ELSEVIER

0308-8146(95)00219-7

*Food Chemistry*, Vol. 56, No. 4, pp. 429–432, 1996

0308-8146/96 $15.00 + .00

# Lactulose, monosaccharides and undenatured serum protein contents in commercial UHT creams and their usefulness for thermal treatment assessment

**N. Corzo,[a]* M. Villamiel[a] & I. Martínez-Castro[b]**

[a]*Instituto de Fermentaciones Industriales, CSIC, Juan de la Cierva 3, E-28006 Madrid, Spain*
[b]*Instituto de Química Orgánica General, CSIC, Juan de la Cierva 3, E-28006 Madrid, Spain*

(Received 7 June 1995; accepted 8 October 1995)

Fifteen UHT cream samples with different fat levels (ranging from 10% to 35%) were analysed for free carbohydrates (lactose, lactulose, glucose, galactose and myoinositol) and undenaturated whey proteins using gas chromatography and high-performance liquid chromatography, respectively. Lactulose concentrations ranged from 7 to 177 mg per 100 ml, α-lactalbumin from 5.1 to 61.6 mg per 100 ml and β-lactoglobulin from 1.0 to 64.0 mg per 100 ml. These values were similar to those reported for UHT milks. The ratio β-lactoglobulin/lactulose was found to be significantly higher than that reported for UHT milk. Copyright © 1996 Elsevier Science Ltd

## INTRODUCTION

Some chemical parameters are used as indicators of reactions that occur during heat treatment of milk. Lactulose concentration and undenatured β-lactoglobulin content have recently been proposed by both the International Dairy Federation (IDF, 1991) and the European Union (EEC, 1992) as analytical indicators to distinguish UHT milk from in-container sterilized milk. A milk sample has to have less than 600 mg litre$^{-1}$ lactulose and more than 50 mg litre$^{-1}$ undenatured β-lactoglobulin to be considered as UHT.

These parameters could be useful to characterize the thermal treatment undergone by other dairy products such as creams; however, it does not establish any criterion. Therefore, the IDF (1995) has proposed, as new work, the standardization of methods for distinguishing heat-treated creams. Data about lactulose and undenatured whey protein content of creams are very scarce (Gallagan, 1991; Pellegrino, 1994) and they are not sufficient for this purpose; also, they could be affected by the fat content.

The effect of fat on the formation of lactulose and the denaturation of whey protein is not yet clear. When lactulose formation has been compared with the fat content of milk, some contradictory results have appeared in the literature. The analysis of commercial samples and the plant experiments of Geier & Klostermeyer (1983) did not show any influence of the fat content (in the range 0–3.5%) on the formation of lactulose. Similar results were described by Andrews (1984). De Koning *et al.* (1990) found more lactulose formation and whey protein denaturation in 3% fat milk than in 1.5% fat milk, while Pellegrino (1994) observed that lactulose formation and protein denaturation were lower in whole milk than in skimmed milk.

The aim of this work has been to obtain data about the content of free carbohydrates and undenaturated serum protein in commercial UHT creams with different fat levels. The values have been compared with those found in UHT milks in order to evaluate the usefulness of these parameters as thermal treatment indicators in fat-rich dairy products.

## MATERIALS AND METHODS

### Samples

Fifteen UHT commercial cream samples with different fat contents from several brands were purchased in local markets.

*To whom correspondence should be addressed.

### Analysis

Free carbohydrates were determined by gas chromatography as their trimethylsilyl ethers, following previously described methods. Disaccharides (lactose and lactulose) were resolved on an OV-17 micropacked column, with phenyl-$\beta$-glucoside as internal standard (Olano *et al.*, 1986) and monosaccharides (galactose, glucose and *myo*-inositol) on an OV-1 capillary column, using methyl-$\alpha$-galactoside as internal standard (Troyano *et al.*, 1994).

