Available online at www.sciencedirect.com
SCIENCE DIRECT®
Food Chemistry 97 (2006) 103–108
Food Chemistry
www.elsevier.com/locate/foodchem

# Evolution of free mono- and di-saccharide content of milk-based formula powder during storage

Jorge L. Chávez-Servín, Meritxell Romeu-Nadal, Ana I. Castellote, M. Carmen López-Sabater *

*Dpt. Nutrició i Bromatologia, Centre de Referència en Tecnologia dels Aliments (CeRTA), Facultat de Farmàcia, Universitat de Barcelona. Avda. Joan XXIII s/n E-08028 Barcelona, Spain*

Received 4 January 2005; received in revised form 16 March 2005; accepted 16 March 2005

**Abstract**

HPLC-RI was used to monitor saccharide evolution during the shelf-life of two types of milk-based formulae powders supplemented with microencapsulated long-chain polyunsaturated fatty acids (LC-PUFA): infant formula and formula for pregnant and/or lactating women. In addition, these powders were subjected to sensory evaluation. The powders were stored at 25, 37 and 47 °C. Lactose was detected in the infant formula (63 g/100 g), while in the formula for pregnant women, the following were found: lactose (25 g/100 g), fructose (15.8 g/100 g), sucrose (9.28 g/100 g) and lactulose (0.9 g/100 g). Microencapsulated LC-PUFA supplementation of milk-based formula powders did not affect sugar changes.


*Keywords:* Milk-based formula powders; Saccharides; Storage; Sensory evaluation

## 1. Introduction

The food industry has made several attempts to improve the quality and the overall nutrient contents of formula-based milk, and to develop special products for specific stages of life (namely intrauterine, newborn, pregnancy and lactation). Two of these are the infant formulae (IF) and formula for pregnant and/or lactating women (FPW). Several studies on infant nutrition have addressed the effects of long-chain polyunsaturated fatty acids (LC-PUFA) on development and growth (Giovannini, Riva, & Agostoni, 1995; Lauritzen, Hansen, Jorgensen, & Michaelsen, 2001) and also their effects on pregnant and lactating women, the fetus and neonate (Hornstra, Al, van Houwelingen, & Foreman van Drongelen, 1995; Gibson, Neumann, & Makrides, 1996; Jensen, Maude, Anderson, & Heird, 2000; Hornstra, 2000; Makrides & Gibson, 2000; Al, van Houwelingen, & Hornstra, 2000; Koletzko et al., 2001).

Some IFs and FPWs include LC-PUFAs. Moreover, the addition of docosahexaenoic acid (DHA, C22:6 n-3) to IF results in improved neurofunctional responses in pre-term infants (Heird, Prager, & Anderson, 1997; Carlson & Neuringer, 1999; Crawford, 2000; Cunnane, Francescutti, Brenna, & Crawford, 2000; Jeffrey, Weisinger, Neuringer, & Mitchell, 2001).

Heating causes most of the chemical changes that occur during the manufacture of milk-based formulae. During heating, lactose undergoes the Lobry de Bruyn-Alberda van Ekenstein rearrangement, thereby initially producing isomeric disaccharides, mainly lactulose. Given that this compound does not occur naturally in milk, but is formed in heated dairy products, it is a good indicator of heat-induced damage during the manufacture of these products (Andrews, 1986; Olano et al.,

* Corresponding author. Tel.: +34 93 4024512; fax: +34 93 4035931.
*E-mail address:* mclopez@ub.edu (M.C. López-Sabater).

0308-8146/$ - see front matter © 2005 Elsevier Ltd. All rights reserved.
doi:10.1016/j.foodchem.2005.03.027

1992; Friedman, 1996; Lopez-Fandino & Olano, 1999; Belloque, Villamiel, Lopez-Fandino, & Olano, 2001; Kockel, Allen, Hennigs, & Langrish, 2002). Formula milk powders are frequently stored for long periods before being consumed, and therefore changes in their composition may occur. The Maillard Reaction is generally slow at ambient temperatures in liquid systems, but in dried food it is fast. (Troyano, Olano, & Martinez-Castro, 1994).

