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Effect of cryoconcentration, reverse osmosis and vacuum evaporation as concentration step of skim milk prior to drying on the powder properties

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ABSTRACT

Skim milk was concentrated by cryoconcentration (CC), reverse osmosis (RO) and vacuum evaporation (VE), and the concentrated milks were spray-dried. The powder morphology and shape were observed by electron microscopy and the mean size distribution by a sieve jet method. The resulting powders were reconstituted to 25% total dry matter (TDM) to carry out different measurements such as particle size distribution in solution, zeta-potential, heat stability, color, wettability, solubility and dispersibility. The surface of the particles was smooth with small amount of visible substructures on powder surface concentrated by VE and RO. The CC and RO corresponding powders have less particle fragments than VE powder. The mean particle size was larger for CC, followed by RO and VE powders. At the size of 250 nm, the CC-powder occupied three times the volume (%) occupied by the RO-powder and the VE-powder. Analysis of reconstituted powders showed that the mean size distribution of casein micelles was found 190 nm, 164 and 164 nm for CC, RO and VE reconstituted powders, respectively. Moreover, it has been shown that RO and VE powder produced firm gels when heated whereas the CC powder presented flocs and weak gels. As it, all powders exhibited high solubility index (˜95%). However, depending on the size, the lowest solubility in water was observed at 75 nm. Regarding the powder dispersibility, they presented a good dispersibility (<90%). The powders wettability was also size-dependent and was similar for all powders at a given particle size.

1. Introduction

Milk powders are widely commercialized and present a particular interest in the food industry [1]. Because of the functional properties of milk proteins and the overall high value of its constituents, milk powders are considered as valuable food ingredients for different applications in food formulations [2]. Considering the fact that storage and handling of milk present some difficulties due to the huge volumes of processed milk, drying is a common way of increasing storage stability and facilitating the handling of milk [3]. Moreover, exportation of milk is largely facilitated when the product is in a powder from. Concentration of milk is a required operation in the process of powder and condensed milk production [4]. However, with the increasing development of concentration techniques, there is a need for the dairy industry to improve the understanding of the effects these processes exert on the quality of powders, including functional and structural properties [5]. Moreover, the modern dairy industry is making huge efforts to improve the quality of milk powder. Thus, there is great emphasis on adding value to powders by using more appropriate operating conditions, including the concentration step which is essential in the process of obtaining milk powders of high quality [6].

Basically, three methods of milk concentration are developed, including the removal of water as a vapor by applying vacuum evaporation, removal of water as a liquid by means of reverse osmosis and removal of water as ice by cryoconcentration technology [7]. Already, evaporation is considered as the most practical approach, offering the lowest capital cost with a highest concentration level obtainable. Moreover, it is used by the dairy industry as a concentration technique before spray-drying, even if it provides the lowest product quality. Indeed, by applying vacuum evaporation, milk is subjected to heat during relatively long period. Witrowa-Rajchert and Lewicki stated that the effect of vacuum evaporation on the organoleptic and nutritional qualities of the concentrated food products is unfavourable in a context where high quality is required [8]. Reverse osmosis is recognised as suitable for milk concentration because it keeps the integrity of the milk constituents, mainly proteins, and does not affect the volatile compounds. However, membrane processes require replacement of the membranes because of the fouling [9,10]. Moreover, the energy involved in membrane processes such as reverse osmosis is high because of the required pressure. Thus, by considering the aforementioned information, it

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appears necessary to investigate other concentrating approaches which are efficient in terms of providing concentrated milk of high quality without requiring high energetic expenditure. Moreover, the possibility of using passive energy is a key element to be considered in such investigation. In this context, cryoconcentration emerges swiftly thanks to its inherent features, involving low temperature processing and selective nature of water removal step. Because of the low temperatures used in cryoconcentration, this technology is gaining in popularity as an alternative technique to the standard concentration techniques currently used in dairy processing, such as vacuum evaporation and membrane technologies [11,12]. It offers the most enhanced functional and organoleptic qualities of concentrated milk [7]. In fact, it decreases the quality deviation by minimizing the heat abuse on sensitive milk components, such as proteins and flavours. In addition, it allows the recovery of heat sensitive biologically active compounds such as proteins, water-soluble vitamins and aromatics [58–60]. Moreover, from thermodynamic point of view, the used of cryoconcentration is energetically highly interesting because of the low water latent heat of freezing in comparison with the water latent heat of vaporization (80 kcal/kg vs 540 kcal/kg). Furthermore, it is possible to use passive thawing as the recovery step of the concentrated fraction. Thus, by combining the low energy needed to freeze milk and the passive thawing, it will be possible to concentrate milk with high energetic and quality efficiency [13]. Thus, given the relative ease with which milk can be concentrated by reverse osmosis and cryoconcentration as well as the high quality of the concentrated milk obtained, these two methods could be used to concentrate milk before spray drying.

