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# Rational design of a versatile lab-scale stirred milk gel using a reverse engineering logic based on microstructure and textural properties

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***Abstract***

The textural properties of stirred yogurts (i.e. stirred milk gels) are directly linked to their microstructure. In this study, a logic of reverse engineering was used to design a lab-scale process producing real-like stirred milk gels (1.5 kg batch). A reference of stirred fermented milk gel was produced at pilot scale (100 kg batch). The conditions of lab-scale homogenization (sonication), acidification (glucono-δ-lactone concentration) and stirring (filter, pumping rate, ultra-smoothing) were adjusted to obtain size distributions of fat droplets and of microgels and rheological properties similar to those of the pilot reference. The final lab-scale process was validated by comparing the properties of stirred milk gels produced at lab and pilot scales with two fat contents (6 and 10 wt%). Although the stirred milk gels obtained at the two scales differed slightly in microgel sizes and in rheological properties, they were similarly ranked according to their fat content. *(147 / 150)*

***Keywords***

Formulation, physico-chemical properties, stirred milk gel, rheology, process, fat reduction.

## 1. Introduction

Fermented milks are obtained by using lactic acid bacteria, which leads to a reduction in pH from 6.5 to 4.5. They are usually manufactured mainly from milk ingredients and the bacteria have to be viable, active and abundant in the final product (FAO/WHO, 2011). Several million tons of yoghurts and other fermented milk products are produced and consumed every year and are important from both an economic and nutritional point of view. Stirred milk gels have been widely studied, especially for their sensory quality of creaminess, mouth coating or smoothness (Cayot et al., 2008; Lee and Lucey, 2006). To be processed into a stirred fermented gel, milk undergoes several typical and well documented unit operations with different physico-chemical conditions that considerably modify the microstructure (Lee and Lucey, 2010; Mokoonlall et al., 2016; Sodini et al., 2004) (see details in Fig. 1). At first, raw milk is a complex colloidal system composed of an aqueous phase (lactose, minerals and solubilized whey proteins), suspended casein micelles and fat dispersed as droplets about 5 µm in diameter. It is a liquid oil-in-water emulsion in which the interface between the aqueous phase and the dispersed fat is a native membrane containing proteins, polar lipids, enzymes and other minor components (Lopez et al., 2014). The raw milk is centrifuged to separate it into skim milk (about 3.5 wt% protein) and cream (about 35 wt% fat and 2 wt% protein). The resulting cream and skim milk are then mixed to obtain a mix with a target composition (in terms of fat and protein contents). Skim milk powder (SMP) (about 35 wt% protein) can be used to adjust the protein content. The resulting mix is pasteurized through a thermal treatment about 85 °C for 30 min or 95 °C for 5 min (FAO,



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2013) leading to the destruction of microorganisms, aggregation of whey proteins and creation of whey protein/κ-casein complexes. The mix is then homogenized under dynamic pressure generally between 10 and 20 MPa (Sodini et al., 2004), which reduces the fat droplets to less than 1 µm and modifies the native interface through the adsorption and reorganization of whey proteins and caseins (Loveday et al., 2013). The pasteurized and homogenized mix is inoculated with lactic bacteria to reduce the pH from 6.5 to the isoelectric point of milk proteins, around 4.5 (Lee and Lucey, 2010). During this acidification step, electrostatic repulsions are eliminated and the micellar calcium is solubilized, allowing the proteins to form a three-dimensional network in which fat droplets can interact via their interface. The stirring stage transforms the resulting emulsion-filled gel into a concentrated dispersion of microgels of between 10 and 100 µm in diameter (Lee and Lucey, 2010; Sodini et al., 2004). Finally, the stirred milk gel is cooled, packaged and stored at 4-10 °C.

By varying the formulation, several studies have shown that the textural or sensory (firmness, graininess perception, etc.) properties of stirred milk gels were directly driven by their microstructure (microgel size, fat droplet size, protein network thinness, etc.) (Abhyankar et al., 2014; Cayot et al., 2008; Foucquier et al., 2012; Lee and Lucey, 2010; Mokoonlall et al., 2016). Genovese et al. (2007) broadly reviewed the impact of size, shape and hardness of particles on the instrumental texture of food dispersions. In addition, by combining instrumental measurements and sensory characterization (trained panelists), Janhoj et al. (2009) showed that perceived creaminess was negatively correlated with perceived graininess and hence with the measured size of the microgels dispersed in a milk gel system. It can be both insufficient and costly to vary factors at a large scale when formulating. Smaller scales offer more versatility and flexibility at lower cost. However, the conditions must be adapted to be as realistic as possible in order to obtain the same fat droplet sizes, interfacial composition, protein network, set gel after acidification, and microgel dispersion (stirred gel) after shearing, as described above in the real product (Fig. 1). Several authors have made stirred milk gels at lab scale to study their properties (Cayot et al., 2008; Chever et al., 2014; Krzeminski et al., 2011; Laiho et al., 2017; Lee and Lucey, 2006). However, in these studies there were still some discrepancies between small and large scale processes. For example, in the lab, even though this step can significantly modify the structure of microgel particles (Mokoonlall et al., 2016), stirring was generally performed either by blending (by hand, in a blender or using an overhead stirrer) or by extrusion (needle) with non-evaluated reproducibility. There is currently no documented process that was specifically designed and validated to well mimic a large scale process typical of that commonly used (i.e. in terms of unit operations, temperatures and shearing) to make stirred fermented milk gels at lab scale using glucono-δ-lactone (GDL) acidification and with a focus on the validity and transferability of the process.

The aim of the present study was to design and validate a lab-scale process to produce stirred milk gels in conditions that match those at pilot scale. A reverse engineering logic based on the structural and textural properties was used and applied step by step (*i.e.* for each unit operation). Several reference samples of stirred fermented milk gels were produced at pilot scale and were carefully characterized to define the target microstructure and rheology. The scale-down rational design was carried out by listing the different unit operations (homogenization, acidification and stirring) comprising the pilot scale process and choosing series of lab-scale unit operations able to perform them. The unit operations were assembled in a small manufacturing line, in which each process step could be studied and used separately. The relevance and sensitivity of this lab-scale process line were proven by comparing stirred milk gel containing different fat and protein concentrations and produced both at lab-scale and with the pilot plant.

**Fig. 1** The typical unit operations of stirred fermented milk production are listed in the middle column, from ingredients at the top to the conditioning at the bottom. The physico-chemical conditions applied throughout the process are listed on the left hand column. The matrix microstructure and its changes during the process are schematized in the right hand column. Adapted from FAO (2013); Lee and Lucey (2010); Mokoonlall et al. (2016); Sodini et al. (2004).

# 2. Materials and methods

## 2.1. Raw materials

Purified water was obtained using a Milli-Q purification system (Millipore, Merck, Germany) and its conductivity was $6.6\times10^{-5}$ $S.m^{-1}$. Skim milk powder (SMP, 25.1 wt% proteins, 18.4 wt% caseins, 6.7 wt% whey proteins of which 68.1% native) was provided by Eurosérum (Sodiaal, Port-sur-Saone, France) and additive-free commercial pasteurized liquid cream (cream) (32.5 wt% fat, 2.2 wt% protein) was purchased from Alsace Lait (Hoerdt, France). Pregelatinized modified rice starch (between 2 and 8 µm in diameter) was provided by the dairy company which made the pilot references. GDL (purity ≥ 99.0%) was purchased from Sigma-Aldrich (Saint-Quentin Fallavier, France).