Milk-based formulae vary in nutrient composition, depending on the type (IFs or FPW) and the manufacturer. Consequently, the speed of the Maillard Reaction and lactose isomerization may differ (Pereyra Gonzales, Naranjo, Malec, & Vigo, 2003).

The FPW and IF used in this study were supplemented with microencapsulated fish oil (MFO), a dry free-flowing powder that consisted of spherical particles made of marine oil, distributed in a food starch matrix, which is a source of DHA. This supplementation could affect the stability of formulae milk. To prevent oxidation and to increase the stability and durability of LC-PUFA, mainly DHA, MFO is coated with caseinate and sucrose. It is stabilized with ascorbyl palmitate, sodium ascorbate and tocopherols as antioxidants. The evolution of milk-based formulae during shelf-life should be monitored through analytical and sensorial methods. The evaluation of organoleptic properties is one of the most useful ways to study quality (Baker, 1983; Valero, Villamiel, Sanz, & Martinez-Castro, 2000). A sensorial analysis is useful for assessing the stability of the product and for determining differences in sensorial characteristics during storage (Valero, Villamiel, Miralles, Sanz, & Martinez-Castro, 2001).

No studies have addressed the evolution of lactose in milk-based powdered formulae when other sugars are added (namely fructose and sucrose), or whether these supplements affect the quality and sensory perception of the products during shelf-life.

Here we studied the evolution of mono-and di-saccharides in two kinds of milk-based formulae supplemented with microencapsulated fish oil. The results of evolution were then compared with those from a sensory analysis.

## 2. Materials and methods

### 2.1. Samples

The first formula was supplemented with MFO (SIF), containing 0.5 g/100 g of total fatty acids as DHA (C22:6 n-3) while the second, control infant formula (CIF), was not. These two formulae were packed in 400 g unbroken, laminated, foil bags and sealed under nitrogen. According to the label, the two formulae contained 58% of carbohydrates (lactose), 26% of fat and 12% of protein (casein/serum proteins, 40/60). Their main ingredients were skimmed milk powder, reduced minerals whey, lactose, minerals and vitamins.

In addition, an experimental FPW supplemented with MFO containing 20 g/100 g of total fatty acids as DHA, was analyzed. According to the label, this formula contained 53.7% of carbohydrates, 20.2% of fat and 18.1% of protein (casein/serum proteins, 80/20). The main ingredients of this formula were whole milk powder, animal fat, fructose, minerals, vitamins and artificial aroma. It was packed in a 15 g unbroken aluminium foil bag flushed with nitrogen. All formulae were obtained from a production plant immediately after manufacture.

### 2.2. Storage

For this study, the formulae were kept in a storage chamber equipped with a heater thermostat and were maintained at three temperatures (25, 37 and 47 °C) from production until 12 months of storage. Storage at 47 °C was for only 120 days after production.

### 2.3. Sensory analysis

External judges were previously selected on the basis of their performance in a Duo-Trio test, using IFs heated for 5 days at 47 °C and comparison with fresh IF. Fourteen judges were chosen and were trained for sensorial tests. In accordance with the manufacturer's instructions, the FPW was reconstituted with cold water (10–15 °C, 15 g in 200 ml) and the IF with warm water (37 °C, 15 g in 100 ml). The samples for both formulae were presented in white plastic cups (30 ml each).

Duo-trio and paired comparison tests were applied. The former, which used two samples (recently produced milk-based formula vs. stored formula), was conducted to determine whether storage affected sensorial quality. The paired comparison test was done to determine whether particular sensorial characteristics (better taste, better smell, and longer-lasting flavour) differed between the two samples. In this study, we accepted the option "no difference". For the statistical analysis we divided these scores evenly between the two samples (Meilgaard, Civille, & Carr, 1987). For each storage period analysis, each judge did three test on separate days. We conducted 42 sensory tests for each storage period analyzed (only at 25 °C) at 0, 5 and 9 months in FPW and 0, 6 and 9 months for SIF.

