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Influence of storage conditions on the functional properties of micellar casein powder

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ABSTRACT

Controlled ageing conditions have been applied to two micellar casein (MC) powders and the consequent impact on their rehydration capacity and colorimetric evolution has been reported. Two characteristic times (fragmentation and total rehydration time) and solubility have been determinated to evaluate the evolution of rehydration capacity with controlled ageing conditions.

For the two MC powders tested, it was shown that the two characteristic times and the browning index increased with storage duration and temperature applied during ageing, whereas solubility decreased. For each MC powder studied, it was shown that (i) there is a correlation (ageing curves) between the rehydration time (the target variable) and the indicator parameters (fragmentation time, browning index and solubility) and (ii) the shape of each ageing curve is independent of the ageing conditions but dependent on the MC powder studied.

These results clearly suggest the (i) possibility to obtain reference ageing curves for each indicator, linking total rehydration time and the following indicators: fragmentation time, browning index and solubility (ii) possibility to identify several ageing similarities between severe and moderate storage conditions (iii) feasibility of applying accelerated ageing conditions to rapidly establish the shape of the reference ageing curves for a given MC powder, and (iv) possibility of predicting the rehydration time of the MC powder studied using reference ageing curves through the measurement of one indicator (fragmentation time, browning index and solubility). This predicting ability of the proposed approach has been ascertained by comparing experimental and predicted values of rehydration time for aged samples having undergone storage accidents.

1. Introduction

Milk protein concentrate (MPC) powders are produced from skimmed milk by spray drying after ultrafiltration (Haque et al., 2010). These dairy-derived powders have a high protein content, with the dry matter content ranging from 40 to 90% (De Castro-Morel & Harper, 2002). The casein to whey protein ratio of MPC is identical to that of skimmed milk.

Abbreviations: MPC, milk protein concentrate; MC, micellar casein; P1, powder 1; P2, powder 2.

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Due to their high nutritional value and favourable functional properties (e.g. foaming, emulsifying, gelling, etc.), MPC powders are incorporated as an ingredient into a wide range of applications in downstream food industries (Selomulya & Fang, 2013). For example, they can be used to standardise the protein content of milk and are used as ingredients in many food applications including raising the protein content of cheese and yoghurt.

For most applications, a prior dissolution of MPC powder in water is mandatory in order to allow the powder to fully express its functional properties (Gaiani et al., 2007a) Consequently, rehydration and solubility are important end-use properties for MPC powders and require accurate assessment.

Unfortunately, several studies have reported that MPC powders are characterised by poor rehydration properties (Gaiani et al., 2007b; Richard et al., 2013; Richard et al., 2012; Schuck et al., 2007). This is even truer for micellar casein (MC) powder, whether freshly prepared or stored. MC powder is a high-protein dairy powder obtained from milk microfiltration retentate. The objective is to eliminate almost all of the whey proteins, lactose and minerals (Schuck et al., 1994) and to concentrate the casein micelles while preserving their native structure. Due to the enriched micellar casein content, MC powder is an attractive material for the food industry, as it can enhance the structure, texture and consistency of various foodstuffs (Paracha, 2011; Singh, 2002). For powder enriched in micellar casein, it was (i) shown that the total rehydration process required more than 3 h at 25 °C under stirring at 900 rpm, and (ii) observed that despite the aforementioned treatment, some undissolved material remained, which is generally referred to as the 'insoluble fraction' (Richard et al., 2013).

The impact of storage on the properties of MPC has been extensively studied (Anema et al., 2006; Fang et al., 2011; Gazi and Huppertz, 2015; Haque et al., 2015, 2012; Havea, 2006; Hunter et al., 2011; Jimenezflores and Kosikowski, 1986; Le et al., 2011a, 2011b; Marella et al., 2015; Mimouni et al., 2010a, 2010b; Sikand et al., 2016; Udabage et al., 2012), with particular focus on the gradual loss of solubility during storage (Anema et al., 2006; Fyfe et al., 2011; Haque et al., 2012; Mimouni et al., 2010b). These studies have shown that the solubility and rehydration properties of MPC were negatively impacted by severe ageing conditions (temperature, humidity and storage time). Poor reconstitution properties, and hence poor functionality of high-micellar casein powders can prevent them from achieving full market potential (De Castro-Morel and Harper, 2002). Thus, various studies have investigated ways of preventing insolubility of a powder, either by attempting to adjust adequate process parameters during filtration and spray drying operations (namely the drying inlet temperature conditions for example (Schuck et al., 1994)), or by adapting the initial retentate formulation. It was found that adding monovalent salts to the ultrafiltered retentate prior to drying may improve solubility while increasing the calcium/total mineral ratio decreases the solubility of MPC (Carr et al., 2002).

