The Effect of Various UHT Processing Parameters and Storage Conditions on the Saturated Aldehydes In Half-and-Half Cream

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ABSTRACT

Raw cream was standardized to 10% fat and processed by ultra-high-temperature (UHT) steam injection at 149°C for 20 s, 149°C for 3.4 s, 138°C for 20 s, and 143°C for 7 s, then aseptically packaged by a Tetra Pak AB3-250 filler. Packages were stored for 12 months at 24°C and analyzed at 0, 1, 3, 6, and 12 months. Fat-soluble carbonyls were extracted from the UHT cream with carboxylic-free hexanes and converted to their 2,4-dinitrophenyl-hydrazone derivatives. Alkanal hydrazones were separated by column chromatography into pure fractions. Spectrophotometry and gas chromatography were used to identify the alkanals. Butanal, 2-pentanone, and 2-heptanone in high-temperature (HTST) milk packaged in glass bottles. Mehta and Bassette (10) reported increases in propanal, pentanal, butanal, heptanal, furfural, acetone, 2-butpanone, ethanol, butanol, 2-pentanone, and 2-heptanone in high-temperature short-time (HTST) milk packaged in glass bottles. Mehta and Bassette (10) reported increases in propanal, pentanal, and hexanal in UHT milk packaged in tin cans. Oxygen could have been the major reason for the increase in aldehyde compounds (21).

Schott et al. (17) proposed that aldehydes of less than 12 carbons in chain length are a result of autoxidation. Parks et al. (14) suggested that C11-C16 saturated aldehydes are present in milk as the result of milk-lipid synthesis or hydrolysis of neutral plasmalogens during heat treatment. Kirk et al. (9) identified increases in ethanal, propanal, butanal, heptanal, furfural, acetone, 2-butpanone, ethanol, butanol, 2-pentanone, and 2-heptanone in high-temperature short-time (HTST) milk packaged in glass bottles. Mehta and Bassette (10) proposed that C11-C16 saturated aldehydes are present in milk as the result of milk-lipid synthesis or hydrolysis of neutral plasmalogens during heat treatment.

Kirk et al. (9) identified increases in ethanal, propanal, butanal, heptanal, furfural, acetone, 2-butpanone, ethanol, butanol, 2-pentanone, and 2-heptanone in high-temperature short-time (HTST) milk packaged in glass bottles. Mehta and Bassette (10) proposed that C11-C16 saturated aldehydes are present in milk as the result of milk-lipid synthesis or hydrolysis of neutral plasmalogens during heat treatment.

The purpose of this research was to determine the changes in concentration of saturated aldehydes in UHT half-and-half cream. The concentration of saturated aldehydes was quantitated because aldehydes are primarily responsible for off-flavors in milk and are indicative of oxidation of milk lipids.

MATERIALS AND METHODS

Half-and-half cream

Four tanks of summer cream averaging 10% fat and 17.9% total solids were obtained from the North Carolina State dairy herd and processed in a No-Bac Aro-Vac (Cherry-Burrell Corp., Chicago, IL) by UHT steam injection using four processes: 149°C for 20 s (P-1), 149°C for 3.4 s (P-2), 138°C for 20 s (P-3), and 143°C for 7 s (P-4). Aseptic packaging into 250-ml containers was accomplished by a Tetra Pak Model AB3-250 filler (Tetra Pak, Inc., Shelton, CT). Samples from each process were stored at room temperature (24°C ± 2°C) and analyzed at 0, 1, 3, 6, and 12 months. Duplicate analyses were conducted on the alkanals.

Fat extraction and reaction

Cream was centrifuged for 45 min at 10,000 g and the fat content of the cream layer was determined by the Mojonier method. Thirty g of cream (testing 50% fat) was impregnated with an equal weight of Celite 545 by mixing with a mortar and pestle.
The fat and carbonyls were eluted through a 2,4-dinitrophenylhydrazine reaction column prepared according to the method of Schwartz et al. (18). Fat and carbonyls were extracted using carbonyl-free hexanes (300 ml). The reaction column was flushed with additional carbonyl-free hexanes (200 ml) to ensure that all fat and carbonyls had been removed from the column.