### 2.4. Analytical determinations

#### 2.4.1. pH

The pH of the reconstituted samples was measured in a pH meter micro-pH 2000 with a glass electrode, (Crison Instruments, SA, Barcelona, Spain).

*2.4.2. Instrument*

The chromatographic analyses were carried out in a Shimadzu high-performance liquid chromatograph equipped with a LC-10AD double pump, a 7725 Rheodyne injector (Cotati, CA, USA) with a 20 μl loop, and a RID-6A Shimadzu refractive index detector. Chromatographic separation was performed in a Tracer carbohydrates column (5 μm particle size; 250 × 4.6 mm i.d.), and an $NH_2$ precolumn (13 mm × 3 mm i.d.), both from Tracer (Teknokroma, Barcelona, Spain).

*2.4.3. Free mono-and di-saccharide analyses*

HPLC-RI was used to determine free mono-and disaccharide contents (Chávez-Servín, Castellote-Bargallo, & Lopez-Sabater, 2004) as follows. The formulae were dissolved with a warm methanol–water mixture; Carrez-I and Carrez-II solutions and acetonitrile were then added. After precipitation of protein, the resulting solution was passed through filter paper and through a C18 Sep-Pak Plus cartridge from Waters (Milford, MA, USA). Finally, it was forced through a 0.45 μm nylon filter from Tracer (Barcelona, Spain). An aliquot was injected into the HPLC system. HPLC-RI analyses were performed in quadruplicate.

*2.5. Statistical analysis*

For the Duo-trio and the paired comparison tests, we used the $\chi^2$-test and the paired *t*-test, respectively. For both sensorial tests, the two-sided hypotheses for each storage time against fresh formula were applied and a significance of $\alpha = 0.01$ (1%) (Meilgaard et al., 1987) was established. For the statistical analysis of mono- and di-saccharides, we used a one-way analysis of variance (ANOVA) for each sugar and formula, in order to detect differences in the formulae during the storage. Statistical analyses were performed using the SPSS package for Windows version 11 (SPSS, Chicago, IL, USA).

## 3. Results and discussion

*3.1. pH analysis*

The pH values of the formula samples were measured upon storage because this can favour either the sugar isomerization (Lobry De Bruyn-Alberda van Ekenstein transformation) or the formation of Amadori compounds (Pellegrino, Denoni, & Resmini, 1995).

In the CIF and SIF, no changes in pH values were detected from 0 to 12 months of storage (25 and 37 °C). The average pH of CIF and SIF (from 0 to 12 months) was 7.02 ± 0.17 and 6.93 ± 0.04, respectively. These values are common for IFs (Ferrer, Alegria, Farre, Abellan, & Romero, 2002; McSweeney, Mulvihill, & O'Callaghan, 2004).

The standard deviation of pH values in FPW, between storage at 25 and 37 °C, showed no variation. The pH averages were 7.02 ± 0.12 and 7.12 ± 0.18, respectively.

*3.2. Sensory analysis*

*3.2.1. Duo-trio test*

This test was chosen because is simple, and has the advantage that a reference sample is presented to panellists. No significant differences were detected in FPW at 0 vs. 5 or 0 vs. 9 months of storage at 25 °C (Table 1).

The panel did not distinguish between fresh SIF and that stored for 6 months at 25 °C. In addition, in 73.83% of the tests, the panel members distinguished between 0 and 9 months of storage. However, this did not represent a significant difference ($p > 0.01$) (Table 1).

*3.2.2. Paired-comparison test*

The paired comparison test was used to determine the way in which the taste, smell and flavour of SIF and FPW changed with storage time, compared with recently manufacturated products. This test is one of the simplest and most used sensory tests (Meilgaard et al., 1987). No significant differences were detected in "better taste", "better smell" or "longer lasting-flavour" between FPW at 0 months of storage and that stored for 5 and 9 months (Table 2). In addition, the sensory characteristics of the SIF were similar when comparing 0 vs. 5 and 0 vs. 9 months of storage at 25 °C (Table 2).

