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# Recovery of caprine whey protein and its application in a food protein formulation

Diana R. Palatnik [a], María Victoria Ostermann Porcel [a], Ulises González [a], Noemi Zaritzky [b], Mercedes E. Campderrós [a, *]

[a] Instituto de Investigaciones en Tecnología Química (INTEQUI)-CCT-CONICET, Facultad de Química, Bioquímica y Farmacia, Universidad Nacional de San Luis, Ejército de los Andes 950, 5700 San Luis, Argentina
[b] Centro de Investigación y Desarrollo en Criotecnología de Alimentos CIDCA (UNLP-CONICET La Plata), Facultad de Ingeniería, UNLP, La Plata, Bs As, Argentina

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ABSTRACT

This study investigated the recovery of proteins from whey caprine cheese which is mostly discarded as waste. A membrane process including microfiltration and tangential ultrafiltration was used to purify and concentrate the protein solution, reaching a 91.4 g/100 g of protein concentration. The concentrate was then freeze-dried and characterized. The good emulsifying properties, high water and oil holding capacity and the rheological behavior suggested the application of the whey protein concentrate in the formulation of a dressing. Physico-chemical characterization indicated that the samples were similar to a commercial dressing in viscosity, texture, moisture and ash contain. Also the sensory analysis demonstrated a good acceptance mainly in color and flavor of the samples. However, the protein content of the product: 0.97 ± 0.12 g/100 g, duplicated the value of the commercial sample incorporating higher added-value to a product with high consumption.



## 1. Introduction

The dairy industry is one of the most important sectors of the economy of industrialized and developing countries. Indeed, this industry generates basic human food, such as milk and a variety of derived foods. In general, cheeses are, in terms of volume, the main destination of the processed milk with nearly 45% of total milk production. Thus, approximately 90% of the milk used in the cheese manufacture is eliminated as whey being the most important by-product. However, whey contains about 55 g/100 g of the total ingredients of milk as lactose, soluble proteins, lipids and mineral salts, so it is now considered a valuable product rather than a waste product (Ha & Zemel, 2003; Jelen, 2003; Sanmartín, Díaz, Rodríguez-Turienzo, & Cobos, 2012). Nevertheless, statistics indicate that a significant portion of this by-product is discarded as effluent which creates a serious environmental problem (Aider, Halleux, & Melnikova, 2009). This observation is more noticeable in the case of cheese manufacture from caprine milk, since producing plants are smaller and craft.

In recent years, there has been a renewed interest in caprines dairy products since caprine milk has been recommended as a good substitute for cow's milk, and has become an alternative food due to their nutraceutical and hypoallergenic properties for babies who cannot be breastfed and for children with cow milk allergy (Maduko & Park, 2011). Caprine milk proteins are less allergenic and the fat is more digestible since, fat globules are smaller than that of cow's milk. Another problem associated with the consumption of cow's milk is lactose intolerance, whereas with caprine milk, the increased rate of gastric passage would be one reason why lactose caprine milk intolerance causes fewer problems being insufficient for a manifest colonic fermentation time (Boyazoglu & Morand-Fehr, 2001).

Whey components have different sizes and are forming a homogenous solution in water, as the major component. Thus, the separation and concentration of whey component by means of membrane technology has been previously outlined (Brans, Schroen, Van der Sman, & Boom, 2004; Rinaldoni, Campderrós, Menéndez, & Pérez Padilla, 2009; Sanmartín et al., 2012). The use of microfiltration (MF) and ultrafiltration (UF) is advantageous over

* Corresponding author. Ejército de los Andes 950. Bloque III Planta Baja, Argentina.
*E-mail addresses:* mcamp@unsl.edu.ar, mcampd@gmail.com (M.E. Campderrós).

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conventional methods, because it can be performed in one step, with low energy consumption, at low temperatures, without the production of contaminating by-products.

Based on the foregoing, in this paper the recuperation of whey proteins from caprine cheese using membrane technology, and its application in the food formulation was assessed. The composition and functional characteristics were investigated as an essential stage for a successful development of a dressing enriched with caprine whey protein.

## 2. Material and methods

### 2.1. Raw material

Caprine whey was obtained from a local cheese-making farm. Caprine cheese whey was obtained from a rennet coagulated cheese that had been produced from pasteurized whole milk (65 °C-15 min). The pasteurized milk was gradually cooled while calcium chloride, thermophile and mesophile yeasts were added. Upon reaching 36 °C, rennet was incorporated, finally sodium chloride was added and the curd was molded and pressed. After 24 h, the mold was removed and the cheeses were salted by immersion. The maturation process started at 8 °C.

After the collection, the whey was passed through a cheese cloth to remove macroscopic impurities and was pasteurized at 63 °C for 30 min. Twenty two liters of whey were processed through UF, in each experience, and the concentrate stream was freeze − dried to obtain the powdered protein concentrate.