Undenatured whey protein determination was performed by reversed-phase high-performance liquid chromatography using a PLRP-S 8 $\mu$m column, (300 Å, 150 mm×4.6 mm i.d.) (Polymer Laboratories, Church Stretton, UK) with a linear binary mobile phase gradient (Resmini *et al.*, 1989). Calibrations were performed by the external standard method. Standard curves of whey proteins $\alpha$-lactalbumin and $\beta$-lactoglobulin (Sigma Chemical Co., St. Louis, USA) were linear at the same concentration range and chromatographic conditions at which samples were run.

Total protein content was determined by the Kjeldahl method (IDF, 1986).

## RESULTS AND DISCUSSION

Figure 1 shows the disaccharides found in the commercial UHT creams with different fat contents. Lactulose and lactose were clearly higher in low-fat creams (samples 1–4) than in high-fat creams (samples 5–15), except for samples 5 and 13 which contained 4010 and 3709 mg per 100 ml lactose, respectively. The variation of lactose was as expected in these type of products (Bassette & Acosta, 1988). Figure 2 shows the galactose, glucose and *myo*-inositol levels found in these samples.

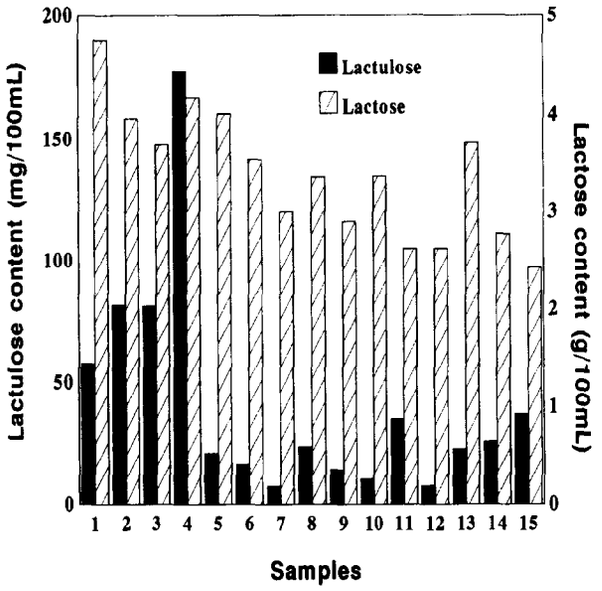


**Fig. 1.** Lactulose (mg per 100 ml) and lactose (g 100 ml) content in UHT commercial creams with different fat levels.

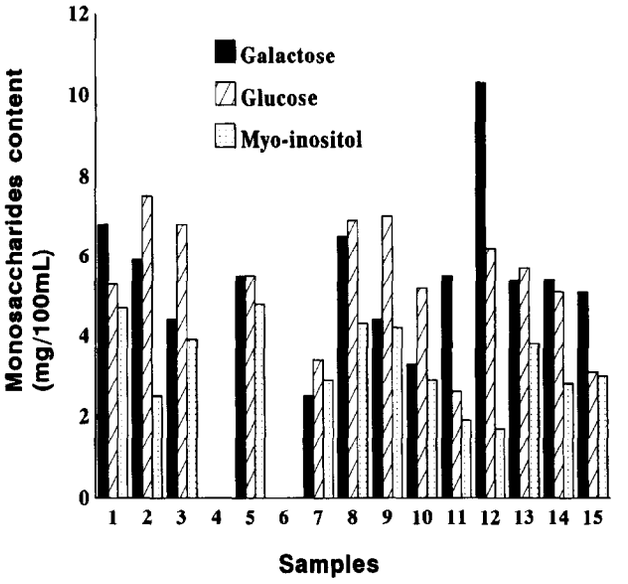


**Fig. 2.** Free monosaccharide content (mg per 100 ml) in UHT commercial creams with different fat levels.

These data are the first reported in creams; however, they are similar to those previously found in UHT milks, their variation ranges being broader (Troyano, 1993).

Figure 3 presents the undenatured $\alpha$-lactalbumin and $\beta$-lactoglobulin contents of creams. In most samples, the level was considerably high. Only two samples had values lower than the minimum proposed by IDF for UHT milks (5 mg per 100 ml).