## 2.2. Manufacture of stirred milk gels at pilot and lab scales

### *2.2.1. Fermented stirred milk gel references produced at pilot scale (100 kg)*

A French dairy company kindly provided a reference of high-fat stirred fermented milk gel produced using their fully controlled pilot process (pilot plant with 100 kg batches). This reference was the result of traditional processing of stirred yoghurt-type products (Fig. 1), including mixing, pasteurization, dynamic homogenization, lactic fermentation at 30 °C and stirring. It was produced from skim milk, cream, SMP and 1.0 wt% starch, with final target contents of 3.1 wt% protein and 10.0 wt% fat (PilotREF(10)). Since fat content is known to impact textural and structural properties, a pilot reference with lower fat content was also manufactured, with a protein content of 3.1 wt% and a fat content of 6.0 wt% (PilotREF(6)). These two references of pilot fermented stirred milk gels were made twice, in two different weeks (four repetitions of each in total). Lactic acidification over time was monitored by the company and lasted 10 ± 1 h.

### *2.2.2. Stirred acid milk gel made at lab scale (1.5 kg) and tested parameters*

Depending on the desired composition, the mix was prepared from reconstituted skim milk, with or without cream and rice starch. The reconstituted skim milk was prepared by dispersing SMP in purified

water under continuous stirring at 250 rpm for 15 min at room temperature and then kept overnight at 8 °C to ensure proper hydration. Depending on the required fat content, cream was added to the reconstituted skim milk and the mix was achieved aiming for a constant final protein content of 3.1 wt%. In some of the mixes, 1.0 wt% of rice starch was added. The pasteurization heat-treatment was carried out in a thermostatically controlled water bath, by holding the sample temperature at 80 ± 2 °C for 30 min. Homogenization was performed by sonication (20 kHz, VCX 130, Sonic & Materials, UK) at 130 W for 15 effective minutes with 10 s pulses and a 13 mm probe. These sonication parameters were selected in preliminary tests on dairy emulsions to obtain droplet sizes similar to those of commercial creams. They remained to be validated on the system under study. Once homogenized, the mix was cooled and then acid-gelled with GDL added at different concentrations (0.50, 0.75, 1.00, 1.25, 1.30, 1.50 wt%), under a constant temperature of 30 ± 1 °C (heat chamber). This temperature was chosen in order to be representative of the pilot process. After the resulting set gel was checked to be sure the pH was below 4.5 ± 0.1, it was stored for one week at 8 °C or stirred before being stored in the same conditions as the set gel (termed 'lab stirred milk gel'). Different types of stirring were tested using different pump flow rates, two filter pore sizes and with or without ultra-smoothing. The gel was first coarsely broken up with a spatula, poured into a beaker (1 L), pumped through a peristaltic pump at different flow rates (50, 100, 200, 300 mL.min$^{-1}$) (L/S Precision Console, Masterflex, Gelsenkirchen, Germany) via two successive pipes (one 40 cm in length with a diameter if 7 mm, and one 100 cm in length with a 3 mm diameter) and then through one plastic mesh filters placed in filter holder (0.5 or 1 mm pores). When the pre-stirred gel was ultra-smoothed, it was passed once through a rotor/stator (Polytron PT 3100 D, PTG 36/4 probe, Kinematica AG, Switzerland) at 1,500 rpm, in batches of 250 mL. Finally, the stirred gels were placed in 100 mL pots at 26 ± 1.2 °C and stored at 8 ± 1.0 °C for one week. Details on the composition and a summary of the parameters tested in each unit operation are listed in Table 1. Two batches of the final lab-scale stirred milk gels with 10 wt% fat (LabREF(10)) and with 6 wt% fat (LabREF(6))) were made in two different weeks (i.e. two repetitions of each).

**Table 1** Summary of the parameters tested and corresponding sample names. The protein content of all the samples was 3.1 *wt%* and pasteurization was performed at 80 °C for 30 min. The amounts of fat, rice starch and GDL are given in *wt%*, the pumping rate in *mL.min*$^{-1}$, the filter pore size in *mm* and ultra-smoothing in *rpm.*

## 2.3. Analysis of particle size distribution by laser diffraction

The particle size distributions were obtained by laser diffraction with a MasterSizer 2000 (Malvern Instruments, UK). The size distributions of the fat globules were measured either in the cream, mixes or milk gels (Mie theory, 1.33 RI for water, 1.47 RI for milk fat) (three repetitions of each). The mixes and cream were diluted 1:10 in purified water and the milk gels were diluted 1:10 in a 1 wt% sodium dodecyl sulfate (SDS) solution to disrupt both the protein network and the aggregation of the fat droplets. The size distributions of the microgels were measured in the stirred milk gels (Fraunhofer theory because microgels are bigger than the laser beam, which is 633 nm), by diluting them (1:10) in purified water (three repetitions

of each). The size below which there were 50% (median diameter, d(0.5), µm) and 90% (d(0.9), µm) of the sample particles and the width of the distribution (span, Equation 1) were recovered.

$$span = \frac{d(0.9) - d(0.1)}{d(0.5)} \quad \text{Equation 1}$$

## 2.4. Monitoring of gelation by DWS during acidification

The acidification kinetics were monitored over time using a multichannel C3060 system (Consort bvba, Turnhout, Belgium). Probes were placed in the products just before GDL was added (pH about 6.5) and maintained until a pH of 4.5 was reached, when possible. Immediately after GDL was added, gelation was monitored using DWS measurement over time (Rheolaser LAB6®, Formulaction, Toulouse, France) using the protocol of Rohart et al. (2016). This DWS method made it possible to obtain the elasticity index (EI, $nm^{-2}$) from the mean square displacement of particles at low decorrelation time (< 0.1 s), the median diameter of the moving particles (i.e. the fat droplets) measured by laser diffraction (2 µm) and the diameter of a model particle used for calibration (TiO2, 1 µm). Since the variability of the acidification kinetics was only ± 1.0% (according to 10 repetitions carried out before the present study), monitoring of the optimization of acid gelation with different GDL concentrations was made once.

## 2.5. Measurement of textural properties

### *2.5.1. Texture analysis by back-extrusion*

Part of the textural properties were measured using a back-extrusion test performed with a TA.HD texture analyzer (Stable Micro System, Godalming, UK). Samples were prepared in 40 mL plastic cups (weight about 30 g, internal diameter 28 mm). Set acid milk gels were poured directly into the cups after GDL was added (before acid gelation) whereas stirred milk gels were poured into the cups after stirring (three repetitions of each). Milk gels were compressed using a flat disc (25 mm diameter, height 2 mm) at a crosshead speed of 1 $mm.s^{-1}$ and a depth of 20 mm (1.5 mm gap). During compression, the normal force (N) increased quite linearly until it reached a plateau. The value of the average force of the plateau ($F_P$, N) was chosen to represent the firmness of the sample.

### *2.5.2. Rheological properties in conditions close to the oral process*

Rheological properties of the stirred milk gels (at lab and pilot scales) were evaluated under conditions that imitated the oral process as much as possible, using the method of Huc et al. (2016). After standardized mixing (consisted in mixing each pots using a small teaspoon rotated four times from the bottom to the top of the pot), a small amount of each sample was placed on a MCR 301 rheometer (Anton Paar, Graz, Austria) at a temperature of 10 °C, using a plate-plate system (steel serrated parallel plate, diameter 5 cm). Measurements were carried out while the temperature was increased from 10 °C to 25 °C (1 mm gap). First, the viscoelastic properties were measured (0.01-10 Hz frequency, 0.1 % strain). Second, viscosity was measured at 60 $s^{-1}$ while the temperature was increased from 10 °C to 25 °C (0.6 $°C.s^{-1}$ heating rate), then the

viscoelastic properties were measured at 25 °C (0.01-10 Hz frequency, 0.1 % strain) after 1 min at 25 °C under 60 $s^{-1}$ shearing. Three repetitions were made for each sample. Several indicators were chosen to describe the rheological properties: viscosity ($\eta_0$, Pa.s) and storage moduli ($G'_0$, Pa, 1 Hz) at the beginning of the mimicked oral process, and viscosity ($\eta_f$, Pa.s) and storage moduli ($G'_f$, Pa, 1 Hz) at the end of the mimicked oral process.

