Spray dried pre-quarg from over acidified milk and whey UF retentates

by

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Summary

UF retentates from skim milk concentrated up to 5 times and whey concentrated up to 10 times, pasteurized at 65°C for 20 min., were inoculated with 5 % *Lactobacillus acidophilus* starter and acidified for 24 hours to pH 3.5-3.8, then they were repasteurized and spray dried. Acidified milk or whey UF retentates powders contained 94-96 % dry matter 45-65 % protein, 6.2-7.8 % ashes, 13-15 % lactic acid, 19-23 % lactose and pH of 10 % water solution was 3.8-4.0.

Acidified and non-acidified milk and whey UF retentates powders had a total bacterial count (c.f.u.) of 4.2×10^4 to 5.5×10^5 per gram. The mould count was $1-10^2$ /g and yeasts $1-10$ /g. There was less than 10 Coliforms and Enterococci per gram.

Reconstituted quargs obtained by mixing acidified and non-acidified milk or milk and whey UF retentates powders with liquid skim milk or fermented milk were characterized by a total bacterial count (c.f.u.) of 4×10^7 to 1×10^{10} /g, moulds from 1 to 2×10^1 /g and yeasts from 1 to 5×10 /g. During 21 days of storage of reconstituted quargs at $6 \pm 2^\circ\text{C}$ Coliforms and Enterococci were absent in 1.0 g, in some samples moulds number increased up to 2×10^4 and yeasts number up to 3×10^3 . Total bacterial count was practically constant during three weeks of storage at 6°C.

Organoleptic quality of thus obtained quargs was good and no major changes were observed during 3 weeks of storage.

Key words:

Quarg, *L. acidophilus*, ultrafiltration, acidified whey retentate powder, acidified milk retentate powder.

Abbreviated title

Spray dried quarg.

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Résumé

Des rétentats d'ultrafiltration de lait écrémé concentré jusqu'à 5 fois et de lactosérum concentré jusqu'à 10 fois, chauffés à 65° C pendant 20 min, étaient inoculés avec 5 % de levain de *Lactobacillus acidophilus* et acidifiés à 43° C pendant 24 h jusqu'à pH 3,5-3,8, ils étaient chauffés à nouveau à 65° C pendant 20 minutes et séchés par atomisation.

Les poudres des rétentats du lait ou du lactosérum avaient une teneur égale à 94-96 % en matière sèche, 45-65 % en protéines, 6,2-7,8 % en cendres, 13-15 % en acide lactique, 19-23 % en lactose et le pH des solutions à 10 % était de 3,8 à 4.

Les poudres de rétentats de laits acidifiés et non-acidifiés avaient une flore totale mésophile de $4,2 \times 10^4$ à $5,5 \times 10^5$ /g; 1-100 moisissures/g et 1-10 levures/g. Le nombre de Coliformes et Enterocoques était inférieur à 10 par gramme.

Les fromages de type quarg étaient obtenus par mélange des poudres de rétentats de laits et de lactosérum acidifiés ou non-acidifiés avec du lait frais ou acidifié. Ces fromages avaient une flore totale mésophile de l'ordre de 4×10^7 à 1×10^{10} /g; 1-20 moisissures/g et 10-100 levures/g. Après 21 jours de conservation de fromages reconstitués à $6 \pm 2^\circ$ C le nombre de Coliformes et Entérocoques était inférieur de 10/g; le nombre de moisissures augmentait dans certains échantillons jusqu'à 2×10^4 /g et celui de levures jusqu'à 3×10^3 /g. La flore totale mésophile était pratiquement constante pendant trois semaines de stockage à 6° C. La qualité organoleptique des fromages était bonne et aucun changement n'était observé pendant 3 semaines de stockage.

Mots clés :

Fromage Quarg, *L. acidophilus*, ultrafiltration, rétentat acidifié de lactosérum en poudre, rétentat acidifié de lait en poudre.

Titre abrégé :

Fromage Quarg en poudre.

INTRODUCTION

Acid curd cheese obtained from milk retentate as described by Maubois and others (Maubois *et al.*, 1969; Maubois and Mocquot, 1971; Brulé *et al.*, 1974; Maubois and Mahaut, 1974; Brulé *et al.*, 1975; Maubois and Mocquot, 1975; Covacevich and Kosikowski, 1977; Puhane and Gallmann, 1981; Gungerich, 1981) is characterised by a higher content of whey proteins and calcium, as compared to the traditional product. Higher retention of whey proteins and calcium increases the nutritional value of acid curd cheese, but sometimes acid taste (Brulé *et al.*,

1975) and gelatin-like texture (Covacevich and Kosikowski, 1978a) were detected. If before ultrafiltration the pH of milk is either slightly decreased (Brulé *et al.*, 1974; Friis, 1981; Puhán and Gallmann, 1981) or markedly lowered to 4.5-4.7 (Stenne, 1973; Mahaut *et al.*, 1982) the retention of calcium decreases and quarg texture becomes typical as in traditional technology.

