

## Changes in sugar profile during infant cereal manufacture

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### Abstract

A sugar profile study of wheat-based, rice-based and oats infant cereals was conducted to determine changes produced during processing. Two extraction procedures were assayed. The fructose, glucose, sucrose, maltose, maltotriose, isomaltotriose and raffinose contents of the cereals were analysed by gas liquid chromatography. In untreated flours, sucrose was the main sugar; glucose, fructose, maltose and raffinose were also detected. During hydrolysis, there were increases in the maltose (2.1–7.6 g/100 g, depending on the sample), glucose and fructose contents and maltotriose and isomaltotriose were also increased. Roller-drying reduced the maltose content. Four commercial samples with rice or wheat were analysed: the sugar composition was 20 g/100 g sucrose and 3 g/100 g maltose. Other ingredients in the formulations were also analysed. © 2001 Elsevier Science Ltd. All rights reserved.

*Keywords:* Sugars; Infant cereals

### 1. Introduction

Breast-feeding is considered to provide the ideal feeding for infants because of the nutritional and immunological benefits it provides and the protection it affords against microorganism-associated diarrhoea (Megraud et al., 1990). However, urbanisation and time constraints (Uwaegbute & Nnanyelugo, 1987) are associated with the early termination of breast-feeding and infants require complementary feeding from around the age of 4–6 months (Waterlow, 1981). Infant cereals are an important energy source for the nutrition of infants in Mediterranean countries and form the basis of their weaning-feeding from the age of 3–4 months.

Infant cereals must be processed to improve their dispersibility in liquids and their digestibility, because the pancreas of a 3–4-month-old baby has a limited ability to digest starch (Delachaume-Salem & Sarles, 1970; De Vizia, Ciccimarra, De Cicco, & Auricchio, 1975; Hardon, Zoppi, Shmerling, Prader, McIntyre, & Anderson, 1968). The processing also increases the acceptability of the product. Several simple “household technologies” have traditionally been used for the

processing of cereals, tubers and legumes in tropical climates. These include toasting, boiling, germination and fermentation (Alnwick, Moses, & Schmidt, 1988). Historically, the Spanish infant population was given foods based on cereals, processed in the home by the toasting and boiling of cereal flours. Nowadays, infant cereals are processed by dietetic product manufacturers in large-scale factories. These products are submitted to toasting and boiling and to a hydrolysis and drying process to improve their sensory qualities, digestibility, safety and shelf-life (Gil, Morales, & Valverde, 1991, 1994).

Toasting is traditionally applied to flours used in infant foods to improve their sensory qualities (Nout, 1993), reduce their water content and produce changes in proteins and starch structures to establish better conditions for hydrolysis. Boiling the flours in water also prepares them for the hydrolysis process but has little effect on the sensory qualities. Both processes add flavour produced by browning reactions. Moreover, the heat treatments drastically reduce the microbiological activity, thus improving the safety of the product (Nout, 1993). Hydrolysis, commonly enzymatic ( $\alpha$ -amylase) hydrolysis, improves the dispersibility in liquids and the starch digestibility, increases the sweetness and reduces the syneresis effect. It has a major impact on the sugar profile. The roller-drying process reduces the water

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content to safe levels, provides the product with a great fluidity and improves the sensory qualities.

Commercial infant cereals are mostly composed of cereal flours with and without gluten and legume flours, such as soy, which have increased protein quantity and quality (Oyeleke, Morton, & Bender, 1985) through their lysine content, a limiting amino acid in most staples. Other ingredients used include sucrose, glucose or fructose syrup, honey, powdered fruit, powdered milk, biscuits, minerals, vitamins and flavours.

Very limited information is available on the composition and processing of infant cereals. Precisely because these foods are designed for infants, it is important to know the composition of the commercial products and the changes produced during the processing of the flours. The objective of this work was to study the sugar content of the materials used in the formulation of infant cereals, the changes in the sugar profile that occur during their processing, and the sugar content of commercial products.