## 2.6. Statistical analysis

Statistical analyses were performed using XLSTAT 2015.1 software (Addinsoft, Paris, France). Analysis of variance (one way ANOVA) was performed to evaluate differences between average values using Tukey's test. A significance level of $p < 0.05$ was used. Principal component analysis (PCA) was used to map samples by analyzing several properties simultaneously.

# 3. Results and discussion

Since the present study used a logic of reverse engineering, the measured parameters were compared with those of stirred fermented gel references made at pilot scale, except for the back-extrusion measurement (because the transfer of the pilot-scale stirred milk gels into 40 mL cups would distort this measurement). However, the rheological analysis enabled the reliable comparison of the textural properties of the pilot- and lab-scale samples (Table 2).

## 3.1. Selection of the lab scale process parameters

### *3.1.1. Effect of sonication on the size distribution of fat droplets*

Fig. 2 shows the size distributions of fat droplets and distinguishes the cream, with a more monodispersed distribution and a median diameter of 3.3 µm, from all the other samples, which had smaller droplets (d(0.5) close to 0.5 µm). The size distributions of the pilot reference treated by high pressure (PilotREF(10)) and the lab-scale stirred milk gel treated by sonication (LabREF(10)) both displayed a shoulder with a first peak close to 0.2 µm and a second peak close to 0.5 µm. The mix obtained just after sonication of the cream (S-Mix) displayed a single peak, around 0.5 µm. The size distributions of the lab-scale samples treated by sonication (LabREF(10) and S-Mix) were broader than one of the pilot reference, with a tail up to larger diameters equivalent to those of fat droplets in cream.

**Fig. 2** Impact of sonication (S) on the size distribution of the fat droplets in the cream (dotted line) measured in the mix (S-Mix, dashed line). Comparison between the fat droplet size distributions in the lab-scale milk gel (LabREF(10)_SDS, solid line) and in the pilot reference (PilotREF(10)_SDS, pale grey solid line).

A median diameter of 0.2 ± 0.05 µm was obtained from skim milk (results not shown, 1:10 dilution in 1% SDS solution, 1.473 RI), suggesting that the first peak of the distributions of the pilot reference and of the lab-scale milk gel corresponds to components of the size of casein micelles, which have a median size of around 0.2 µm (Donato and Guyomarc'h, 2009). The sizes obtained after the high pressure treatment at pilot scale were as expected, in accordance with the literature (Floury et al., 2000). The results also showed that sonication reduced the size of fat droplets in the same range as pilot homogenization, but the final size distribution was larger (increased polydispersity). Sonication (lab-scale) reduces the droplet size mainly close to the probe, and by cavitation and by the resulting turbulence, whereas high pressure homogenization (pilot-scale) does it very uniformly by shearing, micro-turbulence and cavitation. This difference in the transmission of energy to the emulsion possibly explain the observed differences in size distributions. The results are consistent with the literature, where homogenization was shown to be more effective than sonication under similar conditions for emulsions of sunflower oil (20 %) stabilized with whey protein isolate (Calligaris et al., 2018).

### *3.1.2. Effect of GDL concentration on acidification kinetics and on the textural properties of the resulting set milk gel*

The amount of GDL clearly influenced the kinetics of acidification and gelation of the lab-scale set milk gel (Fig. 3). The GDL acidification kinetics shown in Fig. 3 A all began with a marked decrease in pH, which subsequently slowed down and levelled off. The 4.5 target pH was never reached with 0.50 wt% GDL, but was reached in 9 h to 1 h 15 when the concentration of GDL increased from 0.75 to 1.50 wt%. The final pH reached after 18 h of acidification was 4.9, 4.4, 4.2, 3.9 and 4.0 for 0.50, 0.75, 1.00, 1.25 and 1.50 wt% GDL respectively. The GDL acidification kinetics were also faster than the pilot fermentation kinetics, which reached the target pH of 4.5 in 10 h. The acidification kinetics obtained with GDL differed from that during pilot lactic fermentation, which began with a lag time (bacterial growth), then decreased, slowed down and ended with post-acidification in the product (Fig. 3 A).

**Fig. 3 A** Acidification kinetics (GDL) of the lab-scale set milk gel and the fermentation kinetic of the pilot-scale set milk gel with a target pH of 4.5 (horizontal line). **B** Changes in the elasticity index over time (gelling). **C** Changes in the elasticity index during acidification. The increase in the concentration of GDL is shown by the change in the color of the curves from dark to pale grey. The variability of the acidification kinetics was ± 1.0%.

The acidification kinetics obtained with GDL are in good agreement with those shown in the literature (Jacob et al., 2011; Lucey et al., 1998; Rohart and Michon, 2016). It is known that the pH transitions that occur during acidification are associated with different phenomena (solubilization of micellar calcium phosphate, dissociation then re-association of the micellar caseins, suppression of electrostatic repulsion and interaction of casein micelles) leading to gelation (Lee and Lucey, 2010). The different kinetics of pH reduction by lactic ferments or with different concentrations of GDL can therefore influence these phenomena and produce different microstructures (Azim et al., 2010; Jacob et al., 2011; Lucey et al., 1998).

The elasticity index was monitored using the non-invasive DWS method provides information about the impact of kinetics on gelation and the structure depending on the GDL concentration.

The elasticity indexes shown in Fig. 3 B allow gelation to be monitored over time. They all started to increase after a lag time which decreased from 2 h 30 to 30 min when the concentration of GDL increased from 0.50 to 1.50 wt%. These curves also distinguish the samples with 0.50 and 0.75 wt% GDL, for which the elasticity indexes reached a plateau before decreasing to $4.2\times10^{-3}$ $nm^{-2}$, from the other samples, for which the elasticity indexes all reached a same higher plateau of $2.0\times10^{-2}$ $nm^{-2}$. The final plateau of the elasticity index was reached in 8 h, 3 h 45 and 2 h 45 for 1.00 wt%, 1.25 wt% and 1.50 wt% GDL, respectively, the times at which the pH reached 4.5. Fig. 3 C shows that EI, reflecting gelation, started to increase from a pH of 5.5 in all the samples. Finally Fig. 3 C highlights the fact that the EI obtained with 0.50 wt% GDL decreased without ever reaching a pH of 4.5 and the EI obtained with and 0.75 wt% GDL decreased as soon as the pH of 4.5 was reached.

The decrease in the elasticity index may be due to syneresis occurring within the gel leading to its brittleness and/or collapse, but it could also be due to the expulsion of serum due to syneresis on the surface and/or on the walls of the glass tube during the measurement by DWS (Rohart et al., 2016). Back-extrusion measurements were carried out on the set gels after seven days of storage (4 °C) and the resulting normal force values at the plateau increased from 0.7 ± 0.1 N for 0.50 % GDL to 1.1 ± 0.1 N for 0.75 wt%, before reaching a maximum value of 1.6 ± 0.2 N for 1.00, 1.25 and 1.75 wt% GDL. These results show that the decrease in the elasticity index of gels formed with 0.50 and 0.75 wt% GDL was due to their brittleness certainly caused by less cohesiveness of the gels. Moreover, since the amounts of 1.25 and 1.50 wt% GDL meant that the 4.5 target pH was reached too rapidly (< 3 h) compared to the pilot reference (10 h), the amount of 1.00 wt% was most suitable for 3.1 wt% protein and was thus selected as the standard value for all further experiments. This value is in accordance with the literature, where a pH of 4.5 was also obtained with 1 % GDL and 3.5 wt% protein in acidified milk (Azim et al., 2010).