For both types of milk-based formulae, the characteristic "better smell" registered the highest values of "indifferent", indicating that the multiple chemical changes that occurred during storage are not reflected in odour compounds detectable by sensory analysis, at least until 9 months of storage (Table 2).

*3.2.3. Mono- and di-saccharides*

Table 3 shows the mono- and di-saccharide initial contents in the milk-based formulae. The intensity of the heat treatment in the production of IF powder was estimated by determining the amount of lactulose formed. This compound is a good indicator of heat damage in milk products. The International Dairy Federation (IDF) and the European Community (EC) Commission (Fox, 1997) proposed 600 mg/l of lactulose as a marker for distinguishing between UHT milk and from 600 to 1400 mg/l for sterilized milk; reconstituted formula for pregnant women falls within the latter. However, no limit has been established for lactulose content in IFs or milk-based formulae. No lactulose was detected in the CIF or SIF. Because of the mild thermal conditions in milk drying, lactulose formation is not significant (De Block et al., 2003). Moreover, no lactulose was formed during storage conditions at 25, 37 or 47 °C. This result indicates that the formation of

Table 1
Duo-trio test in formula for pregnant women (FPW) and in supplemented infant formula (SIF) stored at 25 °C

| | Formula for pregnant women | | Infant formula | |
|---|---|---|---|---|
| | FPW 0 months vs. FPW 5 months | FPW 0 months vs. FPW 9 months | SIF 0 months vs. SIF 6 months | SIF 0 months vs. SIF 9 months |
| Detected difference (%) | 54.76 | 70.12 | 61.9 | 73.83 |
| No detected difference (%) | 45.23 | 29.88 | 38.1 | 26.19 |

The sensory assessment was made by 14 judges three times on different days. No significant difference was detected ($p > 0.01$).

Table 2
Paired comparison test (preference %) in formula for pregnant women (FPW) and supplemented infant formula (SIF) stored at 25 °C

| Characteristics | FPW 0 months vs. FPW 5 months | | | FPW 0 months vs. FPW 9 months | | |
|---|---|---|---|---|---|---|
| | 0 months | 5 months | Indifferent | 0 months | 9 months | Indifferent |
| Better flavour | 26.19 | 57.14 | 16.67 | 14.29 | 65.71 | 20 |
| Better smell | 23.81 | 23.81 | 52.38 | 23.81 | 33.33 | 42.86 |
| Longer-lasting flavour | 21.43 | 42.86 | 35.71 | 30.95 | 50.00 | 19.05 |
| | SIF 0 months vs. SIF 7 months | | | SIF 0 months vs. SIF 9 months | | |
| | 0 months | 5 months | Indifferent | 0 months | 9 months | Indifferent |
| Better flavour | 23.81 | 47.62 | 28.57 | 21.43 | 60.71 | 17.86 |
| Better smell | 21.43 | 21.43 | 57.14 | 10.71 | 32.14 | 57.14 |
| Longer-lasting flavour | 23.81 | 35.71 | 40.48 | 28.57 | 21.43 | 50 |

The sensory assessment was made by 14 judges three times on different days. No significant difference was detected ($p > 0.01$).