## 2. Materials and methods

### 2.1. Samples

Infant cereal samples were obtained from a Spanish dietetic products company (Gil et al., 1991, 1994). The composition of the samples used in this study is shown in Table 1. Single and mixed cereal flours (A) were analysed when untreated (before being processed), when toasted, when the toasted material was hydrolysed with  $\alpha$ -amylase (Gil et al., 1991, 1994) and when the hydrolysed

material was roller-dried. These samples were grouped according to whether they were based on wheat, rice or oat cereals. Mixed cereal flours with fruits (B) were only analysed after the roller-drying. Any additional ingredients were also analysed, including biscuits, banana powder, orange powder and honey.

Solid samples were stored at  $-50^{\circ}\text{C}$  until their analysis. Hydrolysed samples were freeze-dried and then stored at  $-50^{\circ}\text{C}$  until their analysis.

### 2.2. Preparation of samples

Two extraction sugar procedures were tried: procedure A (AOAC method No. 982.14) and procedure B (AOAC method No. 939.03; AOAC, 1990). The procedure A was chosen and applied, with slight modifications, to extraction of sugars in samples. Two grammes of sample were weighed into a 50 ml centrifuge tube to which 20 ml of ethanol–water (1:1) solution was then added. The tube was sealed with a rubber bung through which a narrow 30 cm long glass tube was passed, to prevent loss by evaporation. The centrifuge tube was placed in a water bath, shaken gently for 30 min at  $80$ – $90^{\circ}\text{C}$ , and then cooled and centrifuged at 5000 rpm for 10 min. This procedure was repeated. The supernatants were then filtered off and the solution was diluted to a total volume of 40 ml with ethanol–water (1:1) solution. Next, 10 or 20 ml of this solution (according to sugar concentration expected in sample) was evaporated to dryness under reduced pressure at room temperature.

### 2.3. Carbohydrate derivatisation

The carbohydrate analysis was based on the preparation of the oxime trimethylsilyl sugar (TMS) ethers, followed by gas chromatographic separation. The sample was dissolved in 5 ml of 5% hydroxylamine hydrochloride (Panreac, Barcelona) dissolved in pyridine (Merck, Darmstadt); 1.6 ml of this solution and 0.5 ml of 0.75% phenyl  $\beta$ -D-glucopyranoside in pyridine (Sigma, St Louis, MO) were then transferred into a glass tube containing 2 g of anhydrous magnesium sulphate (Panreac, Barcelona), which was shaken vigorously for 2 min and maintained horizontal for 8 h. The tube was then placed upright, 1 ml of solution was removed and 1 ml of hexamethyldisilazane (Sigma, St Louis, MO) and 0.1 ml of trifluoroacetic acid (Merck, Hohenbrunn) were added. The tube was shaken and placed in a water bath at  $60^{\circ}\text{C}$  for 1 h; 1.5  $\mu\text{l}$  of the upper layer was then taken for the chromatograph injection.

### 2.4. Gas chromatography

The gas-chromatography was performed on a Perkin–Elmer 3B gas-chromatograph with flame ionisation

Table 1  
Composition of different samples used for sugars determination

Samples	Type of cereal	Ingredients
Single flours	Wheat	
	Oat	
	Rice	
Mixture of flours (A)	<i>Seven cereals:</i> wheat, rice, barley, rye, oat, corn and millet	Soy
	<i>Eight cereals:</i> wheat, rice, barley, rye, oat, corn, millet and sorghum	Soy and honey
	Rice, corn	
	Rice, corn	Soy
Mixture of flours (B)	<i>Seven cereals:</i> wheat, rice, barley, rye, oat, corn and millet	Soy, biscuits, orange and banana powders
	<i>Seven cereals:</i> wheat, rice, barley, rye, oat, corn and millet	Soy, orange and banana powders
	Rice, corn	Soy, orange and banana powders
	Rice, corn	Soy, orange and banana powders