Spray dried milk retentates (Maubois and Mocquot, 1975) and milk retentates acidified to pH 4.6 before drying (Ehsani *et al.*, 1982) makes it possible to prepare a long lasting semi-product from which one can prepare the fresh acid cheese, at home or in a dairy factory.

To increase dry matter and protein content in the UF milk retentates while making Mozzarella and Cheddar cheese Covacevich and Kosikowski (1978b) used freeze-dried UF retentate powder. To acidify UF retentates Ehsani *et al.* (1982) used lactic acid. For the same purpose we used over-acidified milk i.e. milk brought to pH 3.5-3.8 by *Lactobacillus acidophilus* (Korolczuk *et al.*, 1983) or acidified to pH 3.6-3.8 UF milk and whey retentates by *Lactobacillus acidophilus* (Korolczuk and Grzelak, 1984).

As over-acidification, below the isoelectric point of casein gives the opportunity to pasteurize the acidified milk of UF retentates and to spray-dry them at normal temperatures instead of keeping them at 0° C before drying. We used this method to prepare over-acidified UF milk and whey retentates powders to be used as acidifying agent in quarg type cheese technology.

MATERIALS AND METHODS

Pooled skim milk and skimmed sweet rennet whey were heat treated at 65° C for 20 minutes, then cooled to 50-53° C and ultrafiltrated to reach concentration up to 5 times for the milk and up to 10 times for the whey (Fig. 1). A hollow-fiber type UF module (3 × 0.9 m²) equipped with PM 50 membranes (Amicon, U.S.A.) was used. The conditions of ultrafiltration were as follows: inlet/outlet pressure was 2.0-1.8 bar/1.0-0.8 bar, recirculation rate was 2 m³/h, temperature 53 ± 2° C. Ultrafiltration rate at the beginning of the process was about 40-60 l/m².h and at the end about 10 l/m².h. Heat treated (65° C; 20 min.) retentates were inoculated with 5 % starter of *L. acidophilus* 1T (from the collection of the Agricultural University in Warsaw). The starter was prepared one day before, on skim milk heated at 95° C for 30 min., fermented to pH 4.5-4.3 at 43° C and stored overnight at 5° C. Acidification of retentates lasted for 18-24 hours at 43 ± 2° C. Acidified to pH 3.5-3.8 retentates were heat treated (65° C, 20 min.) and spray dried (Niro-Atomizer, Denmark) with inlet/outlet air temperatures 180-170° C/90-60° C. Spray dried, non-acidified milk retentates were prepared by the same procedure but without

Figure 1

Diagram of preparation of acidified milk and whey retentate powders: M-milk, W-whey, UF-ultrafiltration, P-permeate, MR-milk retentate, WR-whey retentate, S₁-L. acidophilus starter, AMR-acidified milk retentate, AWR-acidified whey retentate, SD-spray drying, AMRP-acidified milk retentate powder, AWRP-acidified whey retentate powder.

Figure 1

Schéma du procédé de préparation des rétentats acidifiés de lait écrémé et de lactosérum en poudre: M-lait, W-lactosérum, UF-ultrafiltration, P-perméat, MR-rétenant de lait, WR-rétenant de lactosérum, S₁-levain de *L. acidophilus*, AMR-rétenant acidifié du lait, AWR-rétenant acidifié de lactosérum, SD-séchage spray, AMRP-rétenant acidifié de lait en poudre, AWRP-rétenant acidifié de lactosérum en poudre.

acidification. The powders were sealed in multilayer, aluminium foil bags and kept at room temperature for up to 6 months before using them for the preparation of quarg type cheeses.

Reconstituted quarg samples were prepared on a laboratory scale (10 kg each) by mixing fresh heat treated skim milk (Fig. 2 a) or fermented skim milk (Fig. 2 b) with powders of milk retentate (MRP), acidified milk retentate (AMRP) or acidified whey retentate (AWRP). In the case of fresh pasteurized milk used as a solvent,

Figure 2

Diagram of preparation of regenerated quargs (Q) from milk (M), butter starter (S₂), neutral milk retentate powder (NRP) and acidified milk or whey retentate powder (ARP).

