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# Volatile components in microwave- and conventionally-heated milk

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**Abstract**

Raw milk was microwave-heated in two different modes: at 120°C using closed vessels, and at 70–90°C using a continuous flow device. Conventional heatings were carried out at the same temperatures, using a glycerol bath or a water bath instead of the microwave oven. Samples were analyzed following a dynamic headspace method; the volatile components were quantified by GC and identified by GC–MS. Volatile composition was similar using both heating systems, although when milk was heated in closed vessels some quantitative differences were found between microwave and conventional heating. The latter results were attributed to the unevenness of microwave heating inside the vessels, whereas the continuous flow device seemed to allow a very uniform heating.

## 1. Introduction

Microwave treatment is considered to be an alternative method for heating milk and milk products (Gallmann & Eberhard, 1993). Although this treatment is not common on an industrial scale, a part of consumed milk is heated in domestic microwave ovens. Several studies have recently been carried out on the effect of microwave treatment on different characteristics of milk: foodborne pathogens (Heddleson & Doores, 1994), vitamins (Aktas & Ozilgen, 1992; Medrano, Hernández, Prodanov & Vidal-Valverde, 1994), lactose and whey proteins (Villamiel, Corzo, Martínez-Castro & Olano, 1996), enzymes (López-Fandiño, Villamiel, Corzo & Olano, 1996), early Maillard products (Meissner & Erbesdobler, 1996) and cheese-making properties (Villamiel, López-Fandiño & Olano, 1997). A review covering several aspects of microwave treatment of milk in domestic ovens has been published (Sieber, Eberhard & Gallmann, 1996). From all this work it can be concluded that microwave treatment is very effective against microorganisms, and that the global composition of microwaved milk is similar to that of conventionally heated milk.

Differences in composition of volatiles between microwaved and conventionally heated foods (meat, vegetables and baked products) have been reported (Yeo & Shibamoto, 1991; King, Matthews, Rule & Field, 1995; Emam, Farag & Aziz, 1996). The effect of technological treatments on milk flavour has also been the subject of several articles (Dumont & Adda, 1979; Badings, 1984; Manning & Nursten, 1985; Calvo & de la Hoz, 1992); however, we have not found any information about changes in milk volatiles in samples submitted to microwave heating.

The aim of the present work has been to compare the composition of volatiles in milk submitted to microwave treatment and of milk thermally processed with other techniques. The volatile components of microwave-treated milks have been studied by GC–FID and GC–MS and the results compared with those obtained from conventionally heated samples using similar conditions of time and temperature.

## 2. Materials and methods

Raw milk samples were obtained from a local cow farm and kept under refrigeration until they were processed (less than 4 h). The same batch of milk was used for comparative experiments.

### 2.1. Treatments

Microwave heating was carried out using a MDS-2000 oven (2450 MHz, 532 W, full power) (CEM Corporation, Buckingham, UK), with a probe to monitor

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and control the vessels' internal temperature during the experiments. A series of treatments was performed in duplicate at high temperature (120°C): samples (50 ml) were placed in closed teflon vessels; they reached the required temperature in less than 50 s, and were kept there for 5, 10, 20 min. Comparative experiments using conventional heating were performed in duplicate by placing the samples in Pyrex closed vessels immersed in a glycerol bath. More details of both types of treatments have been published (Villamiel et al., 1996).

For treatments at low temperature, a continuous flow system was implemented in the same oven, using teflon tubing (2.00 m × 5 mm i.d). Milk was pumped through the system with a variable speed peristaltic pump (Millipore, Bedford, MA). The process temperatures (70, 80, 90°C) and the residence time (about 25 s) were adjusted by varying the flow rate of the pump, with a previous equilibration time of about 10 minutes. The conventional treatment was performed with a similar flow system, heating the milk at the same temperatures with the same residence times in a water bath instead of in the oven, and using stainless steel tubing (2.46 m × 5 mm i.d) (López-Fandiño et al., 1996). Triplicate samples were immediately cooled in an ice bath and kept under refrigeration until analysis.

