



## Nanofiltration of lactic acid whey prior to spray drying: Scaling up to a semi-industrial scale



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### ABSTRACT

Along with the increase in the fresh cheese production market, there is a concomitant increase in the volume of its by-product, lactic acid whey (LAW). This type of whey is especially rich in organic acids and ash content, making it more difficult to develop applications for human food. In view of its hygroscopicity, this type of whey is in fact clearly the most difficult to dry properly, and its high level of mineralization narrows its potential uses for nutritional reasons. The aim of this study was to evaluate the ability to use nanofiltration (NF) for the production of partially demineralized LAW powder with regard to the dryability of the concentrate and the quality of the powder at a semi-industrial scale. The strong selectivity of this demineralization process results in a 30% reduction in lactic acid content and a reduction of between 46 and 60% in monovalent ions. The dryability of the NF LAW concentrate is improved as well. Moreover, the energy cost of the overall process is reduced by 43%. These elements highlight the benefit of inserting an industrial NF step into the overall processing of LAW and should significantly contribute to the production of partially demineralized LAW powder.

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### 1. Introduction

Acid whey is the by-product of the cheese and caseinate industries. Its high mineralization, ranging from 12 to 20 g/100 g of dry matter (DM) according to Pearce (1992), makes its further processing difficult and limited. For example, lactic acid whey (LAW) decreases the performance of vacuum evaporators due to the increase in mineral fouling and overall heat transfer resistance. On the other hand, the kinetics of lactose crystallization in the LAW concentrates obtained by vacuum evaporation are slower than for sweet whey concentrates, with an increase in the crystal size dispersion and a decrease in the final crystallization rate (Modler & Lefkovich, 1986). These LAW concentrates are therefore more difficult and complex to spray dry than other whey types because of the increased risk of stickiness and caking due to the high

hygroscopicity of the LAW powder (Alais, 1984). Moreover, their use in human nutrition is limited by their nutritional imbalance (Batchelder, 1987). The low market price of lactic acid whey compared to sweet whey has made it necessary to circumvent these limitations by demineralization prior to drying, either by ion exchange resins (Hoppe & Higgins, 1992) or by electrodialysis, although Chen, Eschbach, Weeks, Gras, and Kentish (2016) revealed that the energy cost of a 90% demineralization of acid whey is comparable to a demineralization of a sweet whey. These two techniques are widely used at the industrial scale but generate high investment and cleaning costs due to the high volume of effluent that needs to be treated in a wastewater treatment plant (Pearce & Marshall, 1991). Whey demineralization by nanofiltration (NF) makes it possible to sidestep many of these disadvantages (Eriksson, 1988). The NF process allows concentration (up to 20–22 g/100 g of DM) and demineralization of the whey in one step (between 25 and 60% or 90% with diafiltration) (Gregory, 1987; Kelly, Horton, & Burling, 1992; Kelly & Kelly, 1995), with lactose losses ranging between 1 and 6%. This technique could be used on milk protein concentrate in the powder state to improve storage stability

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(Cao et al., 2016). The benefits of an NF processing step in the production of spray-dried demineralized acid casein whey powder have already been reported by Jeantet, Schuck, Famelart, and Maubois (1996). Moreover, the removal of lactic acid in whey promotes lactose crystallization by increasing the glass transition temperature (Safari & Langrish, 2014) and decreasing the risk of thickness of the lactic acid whey concentrate (Mimouni, Bouhallab, Famelart, Naegele, & Schuck, 2007). Consequently, there is a great deal of information in the literature about the benefits of using NF on whey, but a semi-industrial scale-up of all these results has not yet been demonstrated. Some authors have used model solutions, small nanofiltration, evaporators and spray dryer pilot plants on the lab scale alone (low membrane surface, low water evaporation capacity, etc.), but they are not sufficiently representative of the dairy industry scale (Chandrapala, Chen, Kezia, Bowman, Vasiljevic, Kentish 2016, Chandrapala, Duke, Gray, Week, Palmer, Vasiljevic 2016; Safari & Langrish, 2014; ). The aim of this study was therefore to take account of the abundant literature available in order to evaluate the feasibility of a semi-industrial scale-up in view of the ability of NF to improve the dryability of NF concentrate and the quality of the LAW powder. Dryability, composition and the physical properties of the LAW and NF LAW powders were particularly emphasized, as was the energy consumption involved in both processing schemes.

## 2. Materials and methods

### 2.1. Process

#### 2.1.1. Lactic acid whey

Liquid lactic acid whey (LAW) at 5.9 g/100 g DM and pre-concentrated lactic acid whey at 33 g/100 g DM were provided by a French factory specialized in the processing of fresh dairy foods (cheeses and yogurts). The pre-concentrate was prepared in the industrial production plant using a falling-film evaporator (FFE) (Fig. 1). The composition of the liquid LAW is given in Table 1 and was in accordance with classical LAW. The product was stable, stored at 4 °C and no bacterial growth was detected over a week (<400 CFU/mL).