Analysis of scientific literature indicated that there is few information dealing with studies comparing different concentration techniques, namely vacuum evaporation with inverse osmosis and cryoconcentration as pre-treatment step of skim milk prior to spray-drying, particularly on the effects they have on physicochemical and techno-functional properties of milk powders. Indeed, several technological properties of milk powders produced by spray-drying are dependent of the particle-particle interactions (e.g. flowability) and the particle-liquid interactions; where wettability and dispersibility are of key concern for ease of powder reconstitution. These interactions, in turn, are influenced by the particle size, shape, density as well as the chemical composition of the particle surface [14]. The shape depends on the type of raw material, degree of heat treatment, and processing parameters (e.g. the pre-treatment) [15]. In general, factors that increase wettability and dispersibility are hydrophilicity, specific surface area, porosity, larger particle size, and smaller particle/water contact angles [16]. An effect that decreases wettability is the higher fat coverage on the powder surface created during conventional spray drying, making the particle more hydrophobic [16,17]. The presence of fat and sugars on the surface of particles is thought to cause stickiness and reduce powder flowability [15]. Furthermore, one of the most important properties of a powder is its heat stability. For a heat-stable skim milk powder to be produced, a high-heat preheating stage is necessary prior to evaporation. However, it has been shown that heat stability could be improved in low-heat powders by reducing ionic calcium in milk prior to its concentration and drying [18]. In spite of the studies on influence of the type of spray dryers and operating parameters on milk powder properties, particle size, surface composition, and morphology of the powder [19], different food defects, in which milk powder is used as ingredient, are generally attributed to the lack of homogeneity between the powder surface and the core or the particle [20,21]. But as properties of condensed milks prior to spray drying affect to a large extent the functionality of the resulting milk powder [22], it seems necessary to evaluate how this negative impact can be avoided. A choice of appropriate and effective concentration technology seems to be a key in solving this issue. In this context, cryoconcentration has been shown to yield milk concentrates with low lactose and slightly reduced mineral content [23]. Thus, pre-treatment by cryoconcentration may promote flow and heat stability of milk powder.

Thus, the aim of this study is to compare cryoconcentration (CC), reverse osmosis (RO) and vacuum evaporation (VE), as a concentration step prior to spray drying of skim milk, on powder properties.

2. Materials and methods

2.1. Initial skim milk

Pasteurized skim milk was purchased from Natrel (Agropur Cooperative, Quebec, Canada) and was used as the start material. The used skim milk proximate composition was the following: total dry matter 9.24 ± 0.15%, lactose 4.91 ± 0.21%, total protein 3.54 ± 0.17%, and ash content 0.79 ± 0.11%, and residual fat (<0.2%). The milk initial pH value was 6.5 ± 0.15.

2.2. Milk concentration procedure

Skim milk was double concentrated (≈18% total solids) by cryoconcentration (CC), reverse osmosis (RO) and vacuum evaporation (VE).

2.2.1. Cryoconcentration

The cryoconcentration procedure was carried out by applying the cascade principle as reported by [24] and slightly modified by [25]. A volume of 20 L of the initial skim milk was frozen at −20 °C in a stainless jar that was maintained in a freezer under forced air convection. When the whole volume was completely frozen, a thawing procedure was conducted under passive (gravitational) conditions. As a consequence of solute expulsion outside the ice crystal, it was easy to collect a concentrated skim milk fraction. The end of the procedure was set when half of the initial frozen volume of milk was collected. The remained fraction was mainly composed of water (in form of ice) with some entrapped solutes such as lactose and minerals.

2.2.2. Reverse osmosis concentration

Skim milk was preheated to 50 °C and concentrated by reverse osmosis on a GEA Niro Reverse Osmosis System (Hudson, WI, USA). The filtration was carried out on a Desal AG25400T1273 GE at 600 PSI outlet pressure with a total membrane area of 0.32 m^2 and nominal NaCl retention of 99%. The reverse osmosis treatment was terminated when half the initial volume of skim milk was removed as water.

2.2.3. Vacuum evaporation concentration

Skim milk was approximately double-concentrated on a Mojonnier low temperature vacuum evaporator (Mojonnier Bros. Co. Chicago, IL, USA). The evaporation temperature was set at 60 °C. The evaporation procedure was stopped when half of the initial volume of skim milk was evaporated.

2.3. Spray drying of concentrated skim milk

Concentrated skim milk by each concentration technique was spray dried in GEA Niro spray dryer (GEA Process Engineering, Soeborg, Denmark). Drying of all samples was carried out under the same operating conditions and on the same day. The spray drier was equipped with a peristaltic pump for feed fine control and cyclone deflector of powder. Drying was carried out according to the method reported by [26] and modified as follows: During the experiments, the milk was feed to the nozzle at a rate of 32 ml/min. The inlet and outlet temperatures were 190 and 95 °C, respectively. After drying, the powder samples were transferred in sealed bags and stored at 4 °C until use for further analyses.