### *3.1.3. Effect of the stirring parameters on the size distributions of the microgels and the texture of stirred milk gels*

All the stirrings applied made it possible to obtain microgel sizes quite close to those of the pilot reference for the lab-scale stirred milk gels (Fig. 4). Based only on the median diameters, the stirring steps without ultra-smoothing led to a d(0.5) of around 19 µm (P200-F0.5) and 16 µm (P200-F1), which were closer to the pilot reference (PilotREF(10)) (18 µm) than the sample treated with ultra-smoothing (P200-F1-US), for which the d(0.5) was around 14 µm (Fig. 4 A). However, the overall size distributions of the two samples without ultra-smoothing contained larger particles (d(0.9) > 41 µm and span > 2.0) than the pilot reference and the ultra-smoothed sample (d(0.9 < 38 µm and span < 1.9) (table below Fig. 4 A). In addition, Fig. 4 B shows that the textural variability of the P200-F1 sample was greater than that of the two other samples. Fig. 4 B also shows that ultra-smoothing significantly reduced the texture of P200-F1-US, compared to the two other samples.

The size distributions of the microgels obtained with the present lab scale stirring steps were smaller than those reported in the literature (by hand, with a blender or overhead stirrer, using needle extrusion),

which usually ranged between 20 and 150 µm (Cayot et al., 2008; Chever et al., 2014; Laiho et al., 2017). The processes investigated in this study are therefore closer to the pilot reference, which underwent industrial-type stirring. In the literature, several authors demonstrated that the presence of particles larger than 40 µm led to the product being perceived as "grainy" or that the presence of particles larger than 150 µm even seemed to limit the perception of "creamy" (Cayot et al., 2008; Hahn et al., 2012). These criteria led to choose stirring with ultra-smoothing, especially since grains were visible in the samples without ultra-smoothing after seven days of storage.

The samples obtained with the pump flow rates of 50, 100, 200 and 300 mL.min$^{-1}$ (1 mm filter (F1) and ultra-smoothing) did not differ significantly either in the sizes of the microgels (Fig. 4 C), or in d(0.9) and spans, or in the plateau values measured by back-extrusion (Fig. 4 D). The variability of the plateau value obtained with a pump flow rate of 300 mL.min$^{-1}$ was the only one parameter that differed, i.e. was higher than the others. A flow rate of 200 mL.min$^{-1}$ was thus chosen to work with the fastest possible pump flow rate, without having too much variability. The final lab-scale process selected was the one of P200-F1-US, with pasteurization at 80 °C for 30 min, homogenization by sonication (130 W for 15 effective minutes with a 10 s pulse), acidification with 1.0 wt% GDL and stirring after gelling using filter pores 1 mm in diameter, pump flow rate of 200 mL.min$^{-1}$ and ultra-smoothing at 1,500 rpm.

**Fig. 4** Effect of pore sizes of filters and ultra-smoothing (**A**, **B**) and of the pump flow rates (**C**, **D**). Size distribution of microgels with corresponding d(0.9) and span below (**A**, **C**) and average value of the normal force at the plateau measured by back-extrusion ($F_P$ force, N) after 7 days of storage of stirred acid milk gels (**B**, **D**).

## 3.2. Comparison of the lab-scale stirred milk gel with the pilot reference based on the impact of a reduction in the fat content

The final lab-scale process was used to produce two batches of lab-scale stirred milk gels with 6 wt% fat (LabREF(6)A and LabREF(6)B) and two other batches with 10 wt% fat (LabREF(10)A and LabREF(10)B). In parallel, the reference pilot-scale process was used to produce two batches of pilot-scale stirred milk gels with 6 wt% fat (PilotREF(6)A and PilotREF(6)B) and two other batches with 10 wt% fat (PilotREF(10)A and PilotREF(10)B). Both the rheological properties and the microgel sizes were systematically measured three times in all the batches and averaged (Fig. 5). The properties of the two batches (A and B) of all types of stirred milk gels (LabREF(6), LabREF(10), PilotREF(6) and PilotREF(10)) were averaged (Table 2).

The rheological indicators ($G'_0$, $G'_f$, $G'_0$, $\eta_f$) obtained at lab scale were lower than those obtained at pilot scale (PilotREF(10) > LabREF(10) and PilotREF(6) > LabREF(6)). The microgels of the lab-scale stirred milk gels were slightly larger than those of the pilot references (PilotREF(10) < LabREF(10) and PilotREF(6) < LabREF(6)) (Table 2). However, the impact of the reduction in fat content from 10 to 6 wt% on the rheological properties did not differ significantly between lab and pilot scales (*i.e.* the reduction in

firmness was of similar order in both cases). The size of the microgels increased with the reduction in fat and this increase was slightly higher in the pilot reference than in the lab-scale stirred milk gel. Since the orders of the reduction in firmness and of the increase in microgel size were similar at both pilot- and lab-scales, the designed lab-scale stirred milk gel is therefore representative of the pilot reference.

**Table 2** Rheological ($G'_0$, $G'_f$, $\eta_0$, $\eta_f$) and structural indicators (median diameter of the microgels) of lab-scale stirred milk gel and the pilot reference with 10 and 6 wt% fat contents. The impact of fat reduction corresponds to the rate of variation (%) between 10 wt% and 6 wt% of fat in the lab or pilot products. Values with different letters in the same row differ significantly ($p < 0.05$). Values with a different number of asterisk(s) (*) in the same row differ significantly ($p < 0.05$).

The principal component analysis (PCA) plotted in Fig. 5 mapped the samples as a function of all of the properties detailed in Table 2 for two batches (A and B) of each (LabREF(10), LabREF(6), PilotREF(10) and PiloteREF(6)). The selected 2D projection (F1; F2) displays more than 95% of the total information. According to the correlation loading plot (Fig. 5 A), the F1 axis explained more than 86% of the total information, through the rheological properties, and a diagonal axis was linked to the median diameter of the microgels. Consequently, the score plot (Fig. 5 B) shows that the samples were mainly distributed on F1 and that they were mainly distinguished based on the production scale (lab or pilot scale) and the fat content (6 or 10 wt%). It also shows that products made with 10 wt% fat were more variable than the products made with 6 wt% fat on the F1 axis from one production session to another. Finally, it shows that the pilot products varied more than the lab products on the microgel axis from one production to another.

As previously demonstrated, the fat droplet distribution in the lab-scale stirred milk gel displayed bigger fat sizes than the pilot reference. This may explain part of the differences in the rheological properties between lab and pilot scales, since smaller and more monodispersed droplets tend to strengthen the protein network (Ye and Taylor, 2009). Moreover, differences in heat treatment and homogenization may change the quantity and quality of the proteins adsorbed at the interface (Cano-Ruiz and Richter, 1997; Millqvist-Fureby et al., 2001; O'Sullivan et al., 2014) and the nature of the interface affects the textural properties. For example, reports in the literature showed that when whey proteins were adsorbed as heat-aggregated, they strengthened the dairy matrix more than if they were adsorbed as native proteins (Cho et al., 1999). As shown previouslye, the acidification kinetics differed between the lab-scale stirred milk gel acidified by GDL and the fermented pilot references. The literature reports that the microstructure and hence the rheological properties of the milk gels obtained by GDL acidification or fermentation were not the same (Lucey et al., 1998). In addition, lactic bacteria may produce exopolysaccharides, which are known for their texturizing properties (Bouzar et al., 1997), and this may also explain why the pilot references was firmer (rheological properties) than the lab-scale stirred milk gels. In addition, GDL acidification is known to be less variable than acidification by fermentation, which probably explains the greater variability of the pilot references. Finally, the greater variability with 10% fat may be due to a higher concentration of fat droplets, whose size and interface may vary from one production to another.

