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Use of ultrafiltration to prepare a novel permeate for application in the functionality testing of infant formula ingredients

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ABSTRACT

Ultrafiltration (UF) permeates produced from reconstituted infant milk formula powder (IMF; 1.3%, w/w, protein) and reconstituted skim milk powder (SMP; 3.2% protein) were compared with simulated milk ultrafiltrate (SMUF) in terms of composition, physicochemical properties and impact, as dispersants, on the heat stability of model infant formula systems. Permeates from IMF and SMP were generated at 15 °C using a lab-scale UF unit with a 10 kDa cut-off polyethersulfone membrane. Operation at optimal cross-flow velocity and sub-critical flux allowed 1 L of IMF to be concentrated by a volume concentration factor (VCF) of 3 in 20 ± 2 min, with minimal flux decline and constant trans-membrane pressure (*TMP*); conversely, UF took 33 ± 4 min for SMP, with a decrease in flux and increase in *TMP* over that time. Permeate from IMF (IMF_p) had a markedly different mineral profile to SMP permeate (SMP_p), with the former having considerably lower levels of the major ions (e.g., calcium, phosphorus and sodium). IMF_p, SMP_p, SMUF or deionised water was used to reconstitute milk protein concentrate (MPC)80 and whey protein isolate (WPI) powders in combination to give 5.5% total protein and a 60:40 ratio of whey protein:casein. These model IMFs were assessed for heat stability at pH 6.8 and 140 °C; the type of dispersant used influenced heat stability strongly, with heat stability decreasing in the order water > IMF_p > SMP_p > SMUF. Calcium-ion concentrations of 0.01, 0.71, 1.51 and 1.77 mM L^{-1} were measured for water, IMF_p, SMP_p and SMUF, respectively, indicating that increased heat stability of proteins dispersed in IMF_p, compared to SMP_p or SMUF, may have been due to lower calcium-ion concentration. This study highlights the influence of serum phase composition on the heat-induced destabilisation of infant formula ingredients and outlines a novel approach for the generation of IMF_p, which is of importance in the development of ingredients which remain stable during the processing of IMF products.

1. Introduction

Research into infant formula is undergoing a period of substantial growth, owing to a number of converging factors, including increased understanding of compositional differences between human and bovine milk [1], diversification of infant formula product ranges and types [24], increase in the demand for infant formulae due to economic growth of nations in Asia, the Middle East, North Africa and South America [3], and a limited literature base related to changes in infant formulae during formulation, processing and storage.

Laboratory-based studies on infant formulae typically necessitate the reconstitution of dried ingredients (e.g., casein and whey protein powders, and lactose) before analysis, to create a model infant milk formula [22,5,21]. During this reconstitution step, water or buffer solutions may be used as dispersants for the powders; in addition, water or buffers may be used as diluents for liquid infant milk formulae in the analysis of certain physicochemical properties (e.g., protein particle size measurement by dynamic light-scattering).

Jenness and Koops [19] formulated simulated milk ultrafiltrate (SMUF) based on the composition of the ultrafiltration (UF) permeate of milk, and SMUF is used in a range of applications in dairy science laboratories. Some of these applications include, *inter alia*, dispersion of milk protein powders [5,8], dilution of milk protein solutions for particle size and zeta potential measurement [14], and modelling of ion equilibria in milk [10]. A buffer simulating the aqueous phase of cheese, synthetic cheddar cheese aqueous phase (SCCAP) has also been developed, based on analysis of the liquid expressed when cheese was compressed in a hydraulic press [4]. Both SMUF and SCCAP are prepared using readily available lab-grade minerals, and the underlying "recipe" can be customised by the end-user for specific applications. For example, O'Mahony

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Nomenclature

CP	concentration polarisation
HCT	heat coagulation time
IMF	reconstituted infant milk formula powder
IMF_p	infant milk formula permeate
J	permeate flux
J_{crit}	critical flux
J_{lim}	limiting flux
J_w	water flux
L_p	hydraulic permeability
Q	volumetric feed flow rate
R_m	hydraulic resistance
SMP	reconstituted skim milk powder
SMP_p	skim milk permeate
SMUF	simulated milk ultrafiltrate
TMP	trans-membrane pressure
V	cross-flow velocity
VCF	volume concentration factor

et al. [25] modified SCCAP with varying levels of calcium chloride to facilitate direct measurement of the effect of colloidal calcium phosphate levels associated with the casein network on cheese textural and rheological properties. In many instances, where it is important to replicate the full complexity of the serum phase of a system such as milk (e.g., lactose, non-protein nitrogen, in addition to minerals), centrifugation, microfiltration or ultrafiltration may be performed to obtain a casein micelle-depleted serum phase [11]; however, guidelines for those wishing to generate these materials at lab-scale are limited.