Fat and dicarbonyl 2,4-dinitrophenylhydrazone derivatives (2,4-DNPH) were removed from the eluate by using activated Celite 545 (10 g) mixed with adsorptive magnesia (10 g) in a chromatographic column. Neutral lipid material passed through the column while 2,4-DNPH adsorbed to the magnesia. Two hundred ml of a 3:1 mixture (v/v) of chloroform and nitromethane was added to elute the monocarbonyl 2,4-DNPH. Dicarbonyl 2,4-DNPH remained on the column as a bright lavender band. The effluent containing the monocarbonyl and the ketoglyceride 2,4-DNPH was evaporated and redissolved in redistilled hexanes (10 ml).

The ketoglyceride 2,4-DNPH in the redissolved sample were removed by using deactivated alumina (20 g) packed in a chromatographic column. The sample was eluted with 150 ml of a 1:1 mixture (v/v) of benzene and hexanes. Ketoglyceride 2,4-DNPH remained adsorbed on the column while the benzene-hexanes eluate contained the monocarbonyl 2,4-DNPH. Solvents were evaporated in a rotary evaporator, and 2,4-DNPH were redissolved into redistilled hexanes (10 ml).

A 1:1 mixture of Celite-magnesia (similar to the defatting column) was used to separate the monocarbonyl 2,4-DNPH into classes. Monocarbonyl 2,4-DNPH were applied to the top of the column. The separation of alk-2-ones was accomplished by eluting with 15% chloroform in hexanes (250 ml). The aldehydes were removed by eluting with 60% chloroform in hexanes (200 ml). Aliquots (5-ml) were collected using a Gilson fraction collector (Gilson Medical Electronics, Middleton, WI). Each fraction was reduced to dryness and redissolved in chloroform (10 ml).

The samples were scanned on a Gilford Spectrometer Model 2600 (Gilford Instrument Laboratories, Oberlin, OH) from 325 nm to 390 nm. Fractions were pooled according to the following wavelength maxima: alk-2-ones at 365 nm and alkanals at 355 nm. Total carbonyl concentration was calculated by using the molar extinction coefficient reported by Schwartz et al. (18). The alk-2-DNPH were taken to dryness and quantitated using the following procedure.

Analysis of fractions

Each sample of alkanal 2,4-DNPH was analyzed by gas chromatography (F&M Model 810, Hewlett-Packard, Avondale, PA) using a Texas Instruments 960A flame ionization detector (Texas Instruments, Houston, TX), a glass column of 2-3% OV17 on Anakrom ABS (110-120 mesh, 3.175 mm OD x 1.8 m), and 40 ml/min helium carrier. Injector and detector temperatures were 290°C and 315°C, respectively. Hydrogen and air-flow rates were 30 and 45 ml/min, respectively. Samples were dissolved in ethyl acetate (1 ml) and 1.0 µl was injected onto the column. Anthracene (1 µM) was added to each 2,4-DNPH sample as a standard. Identification of the individual alkanal 2,4-DNPH within each sample was accomplished by comparing their retention times with those of the C5-C10 authentic standards (Chem. Service, Media, PA). Peak areas were integrated electronically and compared to the standard peak area to determine the concentration of alkanal 2,4-DNPH present.

Statistical analysis

Data from gas chromatographic analyses were subjected to regression analysis to establish the relationship of n-alkanal concentration to storage for each of the four processing treatments. The model \( s = a + bt +ct^2 \) where \( s \) is n-alkanal concentration, \( t \) is storage time, and \( a, b \) and \( c \) are coefficients to be determined was fitted to the four process conditions.