**Fig. 5** Correlation loading plot of the rheological ($G'_0$, $G'_f$, $\eta_0$, $\eta_f$) and structural indicators (median diameter of the microgel "d(0.5) µgel") (**A**) and corresponding score plot of the lab and pilot stirred milk gels with 10 and 6 wt% fat contents and two different batches labeled with the letters A and B (**B**).

In addition, mixes were made by emulsifying either anhydrous milk fat (AMF, melting temperature of 30 °C) or its low-melting fraction (olein, melting temperature of 7-14 °C) in skim milk. The final lab-scale process and the reference pilot-scale process were both used to make stirred milk gels from the AMF and olein mixes. The use of olein instead of AMF resulted in a decrease in the firmness at 8 °C ($G'_0$ and $\eta_0$) of the lab-scale stirred milk gels (-45%) and of the pilot-scale stirred milk gels (-55%) (results not shown). These results are evidence for the relevance and efficiency of the final lab-scale process to test and vary other levers of formulation.

# 4. Conclusions

The lab-scale process designed using a logic of reverse engineering made it possible to reliably mimic what occurred at pilot scale since the microstructural and textural properties of the lab-scale stirred milk gels were consistent with the pilot references (fermented milk gels). This lab process makes it possible to test twice as many recipes per production day with more than 60 times less raw materials than required for the pilot process. The final lab-scale stirred milk gels were less firm than the pilot reference, probably because the size distributions of their fat droplets were slightly wider and their acidification kinetics were not exactly the same as for lactic fermentation. However, the lab-scale stirred milk gel were less variable and were just as sensitive to variations in formulation as the pilot reference. The rational design of the lab scale process resulted in a lab-scale process line (1.5 kg batches), in which part or all of the unit operations can be varied and tailored. Moreover, it is versatile and flexible, making it possible to test parameters that could otherwise be difficult and expensive to test at larger scales. It is therefore a good formulation tool, which allows performing screening designs and which is adaptable to other stirred gel type particle systems.

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442

**Fig. 1** The typical unit operations of stirred fermented milk production are listed in the middle column, from ingredients at the top to the conditioning at the bottom. The physico-chemical conditions applied throughout the process are listed on the left hand column. The matrix microstructure and its changes during the process are schematized in the right hand column. Adapted from FAO (2013); Lee and Lucey (2010); Mokoonlall et al. (2016); Sodini et al. (2004).

**Table 1** Summary of the parameters tested and corresponding sample names. The protein content of all the samples was 3.1 *wt%* and pasteurization was performed at 80 °C for 30 min. The amounts of fat, rice starch and GDL are given in *wt%*, the pumping rate in $mL.min^{-1}$, the filter pore size *mm* and ultra-smoothing in *rpm.*

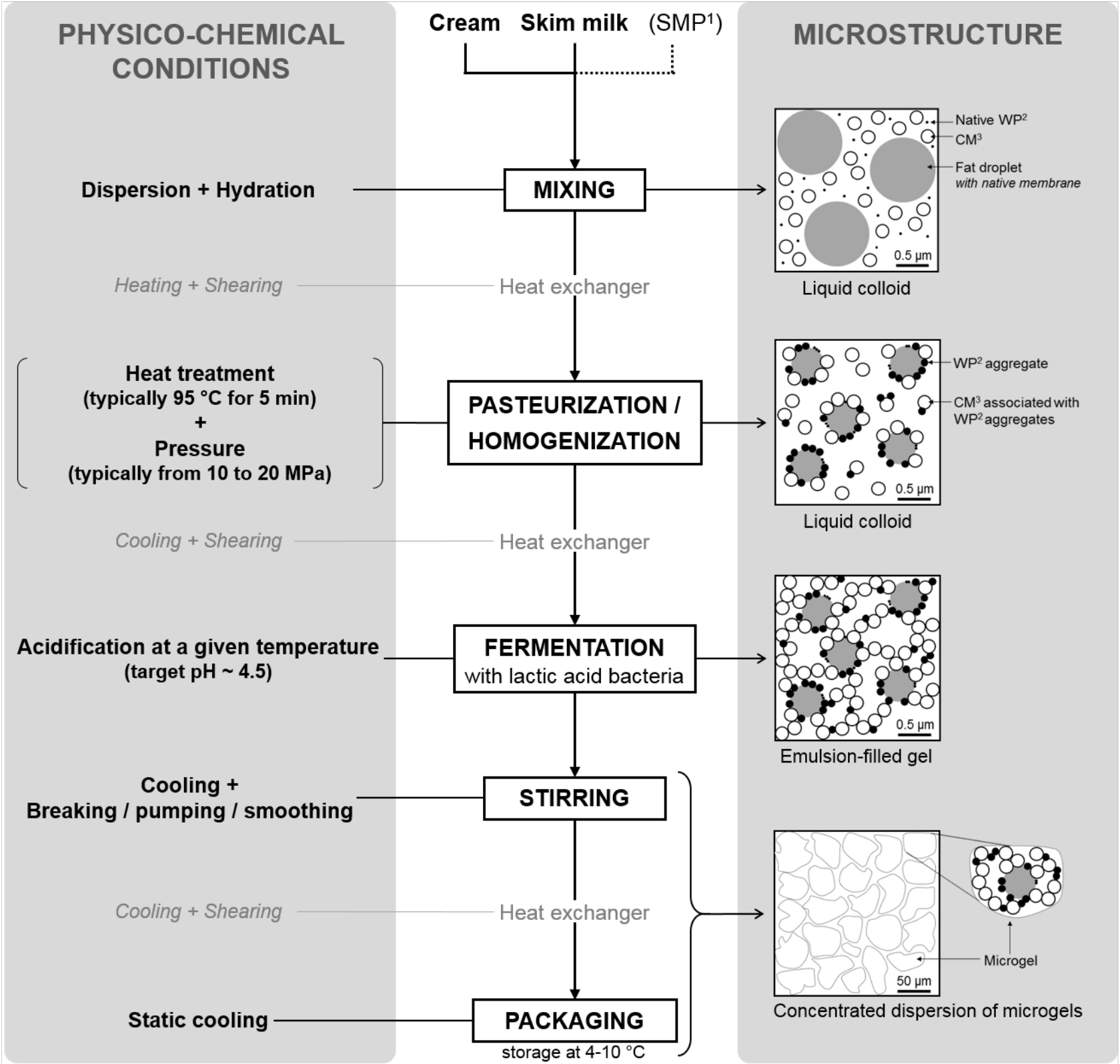
**Fig. 2** Impact of sonication (S) on the size distribution of the fat droplets in the cream (dotted line) measured in the mix (S-Mix, dashed line). Comparison between the fat droplet size distributions in the lab-scale milk gel (LabREF(10)_SDS, solid line) and in the pilot reference (PilotREF(10)_SDS, pale grey solid line).

**Fig. 3 A** Acidification kinetics (GDL) of the lab-scale set milk gel and fermentation kinetic of the pilot-scale set milk gel with a target pH of 4.5 (horizontal line). **B** Changes in the elasticity index over time (gelling). **C** Changes in the elasticity index during acidification. The increase in the concentration of GDL is shown by the change in the color of the curves from dark to pale grey. The variability of the acidification kinetics was ± 1.0%.