### 2.2. *Volatile analysis*

Volatile components were analyzed in triplicate following a method developed in our laboratory (Valero, Miranda, Sanz & Martínez-Castro, 1997). In brief, 50 ml milk was dynamically purged with 50 ml/min nitrogen at 45°C for 180 min and the volatiles trapped on cartridges packed with 100 mg of Tenax TA (previously deactivated under $N_2$ at 300°C for 3 hours). The cartridges were thermally desorbed at 220$^{\underline{o}}$C under carrier gas flow (helium) for 15 min, using an automatic thermal desorption unit (ATD-400 from Perkin–Elmer, Norwalk, CT) connected to a Fisons 8000 gas chromatograph (Fisons Instruments, Milan, Italy) with two detectors: a flame ionisation detector (FID) used for quantitative determinations and a quadrupole mass detector MD-800 (Fisons, VG Masslab, Manchester, UK) operating in EI mode at 70 eV, used for peak qualitative characterization. MS data were acquired and processed through a MassLab data system rel.1.18 (Fisons). Two similar (A and B) capillary columns (20 m × 0.32 mm × 0.3 (m) coated with crosslinked OV-1 were used. Temperature was held at 45°C for 5 min, then programmed at 10°C/min up to 220°C and then held for 10 min. Carrier gas was helium at 56 KPa.

Tentative identification of volatile components was carried out from peak retention times (retention indices) and mass spectral data (Wiley library). Identification was confirmed by using standard compounds when available. Quantitative values (μg/l milk) were calculated from the peak areas of the components and the added internal standard (0.25 μg/l n-nonane), acquired and processed with a Chromcard data system v1.16 (Fisons). Since the aim of the work was to compare samples treated at the same temperature and for the same time, differences in response factor and recovery for these compounds were not taken into account. Blanks were run between samples.

## 3. Results and discussion

### 3.1. *Continuous flow treatments*

Total ion reconstructed (TIC) chromatograms (obtained with column A) from a milk treated at 90°C in a continuous flow system heated either in a water bath (a) or by microwaves (b) are presented in Fig. 1. The qualitative composition (listed in Table 1) was the same in both samples: hydrocarbons, methyl ketones, aldehydes, free fatty acids and esters. Traces of some terpenes were detected. Several siloxane peaks (considered as artifacts) were also found. When unambiguous identification was impossible from GC and MS characterization, only elemental composition was given. The same overall qualitative composition was obtained for samples heated at 80 and 70°C. Similar compounds have been reported in pasteurized milk samples (Badings

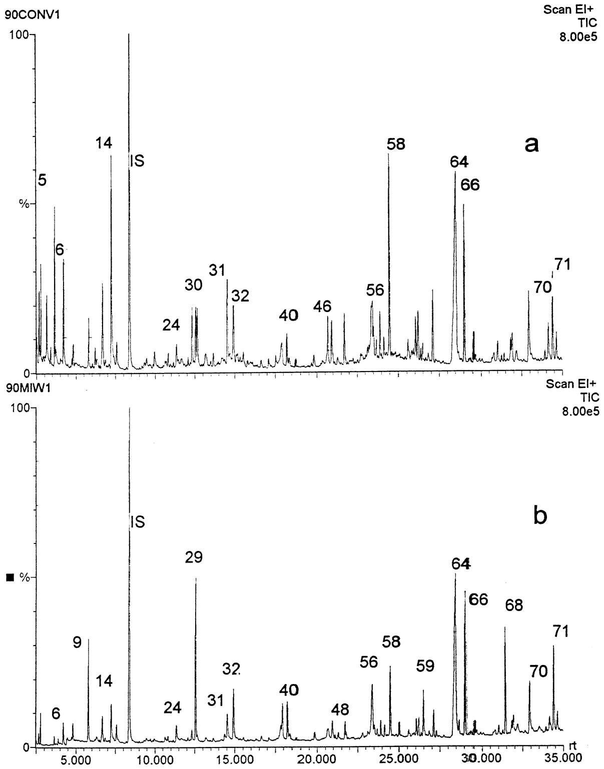

Fig. 1. Total ion reconstructed chromatograms for a milk sample heated in continuous flow at 90°C using (a) a water bath and (b) microwaves. For identification of peaks see Table 1.