#### 2.1.2. Concentration by nanofiltration

A semi-industrial nanofiltration plant (GEA Process Engineering, St Quentin-en-Yvelines, France) was used to demineralize and concentrate the liquid LAW to a volume reduction factor of 3 (Fig. 1). The plant unit was composed of a 100- $\mu$ m pre-filter, a heat exchanger for temperature control purposes, two pumps (3 kW and 5.5 kW) and two spiral wound membranes (molecular weight cut off at 200 kg mol<sup>-1</sup>) with a total effective area of 2  $\times$  7.2 m<sup>2</sup>, made of polymers (Filmtec NF245 -3840/30FF code 319116, Dow Chemical Company, USA) with an isoelectric point close to 4.0 and a water permeability of approximately 5 L/(h.m<sup>-2</sup>.10<sup>-5</sup> Pa). During the experiment, the transmembrane pressure was between 3.2 and 3.4 MPa. The water permeate flux was checked between each trial

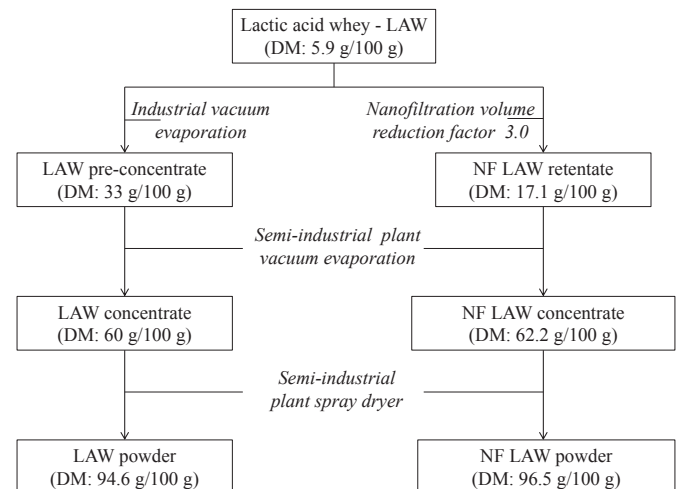


Fig. 1. Technological diagram. Legend: LAW, Lactic acid whey; NF, Nanofiltered; DM, Dry matter.

obtain a nanofiltered (NF) retentate at 17.1 g/100 g DM stored in a tank at 4 °C. The retentate flow was adjusted manually to obtain an average feed flow rate of 225 L/h.

#### 2.1.3. Concentration by vacuum evaporation

The technological diagram is presented in Fig. 1. The LAW pre-concentrate at 33 g/100 g DM and the NF LAW retentate at 17.1 g/100 g DM were heat-treated at 80 °C for 1 min in a tubular exchanger before being concentrated to approximately 60–62 g/100 g DM at Bionov (Rennes, France). A two-stage semi-industrial-scale falling film vacuum evaporator (FFE) (GEA Process Engineering, St Quentin-en-Yvelines, France) with an evaporation capacity close to 300 kg/h was used for this purpose. The temperatures of the first and second stages were 80  $\pm$  2 °C and 60  $\pm$  2 °C, respectively. The concentrates were then cooled to between 30 and 35 °C and stored in a tank to crystallize the lactose, according to the method used by Mimouni et al. (2007), Schuck, 2011, pp. 182–195) (Fig. 1). Lactose crystallization in LAW and NF LAW was followed by refractometry. It has been reported that when crystallization occurs in whey concentrates during batch experiments, DM content decreases with decreasing lactose content in the soluble phase (Mimouni, Schuck, & Bouhallab, 2005). Furthermore, Schuck, Méjean, Dolivet, Beaucher, and Famelart (2005) reported a linear relationship between the DM content of whey concentrates in the soluble phase and refractive index (RI) expressed in °Brix. Therefore, as in the case of the pure lactose solution (Mimouni et al., 2005), lactose crystallization kinetics in LAW concentrate were followed by monitoring the RI as a function of time, using a handheld Atago refractometer (Atago, Tokyo, Japan). The lactose crystallization content (in g/100 g (lactose basis)) was calculated according to the following equation:

$$\text{Lactose crystallization content} = \left( \frac{100(\text{Initial refraction index} - \text{Final refraction index})}{\text{Lactose} \times \left( 100 \times \frac{\text{Final refraction index}}{0.95} \right)} \right) \times 100 \quad (1)$$

to ensure that at least 95% of the flux could be restored. Three thousand liters of liquid LAW were nanofiltered at 11  $\pm$  1 °C to

where the lactose content is expressed in g/100 g (dry basis).

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