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Chemical and sensorial changes in milk pasteurised by microwave and conventional systems during cold storage

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Abstract

Raw milk was submitted to a continuous-flow microwave treatment at 80 or 92°C for 15 s or in a conventional heat exchanger under the same conditions. After pasteurisation, samples were bottled in sterile containers and stored at 4.5 ± 0.5°C for up to 15 days. Samples were taken every 2 days and analysed for pH, sensory properties, volatile compounds and monosaccharides. Volatiles (including aldehydes, ketones, alcohols, esters and aromatic hydrocarbons) in freshly pasteurised samples were very similar to those found in raw milk. Evolution of all analysed parameters during storage was similar for the two series of samples; microwave heating had no adverse effects on flavour.

1. Introduction

It is generally accepted that the quality of refrigerated pasteurised milk has to be evaluated, not only for its microbiological characteristics, but also for its sensory attributes. Baker (1983) stated that one of the most useful means for milk quality characterization is the assessment of its organoleptic properties. Maruri, Christen, Melton, Penfield and Baird (1995) studied the effect of storage time and temperature on concentration of volatile compounds and sensory attributes during shelf-life of pasteurised milk. Vallejo-Córdoba and Nakai (1994) found a high correlation between off-flavour detection, by sensory evaluation, and chromatographic peak areas, obtained by dynamic headspace gas chromatography, which allowed them to predict shelf life of pasteurised milk. Microorganism growth and enzymatic activity, responsible for off-flavours during milk storage, can also produce changes in the free monosaccharides fraction. Recio, Villamiel, Martinez-Castro and Olano (1998) studied modifications of this fraction during the storage of UHT milk, but no data are available on refrigerated pasteurised milk.

The use of continuous-flow microwave treatment has been proposed for milk pasteurisation due to its potential advantages. López-Fandiño, Villamiel, Corzo and Olano (1996) designed a continuous-flow system which provided the required conditions for the proper pasteurisation of milk. Villamiel, Lopez-Fandiño, Corzo, Martinez-Castro and Olano (1996) showed that microwave treatment of milk by continuous-flow is a mild and efficient way to achieve a product with satisfactory microbial and sensory quality without causing an extensive heat damage.

The aim of this work was to study the changes in the composition of volatile compounds, monosaccharide concentrations and sensory quality during the shelf-life of microwaved milk in continuous-flow. A comparative study was carried out by using a conventional heating system under the same treatment conditions.

2. Materials and methods

2.1. Milk samples

Raw cows' milk was obtained from a local farm. Milk was held at 5°C for up to 5 h until it was processed.

2.2. Microwave and conventional treatments

Microwave heating was carried out using a MDS-2000 oven (2450 MHz, 532 W full power from CEM

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Co., Buckingham, UK), as previously described (Villamiel et al., 1996). The milk, preheated at 20°C, was pumped through the system in a Teflon™ tube (0.58 × 200 cm), by using a variable speed peristaltic pump (Millipore, Bedford, MA, USA). The process temperatures (80 ± 1°C or 92 ± 1°C) were adjusted by varying the pump flow rate. Inlet and outlet temperatures were controlled continuously by means of digital thermometers situated just outside the oven cavity. Milk leaving the oven was passed through insulated Teflon™ tubes, the dimensions of which were calculated in order to provide a holding time of 15 s at 80 or 92°C, respectively. The processed milk was then cooled immediately in a 0.5 cm × 2.0 m coil of Teflon™ tubing immersed in an ice/water bath. Samples were taken for analysis during the steady-state conditions, achieved after 10 min (López-Fandiño et al., 1996).

The conventional heating system was very similar to the microwave continuous-flow equipment but the heating section was a coil of stainless steel tubing with 0.45 cm i.d. and 246 cm length (with the same internal volume as the Teflon™ coil) and 0.93 mm of wall thickness, immersed in a temperature-controlled water bath. The temperature of the water bath was adjusted to provide the same temperature as the microwave oven under the same flow rate conditions, thus ensuring the same heating rate (López-Fandiño et al., 1996).

All heat treatment experiments were performed in duplicate.

2.3. Storage conditions of the heated milk

Heated milk was collected in 1 l sterile containers under aseptic conditions, stored at 4.5 ± 0.5°C for up to 15 days and analysed for pH, sensory properties, volatile compounds and monosaccharides every second day during storage.

2.4. Analytical determinations

All analytical determinations were carried out in duplicate.