detector (FID; Norwalk, CT) and Perkin–Elmer data station model Sigma 15 (Norwalk, CT). The fused silica capillary column (C-382 25QC2/SGL-1; 25 m×0.25 mm i.d., 0.25 µm film thickness; Sugelabor, Spain) was coated with 100% dimethyl polysiloxane. The temperatures of the injector and detector were 290 and 300°C, respectively. The initial temperature was 180°C, followed by a heating rate of 3°C/min to 280°C. The final temperature was maintained for 23 min. The flow-rate of the carrier gas (nitrogen) was 1.5 ml/min. The gas-chromatograph system had a split ratio 1:20. The identification was made according to the retention time relative to the internal standard (phenyl β-D-glucopyranoside) and gas liquid chromatography/mass spectrometry. The gas chromatograph was a Hewlett-Packard model 5980 coupled to a Hewlett-Packard 5988 mass spectrometer. The internal standard method was applied for the calibration. The sugar/phenyl β-D-glucopyranoside area and the nanograms of sugar were considered as the variables to obtain the linear regression equation. The range of sugars assayed was from 15 to 8000 ng (0.01–25 g/100 g of sugars in sample). The correlation coefficients were: β-D-(–)-fructose ( $r=0.999$ ), α-D-(+)-glucose ( $r=0.998$ ), sucrose ( $r=0.998$ ), maltose ( $r=0.998$ ), raffinose ( $r=0.997$ ), isomaltotriose ( $r=0.997$ ) and maltotriose ( $r=0.997$ ). The sugars were obtained from Sigma (St. Louis, MO). All the extracts were duplicated and injected twice.

### 3. Results and discussion

#### 3.1. Sugar extraction procedures

The extraction of sugars was assayed following the AOAC methods Nos. 982.14 and 939.03, called procedures A and B, respectively, with slight modifications. Both procedures were assayed on a commercial sample of wheat infant cereal and involved three extractions, with continual shaking in the case of procedure A. Procedure A extracted a greater proportion of sugars than

did procedure B, although the difference was only 0.4% (Table 2). The percentages of sugars extractable were 85 and 68% with one extraction and 99 and 95% with two extractions, for procedures A and B, respectively. With a third extraction, only 0.3 g/100 g (procedure A) and 1.37 g/100 g (procedure B) were obtained for the main sugar (sucrose). As a result of this exercise, procedure A was selected to determine the sugar profiles in the present study.

The wide linear detection range of the method for the different sugars (15–8000 ng) allowed the same extraction and determination procedure to be used for every study sample. The sugars determined in the different samples were: glucose, fructose, sucrose, maltose, isomaltose, maltotriose, isomaltotriose, raffinose and palatinose.

The precision study was performed using seven samples of roller-dried wheat sample (Table 3). The mean values of the coefficient of variation were 5.5% for monosaccharides (fructose, glucose), 4% for disaccharides (sucrose, maltose) and 14.3% for trisaccharides (maltotriose).

The detection limits in sample were: monosaccharides (0.005 g/100 g), disaccharides (0.025 g/100 g), raffinose (0.03 g/100 g) and maltotriose (0.05 g/100 g), determined as a signal-to-noise ratio > 2.

#### 3.2. Wheat-based cereals processing

The flours of the infant cereals included in this group were over 50% wheat flour. The commercial names were “wheat”, “7 cereals” and “8 cereals and honey”. The sugars identified in these samples were: fructose, glucose, sucrose, maltose, maltotriose and raffinose (Table 4).

The percentages of sugars in the untreated wheat-based flours were: “wheat”, 0.5 g/100 g; “7 cereals”, 1.1 g/100 g; and “8 cereals”, 1.2 g/100 g. The main sugar was sucrose, which represented 50–75% of the sugars determined (Table 4). Maltotriose was not detected in the untreated flours. The values of glucose, fructose,

Table 2  
Comparison of sugar extraction procedures for wheat infant cereals (g/100 g)

	Procedure A <sup>a</sup>			Procedure B <sup>b</sup>		
	1st Ext.	2nd Ext.	3rd Ext.	1st Ext.	2nd Ext.	3rd Ext.
Fructose	0.08	0.02	nd <sup>c</sup>	nd	nd	nd
Glucose	0.09	nd	nd	0.02	nd	nd
Sucrose	20.7	3.50	0.30	17.0	6.80	1.37
Maltose	2.70	0.35	nd	1.82	0.54	0.02
Maltotriose	0.70	nd	nd	0.32	0.24	nd
Total	24.3	3.87	0.30	19.2	7.48	1.39

<sup>a</sup> AOAC method no. 982.14.

<sup>b</sup> AOAC method no. 939.03.