Table 1
Volatile compounds identified in milks heated at 90°C in a continuous flow system

| Peak no. | $I_R$[a] | Compound | GC[b] | MS[c] | Peak no. | $I_R$[a] | Compound | GC[b] | MS[c] |
|---|---|---|---|---|---|---|---|---|---|
| 1 | | Artifact | | + | 37 | 1174 | Benzoic acid | + | + |
| 2 | 700 | Heptane | + | + | 38 | 1181 | Octanoic acid | + | + |
| 3 | 707 | Dimethyldisulfide | + | + | 39 | 1183 | Siloxane | | + |
| 4 | 710 | 2- and 3–Methyl butanol | | + | 40 | 1195 | Decanal | + | + |
| 5 | 712 | Toluene | + | + | 41 | 1197 | $C_{12}H_{26}$ | | + |
| 6 | 726 | Hexanal | + | + | 42 | 1200 | Dodecane | + | + |
| 7 | 760 | $C_8H_{16}$ | | + | 43 | 1210 | $C_{10}H_{16}O$ | | + |
| 8 | 765 | Furfural | + | + | 44 | 1237 | Terpenic alcohol | | + |
| 9 | 806 | Siloxane | | + | 45 | 1243 | Decenal | | + |
| 10 | 818 | Isobutyl acetate | | + | 46 | 1269 | Nonanoic acid | + | + |
| 11 | 837 | $C_8H_{10}$ | | + | 47 | 1270 | Bornyl acetate | + | + |
| 12 | 841 | $C_9H_{20}$ | | + | 48 | 1279 | 2-Undecanone | + | + |
| 13 | 847 | $C_8H_{10}$ | | + | 49 | 1280 | $C_{13}H_{28}$ | | + |
| 14 | 871 | 2-Heptanone | + | + | 50 | 1283 | Siloxane | | + |
| 15 | 884 | Heptanal | + | + | 51 | 1290 | Undecanal | + | + |
| 16 | 896 | Nonene | + | + | 52 | 1292 | Unknown | | |
| 17 | 900 | Nonane (internal standard) | | | 53 | 1300 | Tridecane | + | + |
| 18 | 919 | 2,4-Dimethyl-2,4,6-octatriene | | + | 54 | 1312 | Methyl decanoate | + | + |
| 19 | 924 | Siloxane | | + | 55 | 1346 | $C_{13}H_{26}$ | | + |
| 20 | 927 | Benzaldehyde | + | + | 56 | 1388 | Decanoic acid | + | + |
| 21 | 966 | Methyl styrene | | + | 57 | 1389 | Dodecanal | + | + |
| 22 | 975 | 2-Octanone | + | + | 58 | 1400 | Tetradecane | + | + |
| 23 | 981 | $C_9H_{12}$ | | + | 59 | 1479 | 2-Tridecanone | + | + |
| 24 | 987 | Octanal | + | + | 60 | 1490 | Tridecanal | + | + |
| 25 | 990 | $C_{10}H_{22}$ | | + | 61 | 1500 | Pentadecane | + | + |
| 26 | 996 | Hexanoic acid | + | + | 62 | 1509 | Methyl dodecanoate | + | + |
| 27 | 1013 | Terpinolene | + | + | 63 | 1546 | $C_{15}H_{30}$ | | + |
| 28 | 1017 | *p*-Cymene | + | + | 64 | 1559 | Dodecanoic acid | + | + |
| 29 | 1024 | Siloxane | | + | 65 | 1568 | Siloxane | | + |
| 30 | 1027 | Limonene | + | + | 66 | 1578 | Unknown | | |
| 31 | 1080 | 2-Nonanone | + | + | 67 | 1600 | Hexadecane | + | + |
| 32 | 1090 | Nonanal | + | + | 68 | 1671 | 2-Pentadecanone | + | + |
| 33 | 1100 | Undecane | + | + | 69 | 1686 | Unknown | | |
| 34 | 1122 | Methyl octanoate | + | + | 70 | 1731 | Tetradecanoic acid | + | + |
| 35 | 1148 | $C_9H_{16}O$ | | + | 71 | 1790 | Isopropyl myristate | | + |
| 36 | 1162 | Naphthalene | + | + | 72 | 1798 | Diethyl phthalate | | + |

