Journal of Food Protection, Vol. 46, No. 11, Pages 950-953 (November 1983)

Effect of Process- and Storage-Times and Temperatures on Concentrations of Volatile Materials in Ultra-High-Temperature Steam Infusion Processed Milk[1]

R. BASSETTE* and I. J. JEON

Department of Animal Sciences and Industry, Kansas State University, Manhattan, Kansas 66506

(Received for publication April 20, 1983)

ABSTRACT

Effect of various processing times and temperatures on the composition and subsequent changes in concentration of volatile compounds in UHT milk during storage was investigated. Milk samples were sterilized in a DASI Free Falling Film steam infusion system utilizing the combinations of 138, 146, and 154°C for 1.5, 3.4 and 9.0 s. After processing, aseptically collected samples were stored at refrigeration (2-5°C) and room (25°C) temperature for analysis at monthly intervals. Gas chromatographic analysis showed that processing times and temperatures used had little effect on initial concentrations of most volatile compounds in UHT milk. However, changes in their concentrations during storage appeared to be related to processing temperatures and temperature of storage. Acetaldehyde and n-pentanal increased more rapidly in the milk sterilized at 154°C/3.4 s than 146°C/3.4 s, whereas n-hexanal concentrations were lower in milk sterilized at 154°C/3.4 s. In addition, changes in concentrations of volatile compounds during storage at room temperature occurred primarily in aliphatic aldehydes. Increases in acetaldehyde, n-pentanal, and n-hexanal were closely related to the rapid decrease in product acceptability that was mainly due to increase in the intensity of stale flavor. Relatively little change occurred in the concentration of these aldehydes in milk stored at refrigeration temperature.

The role of neutral volatile compounds in flavor deterioration of ultra-high-temperature (UHT) sterile milk during storage has been studied by a number of researchers (*4,6,9,10,12*). Most indicated that carbonyl compounds increased during storage and were closely associated with development of stale flavor (*6,9,10,12*). However, one study reported decreases in some volatile aldehydes (*4*). Kirk et al. (*9*) observed that staling in stored UHT milk paralleled increases in carbonyl GLC peaks as well as the disappearance of many unidentified, less volatile compounds. Jeon et al. (*6*) suggested that although odd-numbered methyl ketones ($C_{3\text{-}13}$) were most abundant, n-aldehydes ($C_{3,5,6,7,8,9}$) contributed most to off-flavors of stored UHT milk. Mehta and Bassette (*10*) observed that increase in stale off-flavor intensity in UHT milk stored at 22°C occurred concurrently with increases in propanal, n-pentanal and n-hexanal. They also observed that when UHT milk was stored in aseptic aluminum foil-lined or plain polyethylene-lined cartons, propanal, n-pentanal, n-hexanal, and two unidentified components were all higher in the polyethylene-lined cartons than in the less air permeable aluminum foil-lined cartons (*12*).

In this study we investigated the effect of various UHT processing times and temperatures on the composition of volatile compounds and subsequent changes in their concentration during storage.

MATERIALS AND METHODS

Sample preparation

Milk samples for this research were prepared utilizing a DASI Free Falling Film steam infusion unit (*13*) at the University of Maryland Dairy Processing Plant. Milk was preheated to 82.2°C and then sterilized by combinations of 138, 146 and 154°C and 1.5, 3.4 and 9.0 s. Each combination of these times and temperatures was evaluated. Since only one holding time was workable in a single processing trial, three separate trials, approximately 2 weeks apart, were necessary to complete all combinations of times and temperatures used. Once processed, milk was packaged and sealed aseptically in sterile 500-ml glass serum bottles in a glove box, as described by Nahra and Westhoff (*13*). Bottles were refrigerated at 2 - 4°C within 2 - 4 h of processing and shipped by air to Manhattan, KS on the same day. Eight bottles of each time - temperature series were shipped to Kansas State University. On arrival, bottles were examined for damage, rinsed with an 800 ppm chlorine solution and wrapped in aluminum foil. One of the bottles was analyzed for sterility, flavor, and volatile materials by GLC within 2 d of the time of processing. Three of the remaining seven samples were stored at refrigeration temperature, 2 - 5°C, and the other four were held at 25°C for analysis of sterility, flavor and volatile materials at monthly intervals.

---

[1]*Contribution No. 83-131-J, Department of Animal Sciences and Industry, Kansas Agricultural Experiment Station, Kansas State University, Manhattan, Kansas 66506.*

Microbiological analyses

Two microbiological determinations were made. One milliliter of milk was aseptically drawn, plated, and incubated at 32°C for a Standard Plate Count (SPC). In addition, a 10-ml portion of "sterile" milk was aseptically transferred to a sterile tube and incubated at 32°C for 1 week and then analyzed for SPC. Aseptic sample drawings were not only essential for aseptic transfers but also were necessary to save "sterile" milk samples for later use in organoleptic and GLC analysis. As the study progressed, it was possible to detect incidence of non-sterility by observing the physical appearance of milk in the bottles. Where deterioration was noted or where tests indicated lack of sterility, those samples were discarded and where possible tests were done again.