Figure 2

Schéma du procédé de préparation de fromage frais type quarg reconstitué (Q) à partir de : M-lait ; S₂-ferment à beurre, NRP-rétenant neutre de lait en poudre ; ARP-rétenant acidifié du lait ou de lactosérum en poudre.

5 % of commercial butter starter (WCK, Poland) was added to increase the number of viable lactic acid bacteria and to develop the flavour. The starter was a composition of *Str. lactis*, *Str. cremoris* and *Str. diacetylactis*. The proportions of ingredients were calculated as to obtain a protein concentration in the final product equal to or higher than 12.5 % characteristic for normal quargs and a lactic acid content of about 1.4-1.2 % equivalent to pH 4.8-4.6.

Reconstituted quarg samples, closed in glass jars (250 g each) were kept for 21 days at $6 \pm 2^\circ \text{C}$.

The pH of retentates and quargs was determined by means of a pH-meter type PHM-64 (Radiometer, Denmark). The titrable acidity was determined by using a titrator type TTT-60 (Radiometer, Denmark) and expressed in percentage of lactic acid. Nitrogen was determined by Kjeldahl method using a Kjel-Foss Automatic apparatus (A/S N. Foss Electric, Denmark). Moisture content was determined at 102°C for 5 hours and ash at 550°C (AOAC, 1975), lactose by the method of FIL-IDF (1974 a). Microbiological analysis

and sensory evaluation were carried out using standard methods (FIL-IDF, 1971a, b; 1974b; 1980; 1981a).

RESULTS AND DISCUSSION

Drying process milk retentates acidified to pH 3.6-3.8 caused some browning when outlet temperatures were about 80-90°C and the powder left in a cyclon for over half an hour. The reduction of outlet temperature to 60-65°C by introduction of room temperature air at the bottom of the drier solved this problem. This system is commonly used in the industrial scale driers, so it would cause no problems when scaling up the process.

Powders obtained from UF whey or milk retentates or from acidified retentates contained 94-96 % dry matter (Fig. 3), 65 %

Figure 3

Chemical composition of: ● milk retentate powder, ■ acidified milk retentate powder, ○ whey retentate powder, □ acidified whey retentate powder.

Figure 3

Composition chimique des poudres de: ● rétentat de lait; ■ rétentat acidifié de lait; ○ rétentat de lactosérum; □ rétentat de lactosérum acidifié.

protein in the case of milk retentate and about 46-50 % protein in the case of whey, acidified whey and acidified milk retentates powders. Lactic acid concentration in the AMRP or AWRP was about 13-15 %. In some samples lactic acid content was over 20 %, but in this case the powder stucked too much to the dryers walls and formed a cake-like layer. Lactose concentration was of the level of 18-23 % for

MRP, AMRP and AWRP and about 33 % for neutral whey retentate powder (WRP). Ash content was about 7.6-8 % for milk and 6-6.8 % for whey retentate powders. As about 80 % of calcium is retained by casein, so the milk retentates were rich in minerals. The pH of 10 % water solutions of acidified retentates was about 3.8-4.0 which was about 0.2 pH units higher than the pH of acidified retentates before drying. This was probably caused by some losses of lactic acid during the process, and by the dilution effect, as the retentates before drying contained 15-18 % dry matter.

By mixing milk (Fig. 2 a) or fermented milk (Fig. 2 b) with neutral milk retentate powder and acidified milk or whey retentate powder, we obtained four types of quargs. They contained 23-24 % dry matter, 12-13 % protein, 1.7-2.2 % ashes and 1.1-1.4 % of titrable lactic acid (Fig. 4). The protein content was typical to traditional

Figure 4

Chemical composition of regenerated quargs obtained from: milk (M), neutral milk retentate powder (NRP) and acidified milk retentate powder (AMRP) ●; fermented milk (FM), NRP and AMRP ■; M, NRP and AWRP ○; FM, NRP and AWRP □.

Figure 4

Composition chimique des fromages frais type quarg (reconstitué) obtenu à partir de : ● lait (M), poudre de rétentat de lait (NRP) et poudre de rétentat acidifié de lait (AMRP); ■ lait fermenté (FM), NRP et AMRP; ○ M, NRP et AWRP; □ FM, NRP et AWRP.

quargs, but the dry matter was about 6-7 % higher because of higher lactose and minerals content. It is possible to reduce dry matter to a level of about 17.5 % characteristic for traditional quarg but diafiltration has to be used in this case to decrease lactose content in milk and whey protein retentates. But as practically it does not make sense, we renounced this idea for the moment.