<sup>c</sup> nd = Not detected.

maltose, sucrose and raffinose in untreated samples were in the ranges reported by Koch, Gedes, and Smith (1951) and Vaisey and Unrau (1964) in various wheat varieties. The study of sugars after the toasting process showed no changes in sugar profile. Browning indicators (HMF and furosine) show low levels in these materials at this stage (Fernández-Artigas, Guerra-Hernández, & García-Villanova 1999a; Guerra-Hernández, Corzo, & García-Villanova, 1999). Moreover, the small sugar differences could not be determined, because of the variability of the method and the high concentration of sugars in the samples.

The hydrolysis had a major impact on the sugar profile. Fructose, glucose, sucrose and maltose all increased after the hydrolysis step, and maltotriose was detected for the first time. Glucose and maltose contents come from starch hydrolysis. The small increase in the fructose may have derived from sucrose, while the high increase in sucrose came from its addition as an ingredient at the hydrolysis stage. Maltose was the main sugar after the starch hydrolysis (at 3.3–6.2 g/100 g, depending on the sample). The “8 cereals” sample, which included soy and honey, had the highest content of fructose and glucose, due to the addition of the honey, and the lowest content of sucrose (3.7 g/100 g) with respect to the other wheat-based samples (7 g/100 g).

Table 3  
Precision of sugar determination method in wheat infant cereal sample (g/100 g)

Statistic	Fructose	Glucose	Sucrose	Maltose	Maltotriose
Range	0.20–0.23	0.09–0.10	6.26–7.08	5.18–5.86	0.34–0.51
Mean <sup>a</sup>	0.22	0.10	6.76	5.50	0.42
S.D.	0.010	0.005	0.280	0.210	0.060
CV (%)	5.6	5.3	4.1	3.8	14.3

<sup>a</sup> Mean of seven determinations.

Table 4  
Sugar compositions (g/100 g)<sup>a</sup> of wheat-based infant cereals at manufacturing steps

	Fructose	Glucose	Sucrose	Maltose	Maltotriose	Raffinose
<i>Wheat</i>						
Untreated	0.04	0.03	0.25	0.12	nd <sup>c</sup>	0.05
Hydrolysed	0.11	0.27	6.98 <sup>b</sup>	6.16	0.34	0.05
Roller-Dried	0.09	0.23	6.51	5.47	0.37	0.04
<i>7 cereals-soy</i>						
Untreated	0.08	0.09	0.81	0.08	nd	0.04
Hydrolysed	0.02	0.12	6.58 <sup>b</sup>	3.27	nd	0.04
Roller Dried	0.07	0.36	6.39	2.98	0.10	0.04
<i>8-cereals-soy-honey</i>						
Untreated	0.05	0.03	0.93	0.12	nd	0.08
Hydrolysed	1.00	1.31	3.68 <sup>b</sup>	5.64	0.18	0.04
Roller-Dried	0.94	1.03	3.38	5.11	0.25	0.04

<sup>a</sup> Expressed as dry matter.

<sup>b</sup> Added.

<sup>c</sup> nd = Not detected.

The roller-drying is carried out at a high temperature and over a short period of time, achieving a rapid decrease in the water content of the hydrolysed material. The process produces optimum water activity for browning reactions (Labuza, 1980) and a subsequent decrease in sugars (Table 4). Maltose is the main reducing sugar and the heat treatment reduces the maltose content by around 10%. Glucose and fructose decreased in “wheat” and “8 cereals”, whereas they increased in “7 cereals”. The sucrose (non-reducing sugar) decreased between 3 and 8%, which could be due either to caramelisation or hydrolysis. The increase in glucose and fructose could be due to the hydrolysis of the sucrose. The behaviours of the trisaccharides (maltotriose and raffinose) were different and the maltotriose increased whilst the raffinose remained stable. The decrease in reducing sugars in the roller-dried samples coincides with the browning of infant cereal samples measured by furosine and available lysine (Fernández-Artigas, García-Villanova, & Guerra-Hernández, 1999b; Guerra-Hernández et al., 1999).

### 3.3. Rice-based infant cereals processing

The samples included in this group were “rice”, “rice-corn” and “rice-corn-soy”. Rice was the main cereal and constituted more than 50% of the flour used. The rice-based cereals are also known as gluten-free or gliadin-free cereals recommended to prevent celiac sprue. The rice-corn-soy infant cereal has a higher protein content and is recommended for low-weight babies. The sugars determined in these samples were: fructose, glucose, sucrose, maltose, maltotriose, isomaltotriose and raffinose (Table 5).