Table 3
Initial contents of mono and disaccharides in milk-based formulae and in the raw matter with microencapsulated DHA

| Carbohydrate content (g/100 g powder) | | | | | | |
|---|---|---|---|---|---|---|
| Sample | Lactose | Lactulose | Fructose | Sucrose | Glucose | Galactose |
| CIF[c] | 65.0 ± 0.73 | –[a] | – | – | – | – |
| SIF[d] | 63.4 ± 0.92 | – | – | tr[b] | – | – |
| FPW[e] | 25.1 ± 0.74 | 0.9 ± 0.03 | 15.8 ± 0.07 | 9.28 ± 0.23 | – | – |
| MFO[f] A | – | – | – | 28.8 ± 0.11 | – | – |
| MFO[f] B | – | – | – | 28.8 ± 0.12 | – | – |

Values are expressed as means ± standard deviation of four determinations.
[a] = not detected.
[b] tr = traces below quantification limit.
[c] CIF, control infant formula.
[d] SIF, supplemented infant formula.
[e] FPW, formula for pregnant women.
[f] MFO raw matter.

this compound occurs only at elevated temperatures during heating, and not during storage. Nevertheless, in the case of FPW, an initial lactulose content of 0.9 g/100 g of powder was detected, which remained unchanged during the first 5 months of storage. After this time, the amount decreased to 0.39 g/100 g after 7 months and, after 9 months of storage at 25 °C lactulose was not detected (Fig. 1). Lactulose is much more difficult to reduce than lactose, (Hu, Kurth, Hsieh, & Krochta, 1996).

Neither glucose nor galactose was detected in formulae after production or during storage, which is consistent with other findings (Ferreira, Gomes, & Ferreira, 1998). In the IFs, lactose was the only sugar present; however, the SIF showed slight traces of sucrose, which were below quantification limits <0.20 mg/ml (Chávez-Servín et al., 2004).

The decrease in lactose concentration during the first period of storage in the IF and FPW is attributed to the combination of lactose with the ε-amino group of lysine, which leads to the formation of lactulosyl-lysine, (which remains bound to the protein), a key precursor in the formation of coloured products (Olano, Calvo, & Corzo, 1989; De Block et al., 2003). FPW stored at 25 °C showed a decrease in lactose from 25.1 g/100 g to 18.81 g/100 g, after 5 months of storage. In addition, after this time, no alterations in lactose concentrations were observed until 12 months (Fig. 1).

The concentration of lactose in CIF and SIF remained unaltered when these were stored at 25 and

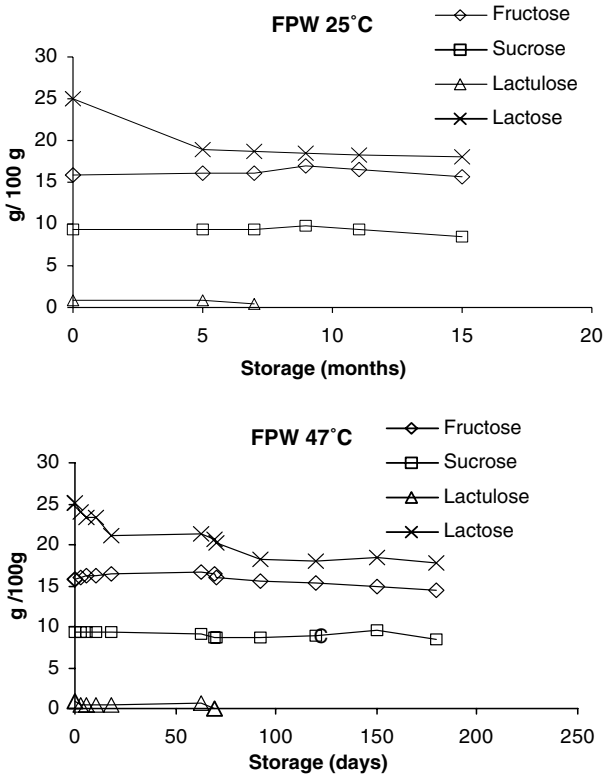


Fig. 1. Evolution of saccharides in formulae for pregnant and/or lactating women (FPW) stored at 25 and 47 °C.