As indicated in fig. 2 and 3 about 83-89 % of the final product total mass was coming from liquid milk or fermented milk. But,

Figure 5

Origin of basic components of regenerated quarks: M + S₂, milk + starter or fermented milk; NRP, neutral milk retentate powder; ARP, acidic milk or whey retentate powder. Samples: A = milk (M) + neutral milk retentate powder (NRP) + acidified milk retentate powder (AMRP); B = M + NRP + acidified whey retentate powder (AWRP); C = fermented milk (FM) + NRP + AMRP; D = FM + NRP + AWRP.

Figure 5

Origine des principaux composants du fromage frais type quarg (régénéré): M + S₂, lait + ferment à beurre ou lait fermenté; NRP, rétentat de lait en poudre; ARP, rétentat acidifié de lait ou de lactosérum en poudre. Echantillons: A = lait (M) + rétentat neutre de lait en poudre (NRP) + rétentat acidifié de lait en poudre (AMRP); B = M + NRP + rétentat acidifié de lactosérum en poudre (AWRP); C = lait fermenté (FM) + NRP + AMRP; D = FM + NRP + AWRP.

if taking into account the dry matter balance (Fig. 5), 44-54 % comes from the neutral milk retentate powder and 12-24 % from acid milk or acid whey retentate powder. About 56-68 % protein comes from neutral and 12-24 % from acid retentates powders. Over 70 % of lactic acid comes from acid retentates if quarg was produced from milk and about 30 % if fermented milk was used. About 44-48 % of the minerals originate from milk retentate and another 41-44 % comes from normal milk. Sixty to 62 % of lactose originated from liquid milk or fermented milk. As over 2/3 of dry matter and over 3/4 of the protein originated from dried neutral and acid UF retentates, the quarg produced by this method could be a good solution for seasonal variations of milk supply and for reduction of transport costs. As about 95 % of milk protein are retained by UF and no

finer are lost in the whey, the proposed method combines the advantages of traditional technology and MMV method (Maubois, Mocquot and Vassal, 1969).

Whey proteins, which represent a higher nutritional value than casein, are almost absent in traditional quargs. If quarg is produced by MMV method (Maubois, Mocquot and Vassal, 1969) the whey protein content increases to about 15-20 % of total protein. When acidified whey retentate powder is used as a source of lactic acid, the level of whey protein could be about 30-40 % of total protein depending if milk or fermented milk is used as a solvent.

Organoleptic quality of quargs was good and no detectable changes were observed during a 3 weeks storage period at 6°C. In some samples obtained from acidified milk retentate we observed sometimes a sandy texture. It might have been caused by poor solubility of dried acid casein. No such phenomena was observed in the quargs prepared from acidified whey retentate powder. This could be explained by the fact, that denaturation of whey protein in acidic solutions is rather low (Harwalkar, 1980) and we did not heat whey retentate at high temperature. The texture of quargs obtained from milk and acidified whey retentate, which contained around 40 % of whey proteins, was much softer than that of pure milk quargs, but no whey expulsions were observed during storage, which indicates good water binding capacity of whey proteins.

Total bacterial count of spray dried retentates was of the order of 10^5 C.F.U./g and about 10^4 C.F.U./g for acidified retentates. Moulds number of neutral retentates was on the level of 10-100 and less than 1/g for acidified retentates (Fig. 6). The yeasts number was lower than 10^1 /g and again lower for acidified than neutral retentates powders. Coliform and Enterococci were absent in 0.1 g, though it happened to detect them in some samples of neutral whey retentates being present in 0.1 g but absent in 0.01 g. Lower contamination of acidified retentates resulted from the combined action of temperature and acidity. Acidified retentates, after fermentation contained about 10^9 lactic acid bacteria (C.F.U.)/g while after heat treatment and drying about 10^4 lactic acid bacteria (C.F.U.)/g. It confirms that at low pH even low temperature long time heat treatment is sufficient to reduce the number of living bacteria by the factor of 10^5 - 10^6 .

Regenerated quargs were characterised by the total bacterial count in the range 4×10^7 - 4×10^{10} (table 1). The higher number was observed, as could be expected in the quargs made from fermented milk. During three weeks of storage at 6°C the number of viable bacteria in some samples increased by about one log, in others it remained constant. Moulds number after one day was about 1-10/g and remained unchanged in most samples. In the samples prepared from the milk and acidified milk retentate powder we observed an increase of moulds number up to about 2×10^4 /g, but the initial number in these samples was about 10 times higher than in other

Figure 6

Microbiological quality of: ● milk retentate powder; ■ acidified milk retentate powder; ○ whey retentate powder; □ acidified whey retentate powder.