### 2.2. Protein concentrate: microfiltration-ultrafiltration and freeze drying stages

The feed (22 L of caprine whey in each experience) was impelled with a centrifugal pump, first through a frontal flow stainless steel filter, with a pore size of 80 μm (Gora, Argentine). The microfiltration reduces the amount of bacteria and spores acting as a cold pasteurization. Moreover this stage protects the ultrafiltration (UF) membrane from fouling. The UF was performed using Pellicon module (Millipore, Bedford, MA, USA), containing two cassettes of modified polyethersulfone membranes with a molecular weight cut-off (MWCO) of 10-kDa, with a membrane area of 1 $m^2$. The concentration of proteins by UF was carried out by continuously removing the permeate stream until the desired concentration was achieved. The operating conditions were the following: transmembrane pressure (ΔP) of 420 kPa and a temperature of 11 ± 0.5 °C. The cleaning of the fouled membrane was performed by applying a "Cleaning in Place" (CIP) procedure according to the manufacturer's instructions. At the end of each run, a cycle of water/alkali (0.2 mol equi/L NaOH, pH 13 ± 0.5)/water wash was applied to the membrane at 24 ± 2 °C and at a transmembrane pressure of 100 kPa. Furthermore, a cleaning step using 0.00403 mol/L NaClO solution (commercial grade) was carried out at the same temperature and pressure to ensure sanitation and cleaning. Measurements of normalized water permeability were performed in order to verify recovery of flow through the membrane and the optimal performance during the separation process.

The caprine whey concentrate, obtained by UF was frozen at −40°C and freeze-dried using a lyophilizer (Rificor S.A., Argentina) at a pressure of 100 kPa for 48 h. The temperature of the samples was measured by a temperature sensor.

### 2.3. Chemical composition

Physicochemical analyses of the feed, permeate and concentrate streams from UF process were determined as follows: pH was measured using a digital pH-meter, (OAKTON Instruments, USA), moisture content by gravimetric method, dry matter by weight difference (AOAC 925.23), ash by incineration (AOAC 945.46); acidity by the official method (AOAC 947.05, 1995); protein content by determination of total nitrogen by the Kjeldahl method using a Digestion Blocks and a semiautomatic Distiller (Selecta, Spain) with a conversion factor of 6.38 (AOAC 991.22); fat content by the Rosse-Gottlieb method (AOAC 933.05); lactose was determined by difference.

All determinations were performed in duplicate.

### 2.4. Functional characterization of the protein concentrates

Functional properties of food proteins are important in food processing and for food product formulation.

The solubility of protein was determined using the method of Morr et al. (1985). The pH was adjusted between 2 and 8 with either 1 mol equi/L HCl or 1 mol equi/L NaOH, respectively. The solubility was calculated as:

$$S = \frac{(W_{C+R} - W_C)}{(W_{C+S} - W_{C+R})} \times 100 = \frac{W_R}{W_W} \times 100 \quad (1)$$

where $W_{C+R}$ is the weight of the dry residue and crystallizer; $W_C$ is the weight of the crystallizer; $W_{C+S}$ is the weight of the saturated solution (prepared as the Morr method requires) and crystallizer; $W_R$ is the weight of the dry residue (g protein); $W_W$ is the weight of the solvent (g water).

Water holding capacity (WHC) was measured as described by Yu, Ahmedna, and Goektepe (2007). *WHC* (grams of water per gram of protein) was calculated as follows:

$$WHC = \frac{(w_2 - w_1)}{w_0} \quad (2)$$

where $w_0$ is the weight of the dry sample (g), $w_1$ is the weight of the tub plus the dry sample (g) and $w_2$ is the weight of the tub plus the sediment (g).

Oil binding capacity (*OBC*) was determined using the method of Chakraborty (1986). The *OBC* (milliliters of oil per gram of product) was calculated as:

$$OBC = \frac{(V_1 - V_2)}{w_0} \quad (3)$$

where $w_o$ is the weight of the protein concentrate (g), $V_1$ is the initial volume of vegetable oil (ml) and $V_2$ is the volume of the supernatant (ml).

Gel strength was determined according to the method described by Chakraborty (1986) and Yu et al. (2007). Protein suspensions containing 2.5 g/100 ml, 5 g/100 ml of protein concentrates were prepared. The pH was adjusted to 3.5; 4.7; 6.1 with 1 mol equi/L NaOH or 1 mol equi/L HCl. Viscosities of these protein suspensions were measured by a Programmable Viscometer (Brookfield, USA). Gel viscosity was determined using a viscometer at different shear rates (10 $s^{-1}$ to 70 $s^{-1}$) at room temperature (21 ± 0.4 °C). Samples were heated at 90 °C in a shaking water bath for 30 min, and then cooled to room temperature without stirring.

The emulsifying capacity (EC) was determined from the technique described by Yu et al. (2007). When a clear emulsion breakage was observed, the total volume of added oil was recorded and used to calculate the EC as the volume (ml) of oil emulsified per gram of protein sample.

For emulsifying stability determination, emulsions of protein were prepared mixing 0.5 g with 100 ml of distilled water under