At present, protein destabilisation due to conformational changes (Fyfe et al., 2011; Haque et al., 2010, 2011, 2015, 2012; Kher et al., 2007) and Maillard reactions in the presence of lactose (Haque et al., 2010, 2011, 2015; Le et al., 2011a, 2011b, 2013) are the most frequently cited causes for the loss of rehydration properties at molecular level.

Development of insolubility of the two high-micelle-casein-content powders, MC and MPC, is believed to share similar mechanisms, nevertheless MC has been less studied and the exact relationship between storage and changes in rehydration properties is not yet precisely known.

From analysis of state of the art, it also appears that the colorimetric change in MC powder with ageing (browning index) has been poorly documented.

Further work is thus needed to establish the influence of storage temperature and time on the ageing of MC powders and to illustrate the link between rehydration time and other ageing indicators (solubility, fragmentation time and browning index). Providing experimental data addressing this issue is the main aim of this study rather than to investigate MC at molecular scale or to establish a relationship between structural modifications and changes in rehydration/browning properties. At present, protein instability in its dry form (conformational modifications and water-protein interaction) and Maillard reaction due to the non limiting lactose content are the most frequently cited causes to explain the loss of **MPC** rehydration properties (Haque et al., 2010; Haque et al., 2011, 2015). However and more specifically, (Nasser et al., 2017) recently showed that the lipid migration towards the particle surface and the surface densification of micellar particles were responsible for major changes in MC rehydration dynamics during storage.

In order to provide such temperature-time correspondences upon ageing, MC was stored at temperatures ranging from 4 to 60 °C for periods up to 12 months. The evolution of various indicators (evolution of browning index, change in solubility, fragmentation time and total rehydration time) upon ageing was evaluated. The first part of this article consists in analyzing the indicators' evolution with time and to assess for any MC the temperature–time correspondences upon ageing. The second part of this paper is devoted to ascertaining whether or not relevant master curves exist between these indicators. If established, these curves will help identify indicators which could be used to rapidly quantify and predict MPC powder's loss of rehydration capacity with ageing.

2. Materials and methods

2.1. *Dairy powders manufacture: physicochemical analysis at inlet and outlet of the spray drying tower and process parameters*

MC concentrates were obtained through microfiltration (pore size = 0.1 μm) of skimmed milk from Ingredia (Arras, France).

Powders were obtained by spray drying an MC concentrate in a pilot workshop (GEA, Niro Atomizer, St Quentin en Yvelines, France) at Bionov (Rennes, France). The same operating conditions as described by Pierre et al. (1992) and Schuck et al. (1994) were used for the spray drying. The inlet temperature was 180 °C ± 10 °C and the outlet temperature was 65 ± 5 °C.

Two batches of MC powder were prepared (P1 and P2). Various physico-chemical analyses were performed on the initial microfiltration retentates and the resulting dairy powders. The nitrogen contents (total nitrogen, non-casein nitrogen, non-protein nitrogen) of initial state-fresh powder were determined as described by Schuck et al. (2012). The total nitrogen content, non-casein nitrogen content corresponding to the soluble fraction at pH 4.6, and non-protein nitrogen content corresponding to the insoluble fraction after their precipitation were determined by the Kjeldhal method. Nitrogen contents were converted into protein contents using 6.38 as multiplying factor. These analyses are reported in Table 1.

MC powders were packed into individual 380 g tin cans after manufacture. The powders were stored at controlled temperatures of 4 °C (referred to as "reference powder"), 20 °C, 40 °C and 60 °C for various durations to a maximum of 12 months.

2.2. *Determination of solubility*

To determine solubility, aqueous solutions of 5% (w/w) MC powder were firstly prepared in distilled water at room temperature for 1 h under stirring. Then, 50 ml of the MC solution was transferred into 50 ml centrifugation tubes and centrifuged using a Sigma 6K15 refrigerated centrifuge (Sigma, Labozentrifugen GmbH, Osterode am Harz, Germany) at 700 × *g* at 20 °C for 20 min. The supernatant was placed in a preweighed moisture dish and weighed. After that, the moisture dish (filled with the supernatant) was dried for 24 h in an oven at 105 °C before being cooled down to room temperature in a desiccator containing dry silica gel to avoid condensation and then reweighed. The percentage of soluble material (σ) of