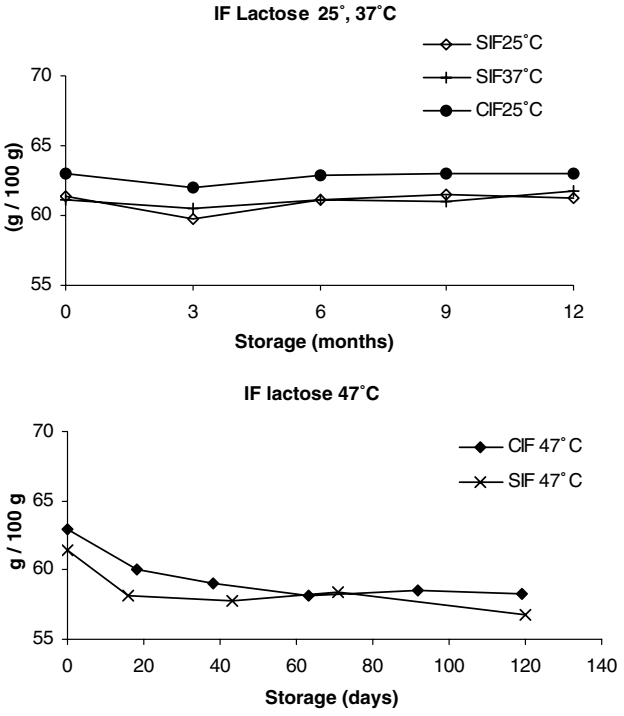


Fig. 2. Evolution of lactose in the infant formulae stored at 25, 37 and 47 °C. SIF, supplemented infant formula; CIF, control infant formula.

37 °C. However, at 47 °C, the concentration of this sugar decreased by about 4.6% at 18 days, 6% at 28 days and 7% from 63 until 120 days, compared with the initial content (Fig. 2).

No differences between the lactose evolution in CIF and SIF were observed, indicating that the addition of MFO did not affect the evolution of this sugar upon storage.

Sucrose forms part of the coated MFO. To observe the evolution of sucrose in this raw matter, the MFO was also stored at 47 °C. No changes were observed in the concentration of this sugar in two batches, (production separated by 1 year) after 120 days of storage (Fig. 3).

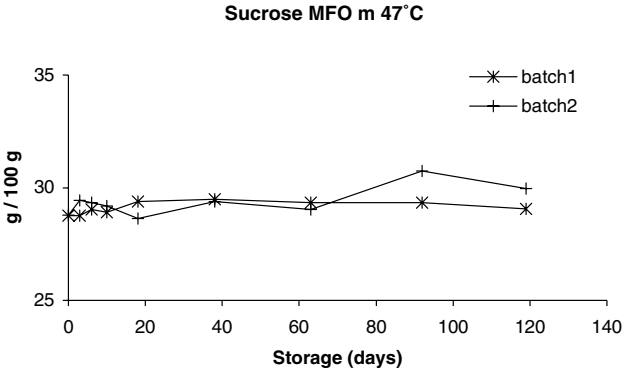


Fig. 3. Evolution of sucrose in the MFO raw matter in two batches stored at 47 °C.

Finally, fructose, which is listed in the contents label of FPW, is used as a sweetener. The initial content of this sugar after production in FPW was 15.82 g/100 g. The concentration of fructose did not show significant changes during storage at 25 °C or 47 °C. This observation is consistent with the results obtained from the sensory analysis.

### *3.2.4. Conclusions*

The sugar evolution of IF and FPW was studied; not significant differences in sugar contents were found when products were stored at 25 or 37 °C.

On the basis of our findings, the addition of MFO to the milk-based formulas did not affect sugar evolution. Hardly any changes in sensorial quality were observed. The use of this MFO in the milk industry has considerable potential; however, more studies on the stability of supplemented products with this material are necessary, including more batches and further sensory analyses.

## Acknowledgments

The authors are grateful to Laboratorios Ordesa S.L. (Sant Boi de Llobregat, Barcelona, Spain) for providing the samples, and Robin Rycroft for correcting the English. Special thanks are given to CONACYT (Mexico) for their grant to J.L. C-S.