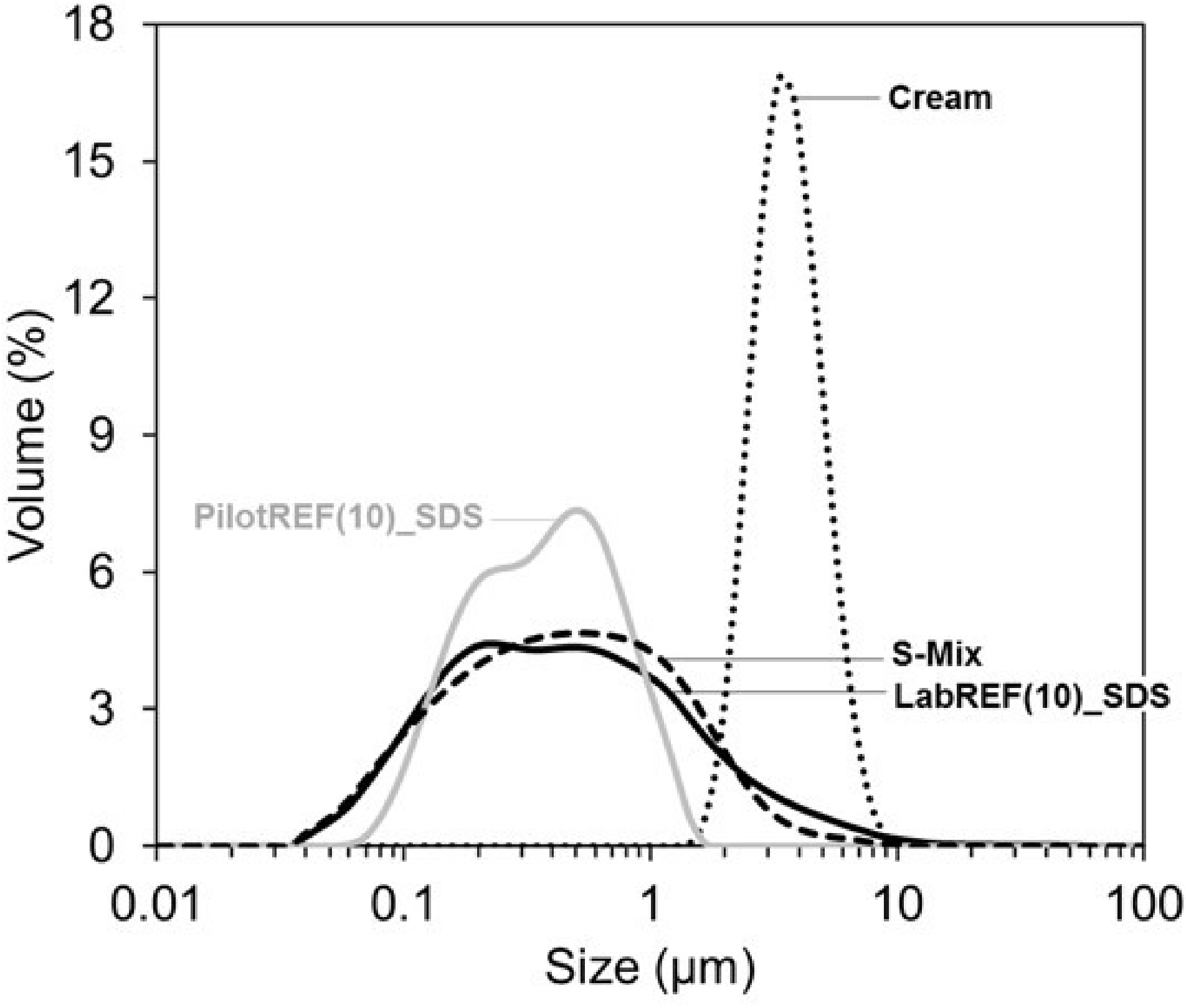
**Fig. 4** Effect of pore sizes of filters and ultra-smoothing (**A**, **B**) and of the pump flow rates (**C**, **D**). Size distribution of microgels with corresponding d(0.9) and span below (**A**, **C**) and average value of the normal force at the plateau measured by back-extrusion ($F_P$ force, N) after 7 days of storage of stirred acid milk gels (**B**, **D**).

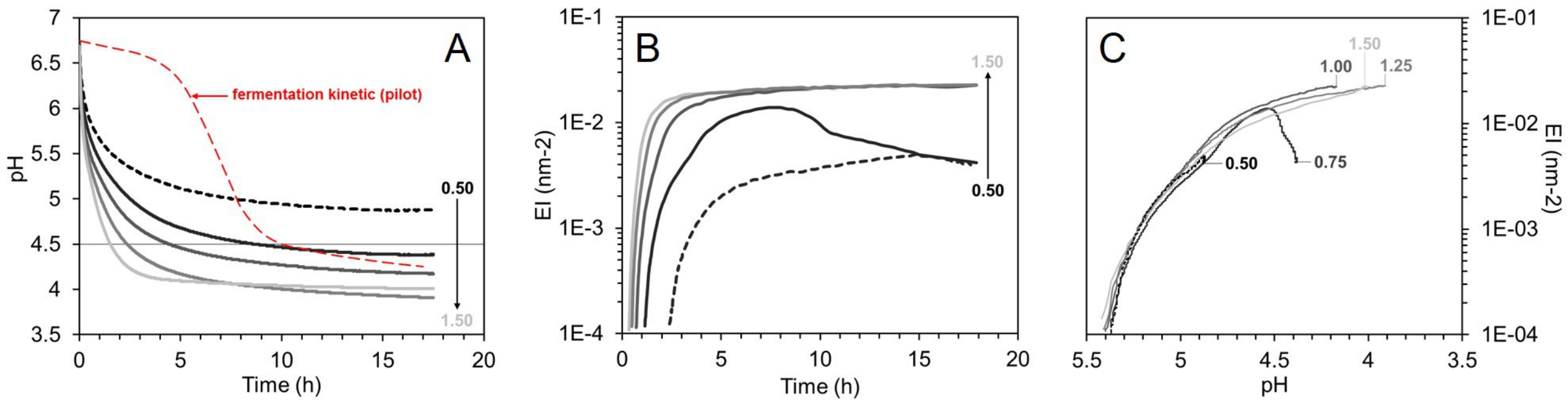
**Table 2** Rheological ($G'_0$, $G'_f$, $\eta_0$, $\eta_f$) and structural indicators (median diameter of the microgels) of lab-scale stirred milk gel and the pilot reference with 10 and 6 wt% fat contents. The impact of fat reduction corresponds to the rate of variation (%) between 10 wt% and 6 wt% of fat in the lab or pilot products. Values with different letters in the same row differ significantly ($p < 0.05$). Values with a different number of asterisk(s) (*) in the same row differ significantly ($p < 0.05$).

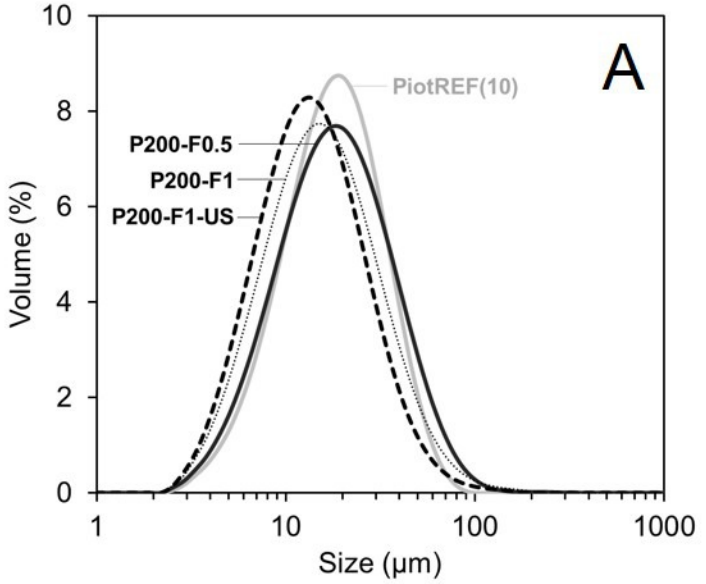
**Fig. 5** Correlation loading plot of the rheological ($G'_0$, $G'_f$, $\eta_0$, $\eta_f$) and structural indicators (median diameter of the microgel "d(0.5) µgel") (**A**) and corresponding score plot of the lab and pilot stirred milk gels with 10 and 6 wt% fat contents and two different batches labeled with the letters A and B (**B**).