Gas chromatographic analysis of sterile milk

Concentrations of acetaldehyde, n-pentanal, and n-hexanal in sterile milk as a function of time and temperature of storage were determined by the head space gas sampling and GLC methods of Bassette and Ward (*1*). All analyses were in duplicate and concentrations of the aldehydes were calculated from regression equations of standard curves of each aldehyde over the range of concentrations observed.

Organoleptic flavor analyses

A panel of five judges, experienced in grading milk, evaluated all of the samples throughout the study. Milks were scored using the 10-point dairy products collegiate judging system. A coded sample of fresh pasteurzied milk was included with each flavor panel session. Although panelists were encouraged to report all criticisms, they were instructed to give particular attention to the appearance and intensities of cooked and stale flavors.

RESULTS AND DISCUSSION

The purpose of the bacteriological examinations of the milk was to confirm sterility and ensure elimination of any off-flavors from bacteriological deterioration. At least one bottle of the processed milk from each trial was not sterile when tested 2 d after processing. This was not related to processing temperatures and times. In the first trial, the 138 and 146°C/1.5 s samples were sterile, whereas a bottle of the 154°C milk was not. A few bottles of 146°C and 154°C/3.4 s of the second trial and a few of 138°C and 146°C/9 s of the third tiral were not sterile. Where there was bacterial growth in the room temperature samples after 1 month, non-sterility was obvious and these bottles were discarded. In refrigerated milks, bacterial growth was not visually apparent after storage but was detected by bacteriological examination. If bacterial growth was found the sample was discarded.

Gas chromatographic analysis of fresh UHT milk showed most of the volatile compounds identified previously by Mehta and Bassette (*10*). Among those detected were acetaldehyde, methyl sulfide, acetone, n-pentanal, and n-hexanal, with the surprising absence of n-propanal, which had been found in the previous studies (*10,11,12*). Table 1 shows the effect of various processing times and temperatures on the initial concentrations of volatile compounds in UHT milk. These data indicate that the processing temperature-and time-range used (138-154°C for 1.5-9.0 s) had little effect on the initial concentrations of most volatile compounds in milk. Although sulfur compounds and methyl ketones can occur upon heating milk (*2,7,14*), methyl sulfide and acetone concentrations were not higher at 154°C sterilization than at 138°C. Little difference in concentrations of these volatile compounds may reflect the relatively mild UHT processing conditions of the DASI steam infusion system. Also, decreases in concentrations of some volatile materials in milk exposed to the highest heat might represent removal of those compounds during the flash evaporative cooling stage of the process. It has been reported that the evaporative process removes a major portion of aromatic and sulfur components, as well as moisture and oxygen in UHT milk (*5*).

Changes in concentrations of volatile compounds during storage occurred primarily in aliphatic aldehydes. Acetaldehyde, n-pentanal, and n-hexanal all increased in concentration at room temperature with little change at refrigeration temperature (Fig. 1, 2 and 3). Similar results were reported by Jeon et al. (*6*) with the indirect UHT milk stored at 4, 22 and 37°C. These results indicate that changes in aldehydes are highly temperature-dependent. Early and Hansen (*4*), however, observed decreases in some aldehydes, such as methanal, propanal, and butanal, during storage of UHT steam injection-processed milk at 24 and 40°C, but surprisingly no detectable quantities of other aldehydes. Since oxygen also plays an important role in the formation

TABLE 1. *Effect of processing time and temperature on concentration of major volatile compounds in freshly-processed UHT milk.*

UHT processing		Concentration of volatile compounds (ppb)[a]				
Time (s)	Temperature (°C)	Acetaldehyde	Methyl sulfide	Acetone	n-Pentanal	n-Hexanal
1.5	138	20	104	2220	77	8
	146	21	139	2610	120	4
	154	20	104	1900	110	6
3.4	138	-[b]	-	-	-	-
	146	15	79	1420	100	6
	154	15	66	1760	86	6
9.0	138	22	49	1490	86	31
	146	25	57	1830	110	29
	154	34	43	1620	97	16

[a]Samples were 2 d old at a refrigeration temperature when analyzed, and each data point represents an average of duplicates.
[b]-No data available.

of aldehydes during storage of UHT milk (*3,9*), these differences may reflect different levels of dissolved oxygen caused by the processing methods or permeability of packaging materials used. It also could be the result of different analytical methods used.