Sample pH was measured using a pH-meter Digit 501 (Crison Instruments, S.A., Barcelona, Spain). The intensity of the heat treatment undergone by samples was estimated by determining the amount of lactulose formed. The analyses were carried out by gas chromatography of the trimethylsilyl derivatives of the free sugar, using 1-*O*-phenyl-β-D-glucoside (Sigma) as internal standard (Olano, Calvo & Reglero, 1986). Monosaccharides were determined as their trimethylsilyl derivatives by gas chromatography, using α-methyl galactoside (Sigma) as internal standard (Troyano, Olano, Fernandez Diaz, Sanz & Martinez-Castro, 1991).

A Purge&Trap Concentrator (Hewlett-Packard, Palo Alto, CA) coupled to the gas chromatograph by means of a fused silica transfer line was used for volatile analysis; a 25 ml non-fritted sparger, a trap containing a mixture of Carbosieve-SIII and Carbopack-B 60/80) as well as a liquid nitrogen cryofocusing unit were used. 2-hexanone (Analytical Standards, Polyscience Co., Niles, IL) was added as internal standard to the milk in the sparger. Helium was the carrier gas. Operating conditions were as follows: purge flow (vent), 35 ml/min; sample temperature, 45°C; purge time, 15 min; desorption, 5 min at 220°C; cryofocus temperature, −100°C; injection within 2 min from −100°C to 200°C. A blank was run after every sample.

The gas chromatograph was an HP-5890 (Hewlett-Packard, Palo Alto, CA), equipped with a quadrupole mass detector HP-5971A operating in EI mode at 70 eV. Helium was used as carrier gas with an inlet pressure of 100 kpa; a BP-20 capillary column (50 m × 0.22 mm × 0.25 μm) was kept at 40°C for 15 min, heated at 5°C/min until 180°C and then held for 15 min. MS data were acquired and processed through an HP Chemstation. Identification of volatile components was carried out from their retention times and mass spectral data from the Wiley Library (McLafferty & Stauffe, 1989) and confirmed by using standard compounds when available. Quantitative values (μg/l) were calculated from peak areas of volatile compounds and internal standard. Since the aim of these analyses was to compare samples submitted to similar treatments, differences in response factor and recovery were not taken into account.

2.5. Sensory analysis

Sensory evaluation was carried out following the triangle test procedure (ISO Standard 4120), by a sensory panel of 12 trained members. Each panellist was given two groups of three milk samples (30 ml each), distributed so that, in each group, two samples were equal and another was different, in a random order. Panellists were asked to identify the different sample.

Milk was also evaluated for overall flavour. At each session, the two samples (microwave and conventional) were presented to the panellists in random order. Acceptability of the milk was recorded on a 9-point scale ranging from extremely unacceptable (1), to extremely acceptable (9). Assessors were also asked to note and describe any off-flavour they sensed.

3. Results and discussion

Lactulose content was lower than 50 mg/l in all treated milks. According to the IDF recommendations, lactulose content should be lower than this level for pasteurised milk (Schlimme, Bucheim & Heeschen, 1993).

No important differences were observed in the pH values of all the samples treated at 80 or 92°C by

microwaves and conventionally, measured throughout the 15 days of storage; the pH began to decrease after the 15th day. In a study on the effect of pasteurisation and storage conditions on the quality of milk, no pH changes were observed when milk was stored at 3°C for 35 days (Cromie, Schmidt & Dommett, 1989).

With respect to the organoleptic assessment (Fig. 1), milks heated in the microwave oven or in the conventional system were not distinguished by the sensory panel using the triangle test procedure either after processing or during the storage period. The sensory quality was the same for microwave and conventionally treated milks; the score of samples treated at 80°C seemed to decrease slightly on the 8th day. No off-flavours were detected by the panellists. After the 15th day, all samples developed a strong off-flavour, except the sample microwave-treated at 92°C, which also showed hardly any pH change until the 17th day. Previous work showed that shelf-life of milk heated by microwaves at 80°C was longer than that of milk heated in a plate heat exchanger (Villamiel, López-Fandiño & Olano, 1996). That result could be attributed to the different treatment conditions attained in the two systems, whereas in the present work both the microwave and the conventional systems had identical heating and cooling rates.