To the authors' knowledge, a method for preparing a permeate by membrane filtration which replicates the serum phase of an infant milk formula has not been described. The development of such a permeate presents an opportunity to make studies on the techno-functional properties of model infant milk formulae more representative of real systems. The present study involved the preparation of UF permeates from reconstituted skim milk and infant formula powders, and comparison of these with SMUF. Optimal process conditions were determined for a rapid and sustainable UF process. The composition and physicochemical properties of the UF permeates and SMUF were measured. These materials, in addition to deionised water, were then used as dispersants for protein powders to elucidate the influence of serum phase composition on the heat stability of a model whey-dominant infant formula protein system.

2. Experimental

2.1. Materials

Low-heat skim milk powder was provided by the Irish Dairy Board (Fermoy, Co. Cork, Ireland). A commercial first-age infant milk formula was purchased from a local supermarket. Reconstituted skim milk powder (SMP) was prepared by slow addition of powder to deionised water under constant magnetic stirring at 50 °C to obtain a 3.21 ± 0.06% (w/w) crude protein suspension with a native pH of 6.7 ± 0.0. Reconstituted infant milk formula (IMF) was prepared under the same conditions to 1.28 ± 0.03% (w/w) crude protein with a native pH of 7.4 ± 0.1. The pH of the IMF was within the range of values (6.3–7.5) measured for non-acidified commercial infant formulae by Chávez-Servín et al. [6]. Once all powder had been added, solutions were left stirring for ~2 h and then stored overnight at 4 °C. SMUF at pH 6.7 ± 0.0 was prepared as described by Jenness and Koops [19], using analytical grade salts (obtained from Sigma–Aldrich, St Louis, Missouri, MO, USA) dissolved in deionised water.

2.2. Ultrafiltration rig

Lab-scale ultrafiltration (UF) experiments were performed using a pressure-driven, cross-flow filtration device, consisting of a membrane cartridge (Millipore Biomax: 10 kDa molecular weight cut-off, 1000 cm^2 total membrane area, V (viscous)-screen) enclosed within a stainless steel membrane holder (Pellicon 2 mini-holder), all supplied by Merck-Millipore (Tullagreen, Carrigtwohill, Co. Cork, Ireland). Additional information on the geometric and hydrodynamic properties of the membrane cartridge is provided in Table 1.

The feed (1 L) was subjected to continuous magnetic stirring in a glass beaker. A Watson-Marlow 520 s variable speed tri-lobed peristaltic pump (Lennox Pump and Process, John F. Kennedy Drive, Naas Road, Co. Dublin, Ireland) delivered the feed into the feed port of the membrane unit; the volumetric feed flow rate (Q) was controlled by adjusting the rpm of the pump, and values of Q were converted to cross-flow velocity (V) according to Table 1. Braided tubing was installed between the pump and the feed port to minimise oscillatory flow fluctuations caused by pulsation. The retentate passed through a plate heat-exchanger, with counter-flow coolant in alternate channels supplied by a water bath set at 15 °C. The retentate was recirculated to the feed vessel throughout processing, thereby maintaining feed temperature at 15 °C. Two pressure gauges were installed close to the feed and retentate ports, with a valve located on the retentate side for control of trans-membrane pressure (TMP). Permeate was collected in a 1 L graduated cylinder during processing, with all feed materials being concentrated by a volume concentration factor (VCF) of 3. This VCF corresponded to final protein in dry-matter values of 59.5% and 16.5% for retentates of SMP and IMF, respectively. Permeate flux (J) was recorded in duplicate during processing by measuring the volume of permeate generated in a graduated cylinder over 30 s.

2.2.1. Determination of operational parameters

Critical (J_{crit}) and limiting (J_{lim}) flux for SMP and IMF were determined in duplicate in one freshly prepared sample of each at different values of V in constant-TMP mode [27,2] at a constant VCF of 1 using the configuration described earlier, with the exception that both permeate and retentate lines were returned to the feed vessel (i.e., full recirculation mode). TMP was increased step-wise in 0.2 bar increments by partially closing the valve on the retentate side. J was measured in duplicate after an equilibration time of 3–4 min at each TMP. Step-wise increases in TMP were continued until the initially linear TMP–J relationship (pressure-dependant region) reached a plateau (pressure-independent region). Values of J_{crit} and J_{lim} were determined as described by Youravong et al. [29], who performed both constant-J and constant-TMP experiments on skim milk and reported that both methods yielded the same results. Starting values of J for permeate generation were selected for operation in the sub-J_{crit} region. The slope (t) of a linear fit of log J_{lim} as a function of log V was used to assess the influence of turbulence on J for SMP and IMF (Table 1). Hysteresis, due to irreversible fouling [2], was assessed by returning TMP to 0.2 bar and repeating the measurement of J. To determine if the feed