[a] $I_R$, experimentally determinated linear retention indices.
[b] Peak identified from its retention data.
[c] Peak identified from its mass spectral data.

Table 2
Comparative volatile content (µg/l) of some selected compounds in milks processed at 70–90°C in continuous flow at different temperatures

| Compound (µg/l) | Conventional | | | Microwave | | |
|---|---|---|---|---|---|---|
| | 70°C | 80°C | 90°C | 70°C | 80°C | 90°C |
| Heptane | 0.407 | 0.852 | 0.486 | 0.736 | 0.124 | 1.236 |
| Toluene | 0.391 | 0.324 | 0.709 | 0.365 | 0.184 | 0.261 |
| Hexanal | 0.855 | 0.409 | 0.845 | 0.500 | 0.835 | 0.443 |
| 2-Heptanone | 0.212 | 0.097 | 0.112 | 0.037 | 0.045 | 0.452 |
| Heptanal | 0.129 | 0.159 | 0.620 | 0.038 | 0.196 | 0.167 |
| Octanal | 0.140 | 0.100 | 0.388 | Traces | 0.088 | 0.325 |
| 2-Nonanone | 0.017 | 0.246 | 0.334 | Traces | 0.019 | 0.379 |
| Nonanal | 0.285 | 0.639 | 0.480 | 0.388 | 0.257 | 0.280 |
| Octanoic acid | 0.056 | 0.267 | 0.199 | 0.113 | 0.216 | 0.269 |
| Decanal | 0.094 | 0.163 | 0.0191 | 0.305 | 0.142 | 0.124 |
| Decanoic acid | 0.620 | 0.671 | 0.540 | 1.189 | 0.561 | 0.401 |
| Dodecanoic acid | 1.35 | 0.915 | 0.989 | 5.484 | 1.62 | 0.620 |
| Total | 4.55 | 4.84 | 5.89 | 9.15 | 4.29 | 4.93 |

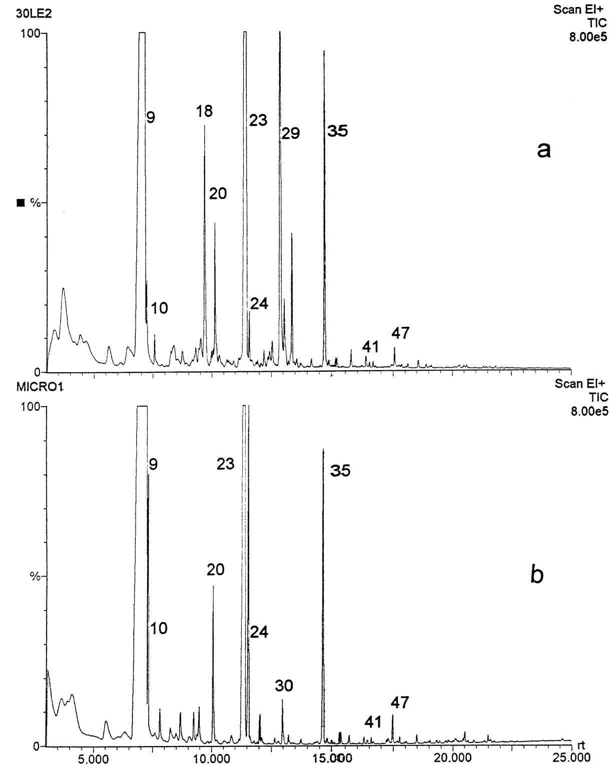

Fig. 2. Total ion reconstructed (TIC) chromatograms of volatile components from a milk sample treated at 120°C in closed vessels heated in (a) a glycerol bath or (b) in the microwave oven. For identification of peaks see Table 3.