Figure 6

Qualité microbologique de poudres obtenues à partir de: ● rétentat de lait; ■ rétentat acidifié de lait; ○ rétentat de lactosérum; □ rétentat acidifié de lactosérum.

ones. The yeasts number was about 1-10/g and in some samples increased up to 10³/g while in others it remained unchanged during 3 weeks of storage at 6° C. Enterococci and Coliforms were absent in all samples during 3 weeks of storage.

The regenerated quargs obtained by the method described here, especially ones prepared by dissolving retentates powders in the fermented milk where characterised by the high level of viable bacteria (10⁹-10¹⁰ C.F.U./g) characteristic for traditional quargs, but at the same time they were not contaminated by Coliforms and Enterococci. As viable lactic acid bacteria may represent an important value from the nutritional point of view, the preparation of quargs by dissolving the powders of acidified and neutral milk and whey retentates in the fermented milk could be recommended.

In this case, the process lasts as long as the traditional one, and special precautions have to be taken to avoid reinfection. But even in this case if comparing with the traditional process the volume of fermentation vats is reduced five times, as the milk and starter volume per kg of quarg is reduced from 4.5-4.6 l to 0.8-0.9 l. If the process follows the procedure shown in fig. 2 only 5 % of butter starter is needed and the flavour rich starter is recommended. In this case however the flavour only develops the next day, and the

TABLE 1

Microbiological quality of regenerated quargs after 1, 10 and 21 days of storage at $6 \pm 2^\circ \text{C}$

Time (days)	Sample Symbol *			
	A	B	C	D
	Total bacterial count, colony forming units/g of curd at 30° C			
1	5.0×10^7	1.1×10^{10}	4.0×10^7	5.3×10^9
10	7.3×10^7	1.2×10^{10}	3.2×10^9	7.8×10^9
21	6.1×10^8	4.0×10^{10}	1.4×10^9	1.7×10^9
	Coliform			
1, 10, 21	less than 10 per gram			
	Enterococci			
1, 10, 21	less than 10 per gram			
	Moulds number per gram of curd			
1	2.0×10^1	(in samples B, C, D, during 21 days of storage)		
10	2.1×10^2			
21	1.8×10^4			
	Yeasts number per gram of curd			
1	< 1	(in samples B, C, D, during 21 days of storage)		
10	1.1×10^2			
21	7.9×10^2			

* Sample symbols: A-quarg obtained by the procedure 2 a with acidified milk retentate powder, B-quarg obtained by the procedure 2 a with acidified whey retentate powder, C-quarg obtained by the procedure 2 b with acidified milk retentate powder, D-quarg obtained by the procedure 2 b with acidified whey retentate powder.

few hours lasting fermentation at $18-25^\circ \text{C}$ before cooling improves the organoleptic quality of quarg.

The method described here gives the opportunity of creation of a large variety of fresh dairy products. We have chosen quarg as an example, but it could be any fermented milk food, as for example yoghurt type products or semi hard acid cheese type tvarog. We used milk and fermented milk as solvents of neutral and acid milk or whey permeate powders. It could be water or fruit juice as well. Special attention has to be stress on using over-acidified whey retentate powder as an acidifying agent, as whey protein has higher nutritive value than casein, but also as it gives smoother texture than over-acidified milk retentate powder. Infant formulas of whey-

milk-fruit quargs or drinks in powder could be elaborated. We used *L. acidophilus* strain 1T which acidifies milk to pH 3.1-3.2 and milk retentate to pH 3.5.

Similar results we have obtained with few strains of *L. helveticus* and *L. bulgaricus*, finally we have chosen *L. acidophilus* 1T because it produced no flavour at all and gave the best texture. *L. bulgaricus* for example gave yoghurt like flavour, which is not acceptable in the case of quarg type products. Two strains of *L. helveticus* we have tried, gave glue like texture, not acceptable for quarg, but eventually acceptable for yoghurt type products.

If taking into account export products, the mixture of spray dried powders of neutral and acid milk or whey retentates together with spray dried or lyophilised flavour rich starter, eventually enriched with fruit flavours, could be a good composition for a "semi-product" to be regenerated in water or milk at home or in a dairy factory.

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