RESULTS AND DISCUSSION

Changes in concentration of C5-C10 C6-C10 alkanals over time from each process are shown in Fig. 1-4. Each plot contains data from the four processes used to treat the cream. In reviewing Fig. 1, P-1 (149°C for 20 s) showed decreases in propanal until 6 months then the propanal concentration increased. P-2 (149°C for 3.4 s) and P-4 (143°C for 7 s) showed only slight increases in propanal from time zero to 12 months. P-3 (138°C for 20 s) showed slight increases in propanal to 6 months then decreased. Butanal increased slightly in concentration in P-2. P-3 and P-4 showed increases in butanal to months 6 and 4, respectively, then decreased. P-1 decreased from 1.280 µmoles butanal/g fat to 0.272 µmoles butanal/g fat at 12 months.

Figure 1. Changes in propanal and butanal concentration in UHT half-and-half cream processed at 149°C for 20 s (Process 1), 149°C for 3.4 s (Process 2), 138°C for 20 s (Process 3), 143°C for 7 s (Process 4), and stored at 24°C over time.
In Fig. 3, octanal decreased in every process with slight exceptions in P-1 and P-3 which showed slight increases for 3 and 5 months, respectively, then decreased. Nonanal decreased in every process with the exception of P-4 which showed an erratic increase for 5 months before decreasing. In Fig. 4, decanal decreased in P-1, P-2, and P-4, but P-3 increased slightly to 6 months before decreasing.

Total aldehyde concentration decreased during storage for each of the process conditions with the exception of propanal and hexanal which increased in P-1 after 4 and 6 months, respectively. Butanal increased in P-3 then decreased, whereas P-2 increased slightly. The increase of propanal, butanal, and hexanal in the different processes may be from the oxidation of unsaturated fatty acids.

The total carbonyl (aldehydes plus ketones) concentration decreased in every process (Fig. 5). The total carbonyl concentration was calculated to be 3.750-6.428 μmoles/g fat higher in P-2, than in P-1, P-3, and P-4. Carboxyls in P-2 decreased to 0.107 μmoles/g fat by 9 months before slowly increasing. P-3 decreased to zero in 10.5 months, while P-4 decreased to 0.107 μmoles/g fat by 9 months before slowly increasing. P-3 decreased to zero in 10.5 months, while P-4 decreased to 0.107 μmoles/g fat by 9 months before slowly increasing.
1 and P-4 had approximately 1.178 and 1.929 µmoles/g fat at 12 months, respectively. The loss of short-chain aldehydes was more erratic than long-chain (C₅-C₉) aldehydes which showed a progressive loss each month.

The decrease in the concentration of the various aldehydes may be the result of their binding to heat denatured milk proteins or peptides as reported by other researchers (3,6,12,13). Heat denatured proteins have been shown to have an increased capacity for binding aldehydes (13).

It has also been shown that polyethylene and other types of packaging materials will sorb flavor compounds (16). As aldehydes become more nonpolar, there is a greater sorption of long-chain aldehydes into the packaging material (16) which agrees with the results of our experiments.

Unlike previous research (9,10,11) which showed increases in alkanal concentrations, our data show that the total butanal, hexanal, heptanal, octanal, and decanal concentrations declined during storage. The discrepancy between other research and the work reported here may be the result of raw milk quality, metallic catalysts, oxygen dissolved in the milk, or packaging material. Also, other published research that reported the development of oxidized flavors during storage did not deaerate the samples before packaging and contained headspace in the container (9,11,21).

Figure 5. Changes in total carbonyl (aldehydes, ketones, enals, dienals, ketoacids, ketoglycerides, and dicarbonyls) concentration in UHT half-and-half cream processed at 149°C for 20 s (Process 1), 149°C for 3.4 s (Process 2), 138°C for 20 s (Process 3), 143°C for 7 s (Process 4), and stored at 24°C over time.

REFERENCES