& Neeter, 1980; Urbach, 1987; Vallejo-Córdoba & Nakai, 1993; Imhof & Bosset, 1994; Moio, Etievant, Langlois, Dekimpe & Addeo, 1994).

The quantitative estimation of 12 components, selected among those having the highests concentrations in samples, is presented in Table 2. Coefficients of variation of individual components were within 10 and 23%. The differences among treatments were slight and not significant ($p < 0.05$), probably because of the low thermal production of volatiles within the used range, and the uniformity of heating in the continuous flow system, which has been proved to give reproducible and uniform temperatures when heating is performed either with microwaves or with a water bath (Aktas & Ozilgen, 1992).

### 3.2. Heating milk in closed vessels

Total ion (TIC) chromatograms (obtained with column B) of volatile components from milk treated at 120°C in closed vessels heated either in (a) a glycerol bath or in (b) the microwave oven (b) are presented in Fig. 2. The qualitative composition (Table 3) was similar in both series. Besides methyl ketones, aldehydes, dimethyl disulfide, dimethyl trisulfide, alcohols and esters, some furan derivatives were found. As in the pasteurised samples, some terpenes and siloxanes also

Table 3
Volatile compounds identified by GC–MS in milks heated at 120°C in closed vessels

| Peak no. | $I_R$[a] | Compound | GC[b] | MS[c] |
|---|---|---|---|---|
| 1 | 680 | 2-Pentanone | + | + |
| 2 | 696 | Dimethyldisulfide | + | + |
| 3 | 710 | Toluene | + | + |
| 4 | 726 | Hexanal | + | + |
| 5 | 770 | Furfural | + | + |
| 6 | 793 | Siloxane | | + |
| 7 | 816 | $C_8H_{10}$ | | + |
| 8 | 832 | Xylene | | + |
| 9 | 879 | 2-Heptanone | + | + |
| 10 | 882 | Heptanal | + | + |
| 11 | 907 | $C_8H_{10}$ | | + |
| 12 | 929 | Benzaldehyde | + | + |
| 13 | 942 | Dimethyltrisulfide | | + |
| 14 | 950 | $C_8H_{14}O$ | | + |
| 15 | 975 | 2-Octanone | + | + |
| 16 | 986 | $C_9H_{12}$ | | + |
| 17 | 987 | Methylfuran | | + |
| 18 | 990 | Octanal | + | + |
| 19 | 1000 | Decane | | + |
| 20 | 1018 | Siloxane | | + |
| 21 | 1026 | Limonene | + | + |
| 22 | 1044 | Acetophenone | + | + |
| 23 | 1078 | 2-Nonanone | + | + |
| 24 | 1092 | Nonanal | + | + |
| 25 | 1100 | Undecane | + | + |
| 26 | 1113 | Methyl octanoate | + | + |
| 27 | 1119 | Nonanol | + | + |
| 28 | 1120 | Camphor | + | + |
| 29 | 1165 | Naphtalene | + | + |
| 30 | 1176 | Siloxane | | + |
| 31 | 1179 | 2-Decanone | + | + |
| 32 | 1191 | Decanal | + | + |
| 33 | 1200 | Dodecane | + | + |
| 34 | 1200 | Siloxane | | + |
| 35 | 1279 | 2-Undecanone | + | + |
| 36 | 1290 | Undecanal | + | + |
| 37 | 1300 | Tridecane | + | + |
| 38 | 1310 | Methyl decanoate | + | + |
| 39 | 1319 | Undecanol | + | + |
| 40 | 1349 | Siloxane | | + |
| 41 | 1375 | 2-Dodecanone | + | + |
| 42 | 1396 | Dodecanal | + | + |
| 43 | 1400 | Tetradecane | + | + |
| 44 | 1418 | Siloxane | | + |
| 45 | 1423 | Siloxane | | + |
| 46 | 1460 | Dodecanol | + | + |
| 47 | 1476 | 2-Tridecanone | + | + |
| 48 | 1492 | Tridecanal | + | + |
| 49 | 1505 | Methyl dodecanoate | + | + |
| 50 | 1524 | Siloxane | | + |
| 51 | 1552 | Siloxane | | + |
| 52 | 1578 | Unknown | | |
| 53 | 1671 | 2-Pentadecanone | + | + |
| 54 | 1791 | Isopropyl myristate | | + |