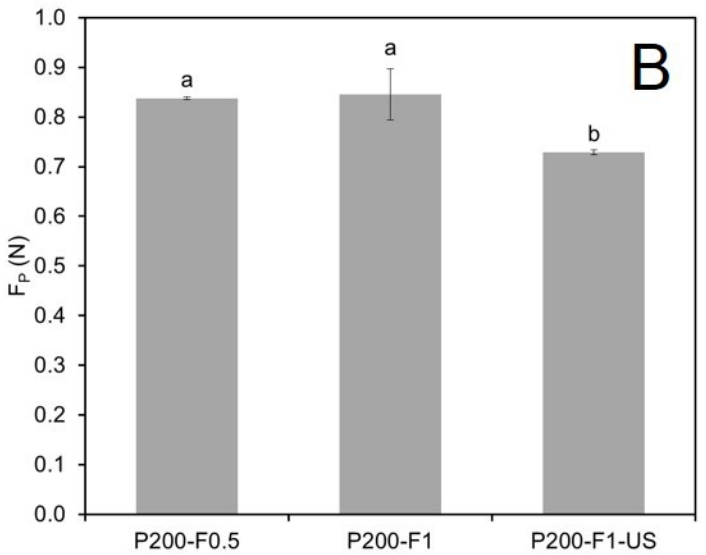

*[1]SMP: Skim milk powder; [2]WP: Whey protein; [3]CM: Casein micelle*

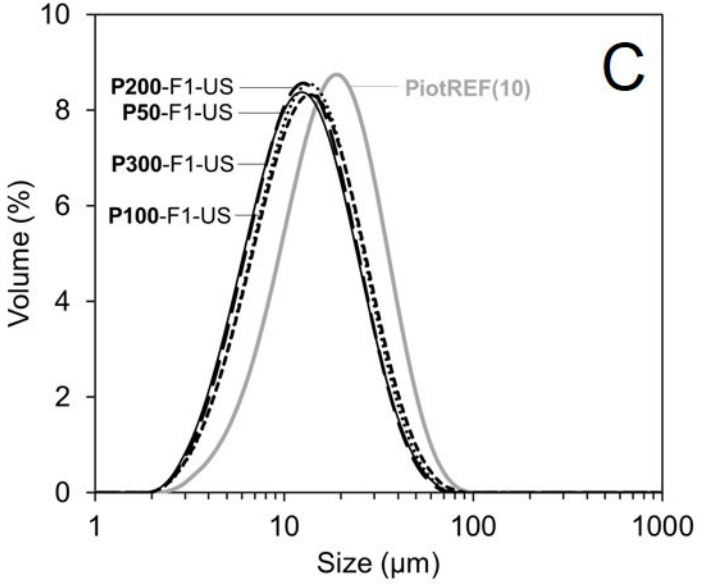

18
15
12
9
6
3
0
Volume (%)
Cream
PilotREF(10)_SDS
S-Mix
LabREF(10)_SDS
0.01
0.1
1
10
100
Size (µm)


A
fermentation kinetic (pilot)
0.50
1.50
pH
Time (h)
B
1.50
0.50
EI (nm-2)
Time (h)
C
1.50
1.00
1.25
0.50
0.75
pH
EI (nm-2)

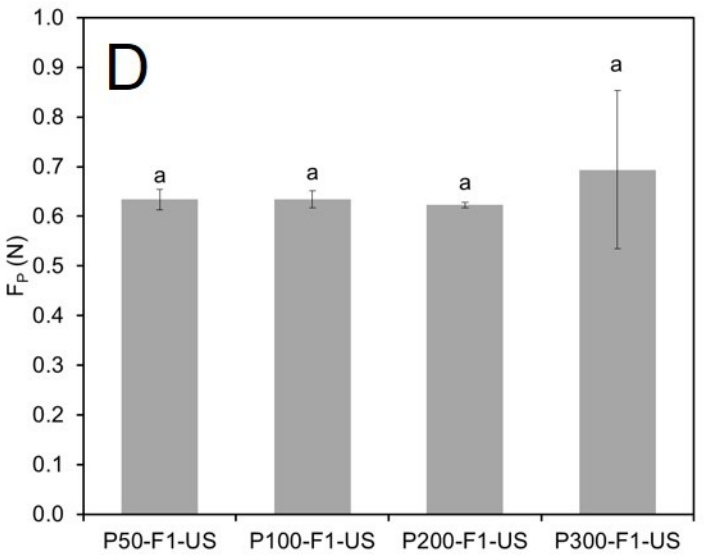


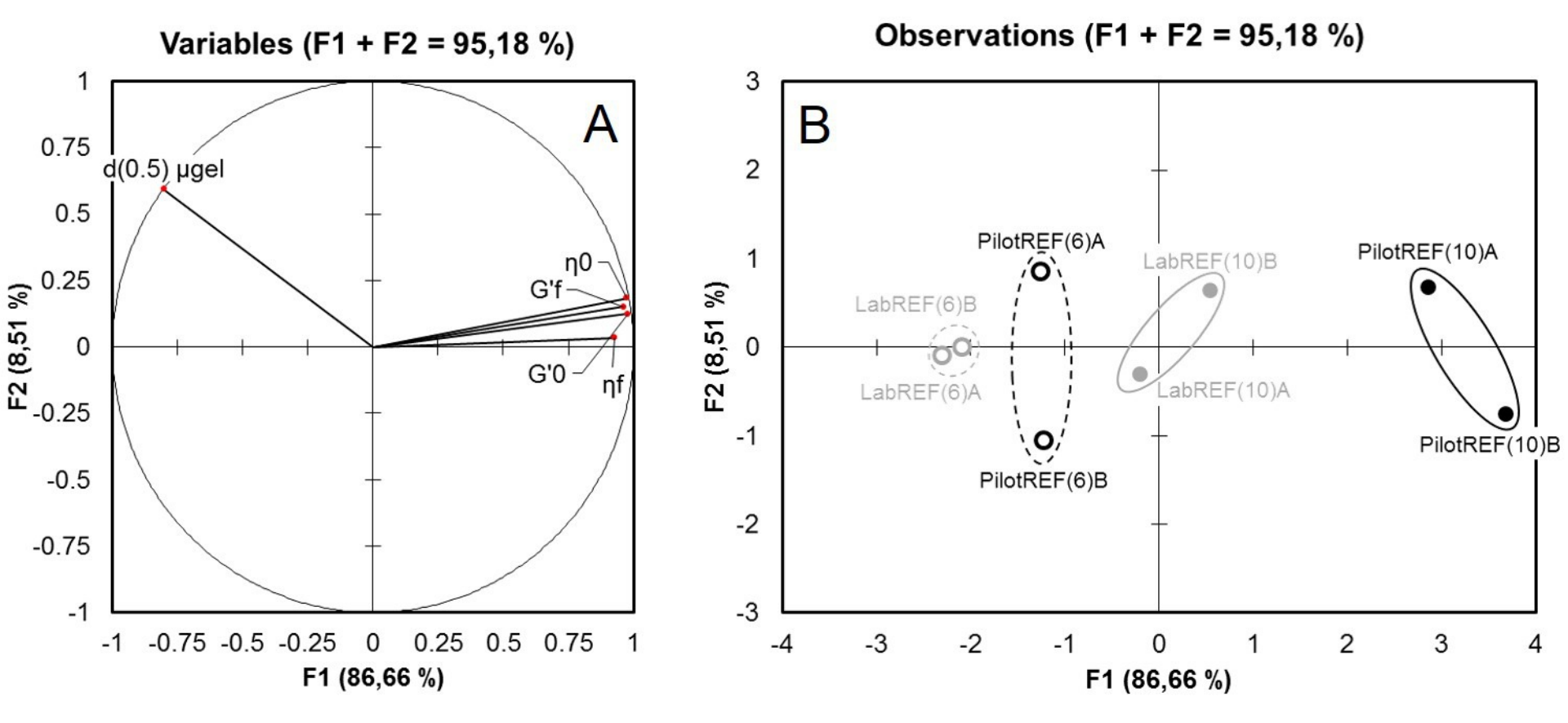

| | PilotREF(10) | P200-F0.5 | P200-F1 | P200-F1-US |
|---|---|---|---|---|
| d(0.9) | 37.6 ± 1.2 $^{c}$ | 46.9 ± 0.3 $^{a}$ | 41.0 ± 2.2 $^{b}$ | 33.8 ± 0.2 $^{d}$ |
| span | 1.6 ± 0.0 $^{c}$ | 2.0 ± 0.0 $^{b}$ | 2.1 ± 0.2 $^{a}$ | 1.9 ± 0 $^{b,c}$ |