Table 1 shows the monosaccharide contents in milk treated at 80 or 92°C for 15 s in continuous flow by microwaves and conventionally, just after processing and after 15 days storage at 4.5°C. In general, similar concentrations of galactose, glucose and *myo*-inositol were found in all samples throughout the storage period; the observed data were within the range found by Troyano, Villamiel, Olano, Sanz and Martinez-Castro (1996) for commercial pasteurised milks. The slight increase observed for *N*-acetyl-glucosamine and *N*-acetyl-galactosamine after 15 days of storage might be due to some glycosidic activity from enzymes present in milk and/or derived from microflora. Recio et al. (1998) reported an increase in *N*-acetyl-glucosamine and *N*-acetyl-galactosamine in UHT milks stored at room temperature. No data on the content of these carbohydrates during the storage of pasteurised milk have been previously reported.

Volatiles (including aldehydes, ketones, alcohols, esters and aromatic hydrocarbons) in freshly pasteurised samples were very similar to those found in raw milk. Similar compounds have been reported in pasteurised milk samples (Badings & Neeter, 1980; Imhof & Bosset, 1994; Moio, Etievant, Langlois, Dekimpe & Addeo, 1994; Urbach, 1987; Vallejo-Cordoba & Nakai, 1993). Dimethyldisulfide has been related to the intensity of thermal treatment (Bosset, Buehler-Moor, Eberhard, Gauch, Lavanchy & Sieber, 1994) and the values found here for this compound were higher in the samples heated at 92°C (0.3–0.6 μg/l) than in those heated at 80°C or in raw milk (0.1–0.2 μg/l).

No qualitative differences were found between microwave and conventionally heated samples throughout the studied period. The quantitative variation during storage was not significant for most volatile compounds (Table 2). Some compounds, such as acetaldehyde, ethanol and ethyl acetate, increased markedly only after the 15th day. Other substances also appearing in these samples were propanol, 2-hexanol and 3-methyl butanol. An increase in acetaldehyde level in pasteurised milk has been found to correlate negatively with sensory acceptability, although sensorial changes were probably due to other compounds (Greig & Manning, 1983). A marked increase in ethanol related to the growth of microorganisms was observed in pasteurised milk during storage, but its level did not correlate well with the impairment of milk flavour (Urbach & Milne, 1987).

Previous studies have shown the effectiveness of continuous-flow microwave heating for pasteurisation of milk based on microbial and enzyme inactivation (Villamiel, Lopez-Fandiño & Corzo, 1996). Present results indicate that taste and odour of microwaved milk were unaffected during treatment and subsequent storage. Since changes during the storage of microwaved milk were similar to those of milk heated in a conventional

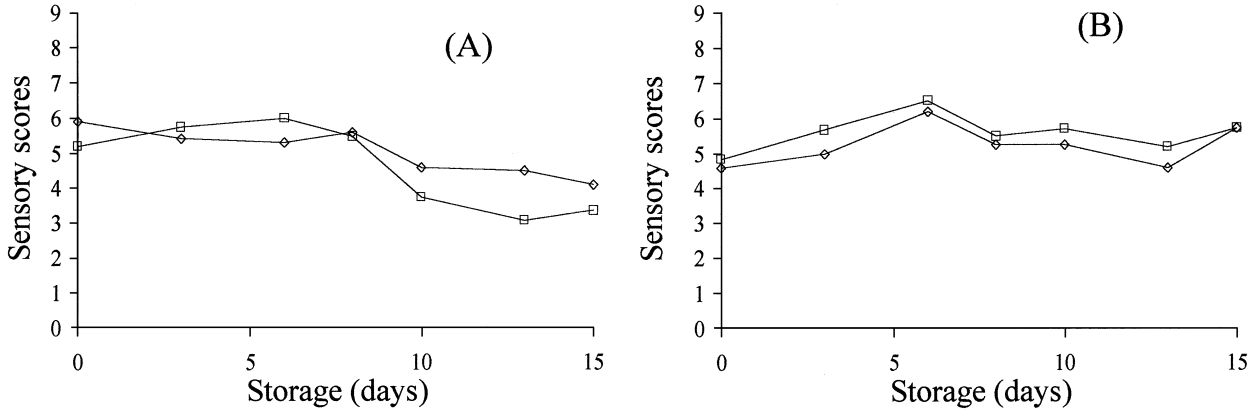

Fig. 1. Sensory evaluation for flavour of microwaved (□) and conventionally (◇) heated milk at 80°C (A) or 92°C (B) during storage at 4.5°C.