## References


Al, M. D. M., van Houwelingen, A. C., & Hornstra, G. (2000). Long-chain polyunsaturated fatty acids, pregnancy, and pregnancy outcome. *The American Journal of Clinical Nutrition, 71*(1), 285S–291S.

Andrews, G. R. (1986). Formation and occurrence of lactulose in heated milk. *Journal of Dairy Research, 53*(4), 665–680.

Baker, S. K. (1983). The keeping quality of refrigerated pasteurized milk. *Australian Journal of Dairy Technology, 38*(3), 124–127.

Belloque, J., Villamiel, M., Lopez-Fandino, R., & Olano, A. (2001). Release of galactose and *N*-acetylglucosamine during the storage of UHT milk. *Food Chemistry, 72*(4), 407–412.

Carlson, S. E., & Neuringer, M. (1999). Polyunsaturated fatty acid status and neurodevelopment: A summary and critical analysis of the literature. *Lipids, 34*(2), 171–178.

Chávez-Servín, J. L., Castellote-Bargallo, A. I., & Lopez-Sabater, M. C. (2004). Analysis of mono-and disaccharides in milk-based formulae by high-performance liquid chromatography with refractive index detection. *Journal of Chromatography A, 1043*(2), 211–215.

Crawford, M. (2000). Placental delivery of arachidonic and docosahexaenoic acids: Implications for the lipid nutrition of preterm infants. *The American Journal Of Clinical Nutrition, 71*(1), 275S–284S.

Cunnane, S. C., Francescutti, V., Brenna, J. T., & Crawford, M. A. (2000). Breasts-fed infants achieve a higher rate of brain and whole body docosahexaenoate accumulation than formula-fed infants not consuming dietary docosahexaenoate. *Lipids, 35*(1), 105–111.

De Block, J., Merchiers, M., Mortier, L., Braekman, A., Ooghe, W., & Van Renterghem, R. (2003). Monitoring nutritional quality of milk powders: Capillary electrophoresis of the whey protein fraction compared with other methods. *International Dairy Journal, 13*(2–3), 87–94.

Ferreira, I. M. P. L., Gomes, A. M. P., & Ferreira, M. A. (1998). Determination of sugars, and some other compounds in infant formulae, follow-up milks and human milk by HPLC-UV/RI. *Carbohydrate Polymers, 37*(3), 225–229.

Ferrer, E., Alegria, A., Farre, R., Abellan, P., & Romero, F. (2002). High-performance liquid chromatographic determination of furfural compounds in infant formulas. Changes during heat treatment and storage. *Journal of Chromatography A, 947*(1), 85–95.

Fox, P.F., (1997). Heat treatments and alternative methods : IDF Special Issue 9602. Published by the International Dairy Federation, Brussels. 1995, 464 pp. ISBN: 92 9098 023 6. Price: 4000 Belgian Francs. International Dairy Journal 7(4) 283.

Friedman, M. (1996). Food browning and its prevention: An overview. *Journal of Agricultural and Food Chemistry, 44*(3), 631–653.

Gibson, R. A., Neumann, M. A., & Makrides, M. (1996). Effect of dietary docosahexaenoic acid on brain composition and neural function in term infants. *Lipids, 31*, S177–S181.

Giovannini, M., Riva, E., & Agostoni, C. (1995). Fatty-acids in pediatric nutrition. *Pediatric Clinics of North America, 42*(4), 861–877.

Heird, W. C., Prager, T. C., & Anderson, R. E. (1997). Docosahexaenoic acid and the development and function of the infant retina. *Current Opinion in Lipidology, 8*(1), 12–16.

Hornstra, G. (2000). Essential fatty acids in mothers and their neonates. *The American Journal Of Clinical Nutrition, 71*(5), 1262S–1269S.

Hornstra, G., Al, M. D. M., van Houwelingen, A. C., & Foreman van Drongelen, M. M. H. P. (1995). Essential fatty-acids in pregnancy and early human-development. *European Journal of Obstetrics Gynecology and Reproductive Biology, 61*(1), 57–62.

