

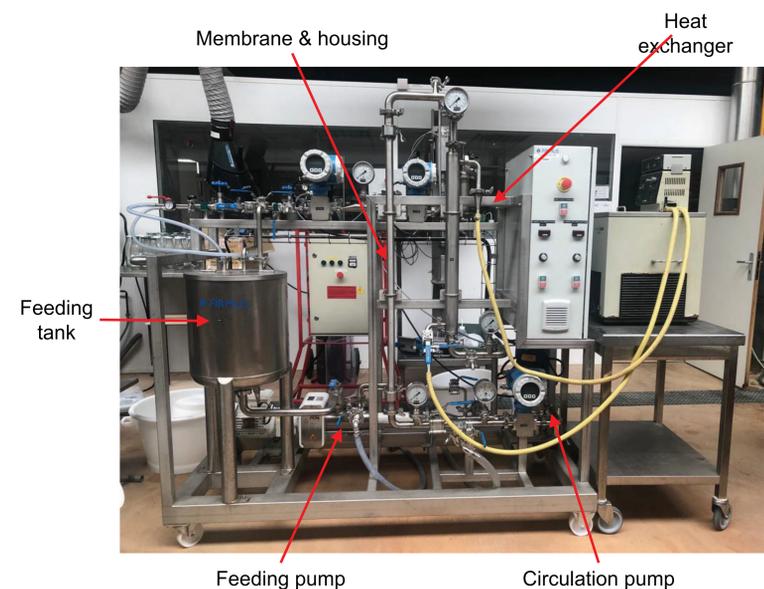
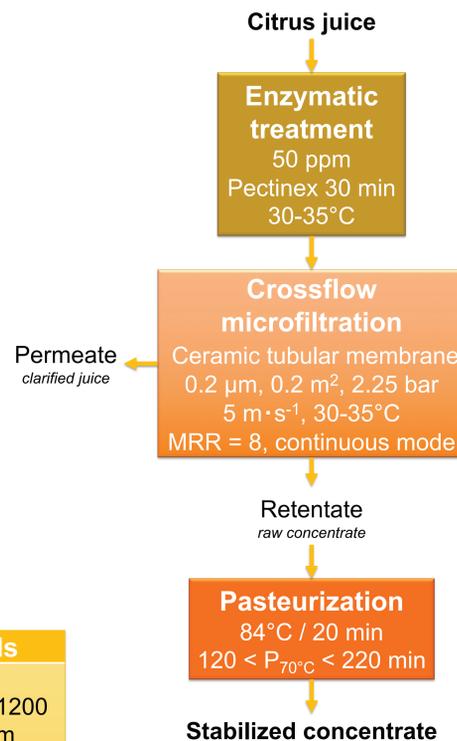
pH, TA	TDM, TSS, SIS	Color	Carotenoids	Flavonoids
pHmeter Tritoline Schott 0.1 N NaOH pH 8.3	TDM: Vacuum drying 70°C TSS: Refractom. Pal3 Pocket Atago SIS: 3000g centrif., vacuum drying 70°C	Chrom. CR-400 Konica Minolta CIE Lab	Saponification Hexane extraction HPLC Agilent 1100 DAD 450 nm C_{30} YMC Gradient water/methanol/MTBE	Extraction HPLC Agilent 1200 DAD 280 nm C_{18} YMC Isocratic water/ CH_3CN /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 ($\text{mg} \cdot \text{kg}^{-1}$)	β -cryptoxanthin	β -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

The authors thank EURABIOTECH (70, rue du Dr Yersin, Parc Eurasanté Ouest, 59120 Loos, France) for financial support of this study