[a] $I_R$, experimentally determinated linear retention indices
[b] Peak identified from its retention data.
[c] Peak identified from its mass spectral data.

Table 4
Comparative concentration of selected compounds in milk samples heated at 120°C in closed vessels for different times

| Compound (μg/l) | Conventional (min) | | | Microwaves (min) | | |
|---|---|---|---|---|---|---|
| | 5 | 10 | 20 | 5 | 10 | 20 |
| 2-Pentanone | – | 0.736 | 0.462 | –– | 2.27 | 0.938 |
| Hexanal | 0.633 | 0.823 | 1.21 | 1.40 | 0.954 | 2.15 |
| 2-Heptanone | 9.95 | 34.9 | 75.4 | 24.3 | 32.2 | 104 |
| Octanal | 0.457 | 0.454 | 0.807 | 0.259 | 0.226 | 0.537 |
| 2-Nonanone | 4.50 | 9.88 | 25.8 | 10.7 | 12.0 | 31.1 |
| Nonanal | 0.425 | 0.307 | 0.551 | 1.479 | 0.839 | 0.656 |
| Decanal | 0.244 | 0.209 | 0.286 | 0.545 | 0.330 | 0.576 |
| 2-Undecanone | 1.04 | 1.46 | 3.60 | 1.29 | 1.77 | 5.08 |
| Dodecanol | 0.272 | 0.216 | 0.222 | 0.221 | 0.141 | 0.347 |
| 2-Tridecanone | 0.409 | 0.166 | 0.325 | 0.502 | 0.401 | 0.392 |

appeared. A series of fourteen C10 to C13 linear alkyl substituted benzene isomers (LABs, Gilbert, 1969) of uncertain origin, were present in all these samples. Since these compounds do not appear in blank runs, their origin from Tenax degradation can be ruled out.

Table 4 shows the concentration of some selected compounds in the samples heated at 120°C in the glycerol bath and in the microwave oven at different times. The main components were methyl ketones (about 50–100-fold more abundant that in pasteurised samples), whereas aldehydes, alcohols, hydrocarbons and esters were found in lower proportion (near 1 μg/l). Methyl ketones containing from 7 to 11 carbon atoms seem to be related to the heating intensity and have also been found as the main volatile components in sterilised milk (Blanch, Calvo, Herraiz & Reglero, 1996) and also in UHT milk (Badings & Neeter, 1980; Vallejo-Córdoba & Nakai, 1993). Many components were present in higher concentrations in the microwave treated samples. Conventionally heated samples presented a colour similar to that of commercial sterilised milks, whereas the milk submitted to microwave treatment had a more intense brownish colour. Similarly, in a series of experiments carried out by heating milk samples at 120°C using the same vessels and oven, lactulose and furosine concentrations were higher after the microwave heating than after the conventional one (Villamiel, Corzo, et al., 1996). This effect could be related to the irradiation unevenness within the oven (Giese, 1992; Villamiel, López-Fandiño, Corzo, Martínez-Castro & Olano, 1996b).

It can be concluded that the volatile composition of milk, as determined by the present method, is the same after a microwave treatment as after a conventional one, provided that the heating is uniform and the temperature control satisfactory. The observed differences are quantitative and seem to be dependent on the heating intensity.

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