Hu, M., Kurth, M. J., Hsieh, Y. L., & Krochta, J. M. (1996). HPLC and NMR study of the reduction of sweet whey permeate. *Journal of Agricultural and Food Chemistry, 44*(12), 3757–3762.

Jeffrey, B. G., Weisinger, H. S., Neuringer, M., & Mitchell, D. C. (2001). The role of docosahexaenoic acid in retinal function. *Lipids, 36*(9), 859–871.

Jensen, C. L., Maude, M., Anderson, R. E., & Heird, W. C. (2000). Effect of docosahexaenoic acid supplementation of lactating women on the fatty acid composition of breast milk lipids and maternal and infant plasma phospholipids. *The American Journal of Clinical Nutrition, 71*(1), 292S–299S.

Kockel, T. K., Allen, S., Hennigs, C., & Langrish, T. A. G. (2002). An experimental study of the equilibrium for skim milk powder at elevated temperatures. *Journal of Food Engineering, 51*(4), 291–297.

Koletzko, B., Agostoni, C., Carlson, S. E., Clandinin, T., Hornstra, G., Neuringer, M., et al. (2001). Long chain polyunsaturated fatty acids and perinatal development (LC-PUFA). *Acta Paediatrica, 90*(4), 460–464.

Lauritzen, L., Hansen, H. S., Jorgensen, M. H., & Michaelsen, K. F. (2001). The essentiality of long chain n-3 fatty acids in relation to development and function of the brain and retina. *Progress in Lipid Research, 40*(1–2), 1–94.

Lopez-Fandino, R., & Olano, A. (1999). Selected indicators of the quality of thermal processed milk. *Food Science and Technology International, 5*(2), 121–137.

Makrides, M., & Gibson, R. A. (2000). Long-chain polyunsaturated fatty acid requirements during pregnancy and lactation. *The American Journal of Clinical Nutrition, 71*(1), 307S–311S.

McSweeney, S. L., Mulvihill, D. M., & O'Callaghan, D. M. (2004). The influence of pH on the heat-induced aggregation of model milk protein ingredient systems and model infant formula emulsions stabilized by milk protein ingredients. *Food Hydrocolloids, 18*(1), 109–125.

Meilgaard, M., Civille, G. V., & Carr, B. T. (1987). *Sensory evaluation techniques* (Vol. II). Detroit, MI, USA: CRC Press Inc.

Olano, A., Calvo, M. M., & Corzo, N. (1989). Changes in the carbohydrate fraction of milk during heating processes. *Food Chemistry, 31*(4), 259–265.

Olano, A., Santa-Maria, G., Corzo, N., Calvo, M. M., Martinez-Castro, I., & Jimeno, M. L. (1992). Determination of free carbohydrates and Amadori compounds formed at the early stages of non-enzymic browning. *Food Chemistry, 43*(5), 351–358.

Pellegrino, L., Denoni, I., & Resmini, P. (1995). Coupling of lactulose and furosine indexes for quality evaluation of sterilized milk. *International Dairy Journal, 5*(7), 647–659.

Pereyra Gonzales, A. S., Naranjo, G. B., Malec, L. S., & Vigo, M. S. (2003). Available lysine, protein digestibility and lactulose in commercial infant formulas. *International Dairy Journal, 13*(2–3), 95–99.

Troyano, E., Olano, A., & Martinez-Castro, I. (1994). Changes in free monosaccharides during storage of dried milk. *Journal of Agricultural and Food Chemistry, 42*(7), 1543–1545.

Valero, E., Villamiel, M., Miralles, B., Sanz, J., & Martinez-Castro, I. (2001). Changes in flavour and volatile components during storage of whole and skimmed UHT milk. *Food Chemistry, 72*(1), 51–58.

Valero, E., Villamiel, M., Sanz, J., & Martinez-Castro, I. (2000). Chemical and sensorial changes in milk pasteurised by microwave and conventional systems during cold storage. *Food Chemistry, 70*(1), 77–81.