It is necessary to correct the above values taking into account the fat content of creams. The corresponding values for lactulose and undenatured $\beta$-lactoglobulin, calculated on the skimmed product, appear in Table 1. Samples 1–4 had more than 60 mg per 100 ml lactulose

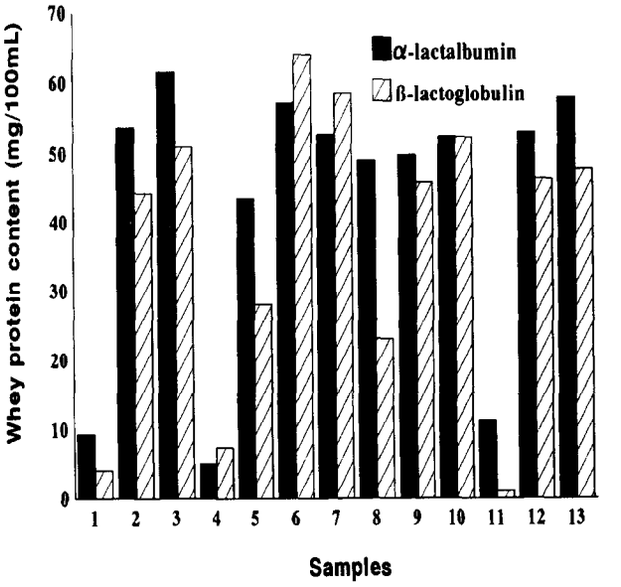


**Fig. 3.** Undenatured $\alpha$-lactalbumin and $\beta$-lactoglobulin content (mg per 100 ml) in UHT commercial creams with different fat levels.

**Table 1. Lactulose and β-lactoglobulin in creams (mg per 100 ml; calculated on the skimmed product) and β-lactoglobulin/lactulose ratio**

| Sample no. | Fat (%) | Lactulose | β-Lactoglobulin | β-Lactoglobulin/lactulose ratio |
|---|---|---|---|---|
| 1 | 10 | 63.9 | 4.6 | 0.07 |
| 2 | 18 | 99.5 | 53.8 | 0.54 |
| 3 | 18 | 99.0 | 62.3 | 0.63 |
| 4 | 22 | 227.2 | 9.5 | 0.04 |
| 5 | 35 | 32.1 | 43.0 | 1.34 |
| 6 | 35 | 24.8 | 98.4 | 3.97 |
| 7 | 35 | 11.2 | 90.0 | 8.03 |
| 8 | 35 | 36.3 | 35.5 | 0.98 |
| 9 | 35 | 21.2 | 70.3 | 3.32 |
| 10 | 35 | 16.0 | 80.3 | 5.02 |
| 11 | 35 | 53.7 | 1.5 | 0.03 |
| 12 | 35 | 11.7 | 71.1 | 6.08 |
| 13 | 35 | 34.9 | 73.2 | 2.10 |

and should be classified as overprocessed according to the limit proposed by the IDF for UHT milk; but at the moment this limit can only be applied to UHT milk. It is possible that these samples had been submitted to an indirect UHT process, whereas the others, having 35% fat, had almost certainly been treated in a direct steam injection plant because of their high viscosity.

Only sample 11 had less than 5 mg per 100 ml undenaturated β-lactoglobulin and could also be considered to be overheated. On the other hand, some samples (6, 7 and 10) showed very high values of undenaturated β-lactoglobulin. They were higher than previously reported in UHT milks (Andreini *et al.*, 1990; López-Fandiño *et al.*, 1993; Pellegrino, 1994). Nevertheless, the total protein content of creams was as expected according to their fat contents (Bassette & Acosta, 1988): 2.9% in sample 1 (10% fat), 2.75% in samples 2 and 3 (18% fat), 2.19% in sample 4 (22% fat), and averaging 1.88% in samples 5–15 (35% fat).