The sugars in the untreated rice sample were fructose, glucose sucrose and raffinose, as previously reported (Fukui & Nikuni, 1959). The total sugar content in

rice-based cereals was lower than in wheat-based cereals (Table 5). The milled rice sample contained 0.24 g/100 g total sugars, with 0.11 g/100 g reducing sugars. Other researchers have reported 0.25–0.53 g/100 g total sugars in milled rice, with 0.05–0.08 g/100 g reducing sugars (Pascual, Singh, & Juliano, 1978). The sugar profile of the rice-based cereals was similar to that of the rice-only product (fructose, glucose, sucrose and raffinose). As in the wheat-based cereals, the sucrose in the rice-based products was mostly free-sugar and the glucose and fructose showed similar proportions, at 0.04–0.13 g/100 g. The samples of rice-corn and rice-corn-soy contained the highest value of sucrose, at 0.60 g/100 g and 0.35 g/100 g respectively. The toasting step did not change the sugar content.

The hydrolysis with  $\alpha$ -amylase produced an increase in the glucose, maltose, maltotriose and isomaltotriose (Table 5). The maltotriose content was higher than in the wheat-based cereals, perhaps due to the higher starch content of rice (70%) with respect to wheat (59%; Belitz & Grosch, 1997). Isomaltotriose was not detected in the wheat-based cereals. The glucose increased from 0.06 to 1.2 g/100 g and maltose, which was not detected in the untreated samples, was found at 5 g/100 g after the hydrolysis. The high increase in sucrose was due to its addition as ingredient. The increase in fructose after the starch hydrolysis could derive from sucrose.

The effects of the roller drying were similar to those observed in the wheat-based samples (Table 5).

### 3.4. Oats infant cereal processing

The nutritional value of oats is greater than that of other cereals, because of the high content of lipids (7%) with non-saturated fatty-acids, protein (13%) and lysine, the low content of starch, and the high content of substances with antioxidant properties. The sugars detected were fructose, glucose, sucrose, maltose and

raffinose (Table 6). The raffinose content (0.22 g/100 g) of untreated oats was higher than that of the untreated wheat-based and rice-based cereals. The other sugars showed similar results to wheat. The toasting step induced no changes in the sugar profile. The hydrolysis process produced maltose values of only 2 g/100 g, compared with the 6.2 g/100 g and 7.6 g/100 g produced in the wheat and rice, respectively. This may be partly accounted for by the lower starch content. The higher sucrose content after hydrolysis was due to its addition.

The roller-drying process produced a decrease in all sugars. At this step, browning reactions are observed by furosine, HMF and loss of available lysine (Fernández-Artigas et al., 1999a, Fernández-Artigas, García-Villanova, & Guerra-Hernández, 1999b; Guerra-Hernández et al., 1999). However, the intensity of browning is lower than in rice infant cereal (with similar proportion of lysine), probably due to the smaller content of reducing sugar.

### 3.5. Commercial infant cereals and ingredients

The commercial samples analysed were “wheat”, “rice-corn-soy with fruit”, “7 cereals with fruit”, “7 cereals with biscuits-orange-banana” (Table 7). The sugar content of the ingredients of the commercial samples is shown in Table 8. The content of reducing sugars (glucose and fructose) was 76 g/100 g in the honey and 17 g/100 g in the orange powder. The sucrose content was approximately 10 g/100 g in the biscuits, banana and orange powder. In the honey, there was a high concentration of maltose (5 g/100 g) and isomaltose and palatinose were also present. The commercial infant cereals with added ingredients had a higher sugar content, mostly glucose and fructose, than the commercial wheat sample without. The high proportion of sucrose in commercial samples is due to its addition

Table 5  
Sugar compositions (g/100 g)<sup>a</sup> of rice-based infant cereals at manufacturing steps