Table 1
Comparative carbohydrate content in milk processed at 80 or 92°C in a continuous microwave system (MW) or in a conventional heat exchanger (CO) and stored at 4.5 ± 0.5°C for 15 days

Carbohydrate (mg/100 ml)	Control	80°C				92°C			
		MW		CO		MW		CO	
		0 days	15 days	0 days	15 days	0 days	15 days	0 days	15 days
Galactose	6.64[a]	5.76	6.20	6.34	6.31	6.20	6.36	6.43	6.82
Glucose	7.02	6.70	7.78	7.02	7.85	6.65	6.35	7.01	8.26
N-AcGaNH$_2$	2.59	2.63	3.59	2.75	3.35	2.17	3.38	2.70	3.44
N-AcGluNH$_2$	7.19	7.10	8.21	7.42	8.86	6.95	8.96	7.36	8.32
Myo-inositol	3.25	3.17	3.76	3.36	3.56	3.18	3.70	3.24	3.66

[a] Mean values (standard deviation ⩽1.36).

Table 2
Comparative volatile content in milk processed at 80 or 92°C in a continuous microwave system (MW) and in a conventional heat exchanger (CO) and stored at 4.5 ± 0.5°C for 15 days

	Raw	MW 80		CO 80		MW 92		CO 92	
Compound		0 days	15 days	0 days	15 days	0 days	15 days	0 days	15 days
Acetaldehyde	1.43	3.08	2.80	0.296	0.800	3.08	3.34	0.320	0.404
Acetone	152	697	566	132	174	133	129	109	98.4
Tetrahydrofuran	0.253	0.373	0.349	0.364	0.347	0.783	0.322	0.373	0.349
2-Methylfuran	0.000	0.105	0.117	0.084	0.116	0.078	0.115	0.105	0.117
Butanal	0.150	0.316	0.380	0.276	0.328	0.327	0.483	0.232	0.152
Ethylacetate	1.48	1.80	0.72	1.02	2.88	2.84	1.28	1.56	1.40
Butanone	17.8	17.0	21.0	17.6	22.1	20.5	22.8	19.1	19.7
2-Methyl butanal	0.165	0.413	0.361	0.079	0.119	0.188	0.094	0.413	0.361
3-Methyl butanal	0.350	0.216	0.296	0.180	0.480	0.325	0.258	0.684	0.740
Ethanol	1.48	3.244	0.976	4.57	3.80	3.08	2.87	2.18	3.32
3-Buten-2-one	0.215	0.552	0.672	0.296	0.564	0.576	0.444	0.564	0.400
2-Pentanone	0.588	0.796	0.800	0.612	0.944	0.776	0.504	0.772	0.708
Diacetyl	0.576	0.600	0.700	0.212	0.272	0.436	0.272	0.448	0.264
4-Methyl-2-pentanone	0.084	0.120	0.080	0.104	0.088	2.04	0.136	0.088	0.152
2-Methylpropyl acetate	0.368	0.404	0.308	0.284	0.112	0.599	0.467	0.544	0.328
Toluene	4.10	25.3	25.5	5.96	8.2	5.63	7.17	3.54	3.91
2-Methyl-3-buten-2-ol	0.387	0.800	0.276	0.404	0.484	9.30	9.24	0.288	0.392
Dimethyl disulfide	0.100	0.156	1.80	0.088	0.176	0.268	0.412	0.606	0.660
Butyl acetate	0.145	0.352	0.400	0.164	0.140	0.269	0.216	0.244	0.156
C_8H_{10}	0.049	0.227	0.225	0.254	0.243	0.207	0.569	0.160	0.120
C_8H_{10}	0.121	0.161	0.486	0.084	0.140	0.072	0.166	0.080	0.080
C_8H_{10}	0.355	0.506	0.126	0.238	0.549	0.336	0.511	0.240	0.200
C_8H_{10}	0.251	0.736	0.405	0.203	0.168	0.093	0.105	0.200	0.120
2-Heptanone	0.559	0.632	0.936	0.504	0.636	0.776	0.517	0.620	0.492
3-Methyl butanol	0.092	0.116	0.160	0.076	0.124	2.70	5.20	0.100	0.112
C_9H_{12}	0.093	0.075	0.267	0.316	0.400	0.170	0.090	0.098	0.115
1-Pentanol	0.084	0.100	0.248	0.088	0.160	2.30	2.80	0.076	0.092

system under the same treatment conditions, microwave heating in continuous flow seems to be a good alternative to traditional pasteurisation methods.

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