The values of β-lactoglobulin and lactulose should be expressed on a skimmed basis when products with different fat content are to be compared. Thus, the ratio β-lactoglobulin/lactulose has two advantages: it combines the two parameters and it is independent of the fat content of the sample. This ratio averaged $2.47 \pm 2.51$ for the analysed creams. These figures were compared with those obtained from 31 whole and 13 skimmed commercial samples of UHT milk analysed in our laboratory (López-Fandiño *et al.*, 1993), which averaged $1.00 \pm 0.98$ and $1.09 \pm 1.19$, respectively. It was found that the ratio was significantly different in creams ($P < 0.05$). Creams having a ratio less than 0.1 could correspond to overheated samples.

At the moment we have no explanation for this difference, because it is difficult to attribute it to either the fat content or to the type of UHT process. As the β-lactoglobulin/lactulose ratio could be of interest as a heating indicator, it would be necessary to do a more detailed study in order to explain the high figures found in some cream samples.

## ACKNOWLEDGEMENTS

The authors acknowledge the financial support given by the Comision Interministerial de Ciencia y Tecnología (Project ALI 95-0046-CO2-O2). We also thank M. J. Rodriguez for technical assistance.

## REFERENCES

Andreini, R., Chiodi, J., De Noni, Y., Resmini, P., Battelli, G., Cecchi, L., Todesco, R., Cattaneo, T. M. P., Rampilli, M. & Foschino, R. (1990). *Sci. Tecnol. Lattiero-Casearia*, **41**, 472–492.

Andrews, G. R. (1984). Distinguishing pasteurized, UHT and sterilized milks by their lactulose content. *J. Soc. Dairy Technol.*, **37**, 92–95.

Bassette, R. & Acosta, J. S. (1988). Composition of milk products. In *Fundamentals of Dairy Chemistry*, ed. N. P. Wong. Van Nostrand Reinhold, New York, pp. 39–79.

De Koning, P. J., Badings, H. T., van der Pol, J. G., Kaper, J. & Vos-Klompmaker, E. A. J. (1990). Effect of heat treatment and fat content on the properties of UHT milk. *Voedingsmiddelentechnologie*, **23**, 11–14.

EEC (1992). Doc. VI/5726, Rev. 2.

Gallagan, L. (1991). Measurement of lactulose formed in dairy products exposed to high heat treatments. 86th ADSA annual meeting. *J. Dairy Sci.*, **74** (Suppl. 1), 91.

Geier, H. & Klostermeyer, H. (1983). Formation of lactulose during heat treatment of milk. *Milchwissenschaft*, **38**, 475–477.

International Dairy Federation (1986). Standard No. 20A. Milk total nitrogen content (Kjeldhal method).

International Dairy Federation (1991). B-Doc. 198.

International Dairy Federation (1995). E-Doc. 610.

López-Fandiño, R., Corzo, N., Villamiel, M., Delgado, T., Olano, A. & Ramos, M. (1993). Assessment of quality of commercial UHT milks by chromatographic and electrophoretic methods. *J. Food Protect.*, **56**, 263–264.

Olano, A., Calvo, M. & Reglero, G. (1986). Analysis of free carbohydrates in milk using micropacked columns. *Chromatographia*, **21**, 538–540.

Pellegrino, L. (1994). Influence of fat content on some heat-induced changes in milk and cream. *Neth. Milk Dairy J.*, **48**, 71–80.

Resmini, P., Pellegrino, L., Hogenboom, J. A. & Andreini, R. (1989). Thermal denaturation of whey protein in pasteurised

milk. Fast evaluation by HPLC. *Ital. J. Food Sci.*, **3**, 51–62.
Troyano, E. (1993). Monosaccharides and organic acids resulting from chemical degradation of lactose during processing of milk and milk products. Ph. D. thesis, Universidad Complutense of Madrid, Spain.
Troyano, E., Olano, A. & Martínez-Castro, I. (1994). Changes of free monosaccharide fraction during storage of dried milk. *J. Agric. Food Chem.*, **42**, 1543–1545.