	Fructose	Glucose	Sucrose	Maltose	Maltotriose	Isomaltotriose	Raffinose
<i>Rice</i>							
Untreated	0.05	0.06	0.13	nd <sup>c</sup>	nd	nd	<0.03
Hydrolysed	0.21	1.47	6.92 <sup>b</sup>	7.57	0.55	0.11	<0.03
Roller-Dried	0.34	1.32	6.26	7.63	0.81	0.09	0.04
<i>Rice-corn</i>							
Untreated	0.04	0.09	0.60	nd	nd	nd	<0.03
Hydrolysed	0.17	1.05	6.80 <sup>b</sup>	3.78	0.28	0.06	0.05
Roller-Dried	0.15	1.21	5.89	3.02	0.29	0.06	0.04
<i>Rice-corn-soy</i>							
Untreated	0.01	0.03	0.35	nd	nd	nd	<0.03
Hydrolysed	0.20	1.09	5.72 <sup>b</sup>	3.21	0.23	0.04	0.05
Roller-Dried	0.09	0.57	5.67	2.63	0.35	0.06	0.05

<sup>a</sup> Expressed as dry matter.

<sup>b</sup> Added.

<sup>c</sup> nd = Not detected.

Table 6  
Sugar compositions (g/100 g)<sup>a</sup> of oat infant cereals at manufacturing steps

	Fructose	Glucose	Sucrose	Maltose	Maltotriose	Raffinose
<i>Oat</i>						
Untreated	0.02	0.06	0.56	0.14	nd <sup>c</sup>	0.22
Hydrolysed	0.33	0.58	5.94 <sup>b</sup>	2.11	nd	0.21
Roller-Dried	0.17	0.37	5.74	1.69	0.17	0.20

<sup>a</sup> Expressed as dry matter.

<sup>b</sup> Added.

<sup>c</sup> nd = Not detected.

Table 7  
Sugar analysis (g/100 g) of commercial infant cereals

	Fructose	Glucose	Sucrose	Maltose	Maltotriose	Isomaltotriose	Raffinose
Wheat	0.10	0.09	24.5	3.05	0.70	nd <sup>a</sup>	0.04
Rice-corn-soy <i>with fruits</i>	0.29	1.25	23.4	3.38	1.38	0.18	0.07
7 cereals <i>with fruits</i>	0.28	0.47	23.7	3.68	0.40	nd	0.04
7 cereals <i>with biscuit, orange and banana</i>	0.99	1.45	16.6	2.81	0.34	nd	0.04

<sup>a</sup> nd = Not detected.

Table 8  
Sugar analysis (g/100 g) of ingredients added to infant cereals

	Fructose	Glucose	Sucrose	Maltose	Isomaltose	Palatinose
Biscuit	1.10	1.39	9.65	0.34	nd	nd
Banana powder	2.37	2.13	9.52	0.03	nd	nd
Orange powder	6.84	10.3	10.9	nd <sup>a</sup>	nd	nd
Honey	41.2	35.3	0.03	4.44	0.10	0.12

<sup>a</sup> nd = Not detected.

during the hydrolysis process and after the roller-drying process. The Committee on Nutrition of the European Society of Gastroenterology and Pediatrics (ESPGAN, 1981) recommends an upper limit of 30 g/100 g sucrose for infant cereals that require milk to be ready-to-eat. The infant cereals analysed contained between 16 and 24 g/100 g sucrose. The European Community (Directive 96/5/CE; 1996) has an upper limit of 7.5 g/100 kcal for the sugar content of glucose, sucrose, fructose, glucose syrup and honey added to infant cereals without milk. The samples analysed in the present study had less than 6.2 g/100 kcal.

To summarise, the sugar extraction method selected for the study (procedure A) yields 99% of the extractable sugars after two extractions and shaking procedures. The wide range of the detection system permits the quantification of sugar concentrations from 0.01 g/100 g for fructose to 24.5 g/100 g for sucrose in cereal samples with no changes in the procedure. The sugars detected in the samples were fructose, glucose, sucrose, maltose, maltotriose, isomaltotriose and raffinose. Maltose is the sugar that shows the greatest increases after the enzymatic hydrolysis of the starch. Rice-based infant

cereals contain the highest proportions of reducing sugars. Isomaltotriose is only detected in rice-based samples. The degree of hydrolysis, defined by the maltose content, is different for each infant cereal type. The roller-drying process commonly decreases the sugar levels. Sucrose is added to infant cereals to sweeten them, but the concentrations of this sugar are well within the permitted limits.

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