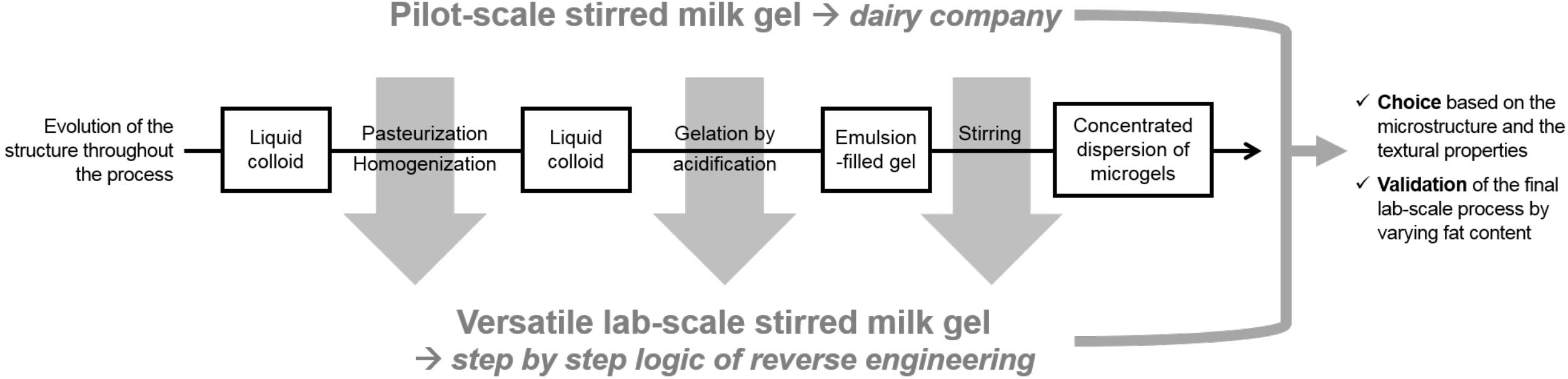

| | PilotREF(10) | P50-F1-US | P100-F1-US | P200-F1-US | P300-F1-US |
|---|---|---|---|---|---|
| d(0.9) | 37.6 ± 1.2 $^{a}$ | 31.0 ± 0.0 $^{b}$ | 32.3 ± 0.3 $^{b}$ | 33.8 ± 0.2 $^{b}$ | 29.3 ± 0.9 $^{b}$ |
| span | 1.6 ± 0.0 $^{b}$ | 1.8 ± 0.0 $^{a}$ | 1.8 ± 0.0 $^{a}$ | 1.9 ± 0.0 $^{a}$ | 1.8 ± 0.0 $^{a}$ |




Variables (F1 + F2 = 95,18 %)
A
d(0.5) µgel
η0
G'f
G'0
ηf
F1 (86,66 %)
F2 (8,51 %)
Observations (F1 + F2 = 95,18 %)
B
PilotREF(6)A
PilotREF(6)B
LabREF(6)B
LabREF(6)A
LabREF(10)B
LabREF(10)A
PilotREF(10)A
PilotREF(10)B
F1 (86,66 %)
F2 (8,51 %)

| Unit operation | Fat | Rice starch | GDL | Sonication (S) | Pumping (P) | Filter pore size (F) | Ultra-smoothing (US) | Nomenclature |
|---|---|---|---|---|---|---|---|---|
| Homogenization | 10 | - | 1.3 | Yes | - | - | - | S |
| Acidification | 10 | - | 0.5 | Yes | - | - | - | 0.5 |
| | 10 | - | 0.75 | Yes | - | - | - | 0.75 |
| | 10 | - | 1 | Yes | - | - | - | 1 |
| | 10 | - | 1.25 | Yes | - | - | - | 1.25 |
| | 10 | - | 1.5 | Yes | - | - | - | 1.5 |
| Stirring | 10 | 1 | 1 | Yes | 200 | 0.5 | - | P200-F0.5 |
| | 10 | 1 | 1 | Yes | 200 | 1 | - | P200-F1 |
| | 10 | 1 | 1 | Yes | 200 | 1 | 1500 | P200-F1-US |
| | 10 | 1 | 1 | Yes | 50 | 1 | 1500 | P50-F1-US |
| | 10 | 1 | 1 | Yes | 100 | 1 | 1500 | P100-F1-US |
| | 10 | 1 | 1 | Yes | 300 | 1 | 1500 | P300-F1-US |
| Final lab-scale stirred milk gel | 10 | 1 | 1 | Yes | 200 | 1 | 1500 | LabREF(10) |
| | 6 | 1 | 1 | Yes | 200 | 1 | 1500 | LabREF(6) |

| | LabREF(10) | LabREF(6) | *Impact of fat reduction (%)* | PilotREF(10) | PilotREF(6) | *Impact of fat reduction (%)* |
|---|---|---|---|---|---|---|
| $G'_0$ (Pa) | 350 ± 40 $^{b}$ | 180 ± 10 $^{c}$ | *- 49 ± 8** | 500 ± 40 $^{a}$ | 200 ± 35 $^{c}$ | *- 59 ± 7** |
| $G'_f$ (Pa) | 100 ± 25 $^{b}$ | 60 ± 6 $^{c}$ | *- 45 ± 8** | 140 ± 15 $^{a}$ | 70 ± 10 $^{c}$ | *- 52 ± 10** |
| $\eta_0$ (Pa.s) | 1.6 ± 0.3 $^{b}$ | 0.9 ± 0.1 $^{c}$ | *- 45 ± 5** | 2.6 ± 0.2 $^{a}$ | 1.2 ± 0.2 $^{c}$ | *- 55 ± 7** |
| $\eta_f$ (Pa.s) | 0.8 ± 0.1 $^{b}$ | 0.5 ± 0.1 $^{b}$ | *- 40 ± 7** | 1.7 ± 0.1 $^{a}$ | 0.9 ± 0.1 $^{c}$ | *- 50 ± 10** |
| d(0.5) of microgels (µm) | 19 ± 5 $^{a,b}$ | 22 ± 1 $^{a}$ | *+ 12 ± 6** | 17 ± 3 $^{b}$ | 20 ± 3 $^{a}$ | *+ 22 ± 3*** |


Pilot-scale stirred milk gel → dairy company
Evolution of the structure throughout the process
Liquid colloid
Pasteurization Homogenization
Liquid colloid
Gelation by acidification
Emulsion -filled gel
Stirring
Concentrated dispersion of microgels
✓ Choice based on the microstructure and the textural properties
✓ Validation of the final lab-scale process by varying fat content
Versatile lab-scale stirred milk gel
→ step by step logic of reverse engineering