Changes in aldehyde concentrations during storage appear to be related to processing temperatures. Figure 1 shows that after 1 month of storage at room temperature, increases in concentration of acetaldehyde were similar in the milks sterilized at 146 or 154°C. After 3 months, however, a sharp increase in acetaldehyde was observed in milk sterilized at 154°C. Although acetaldehyde concentrations increased sharply in the 154°C -sterilized samples, its

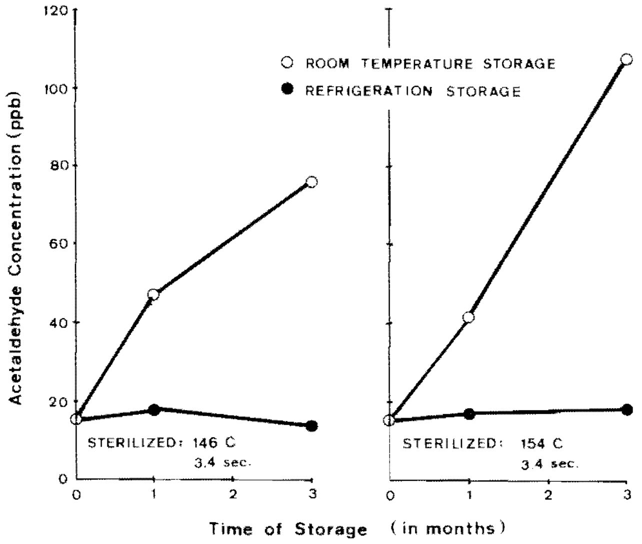

Figure 1. *Changes in acetaldehyde concentrations for the milks sterilized at 146 and 154°C for 3.4 s and stored at room and refrigeration temperatures.*

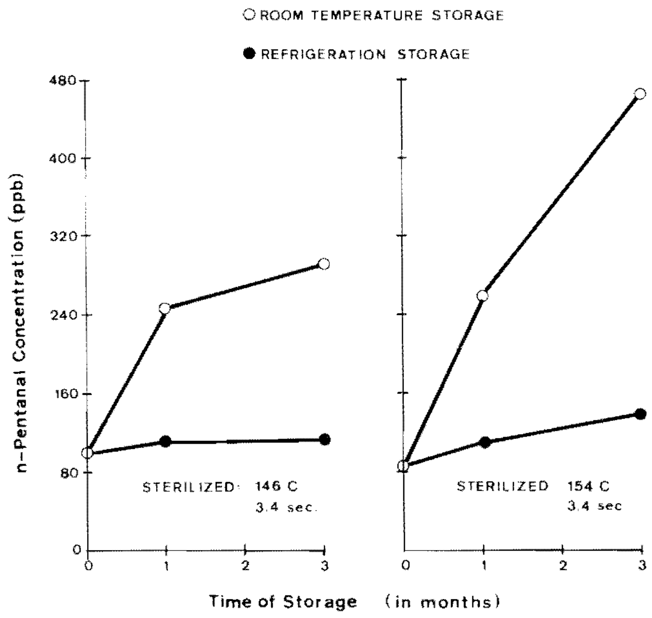

Figure 2. *Changes in n-pentanal concentrations for the milks sterilized at 146 and 154°C for 3.4 s and stored at room and refrigeration temperatures.*

concentrations were well below the reported flavor threshold level of 1.2 ppm in milk (*8*). Changes in n-pentanal concentration during storage were similar for the two processing temperatures (Fig. 2). However, from the beginning, n-pentanal concentrations were close to the reported threshold level of 0.13 ppm (*3*), and increased 2 to 3 times above the threshold level after 3 months at room temperature. n-Hexanal also increased in concentrations in milk from both processing temperatures during storage (Fig. 3), but the increases were higher in the samples sterilized at 146°C than at 154°C. This is a different pattern from that of acetaldehyde and n-pentanal. Initial concentrations of n-hexanal were well below the reported threshold level of 0.049 ppm (*3*). After 3 months at room temperature, however, the hexanal concentration was above the detectable level in the 146°C -sterilized samples and was more than 2/3 of that level in the 154°C -sterilized milk.

The increases in concentration of n-pentanal and n-hexanal from subthreshold to above or near threshold level

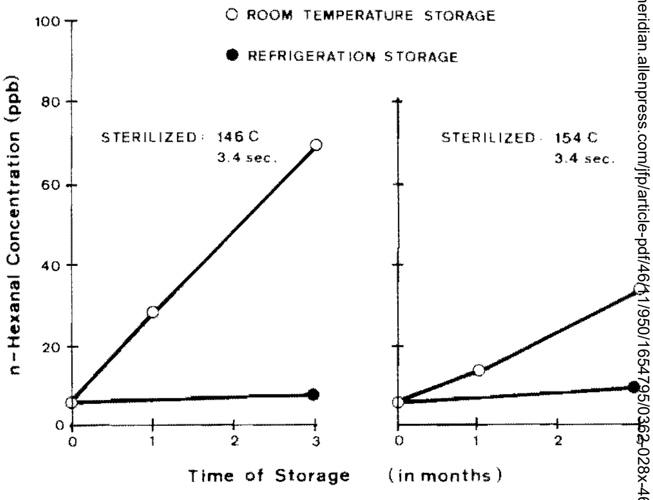

Figure 3. *Changes in n-hexanal concentration for the milks sterilized at 146 and 154°C for 3.4 s and stored at room and refrigeration temperatures.*

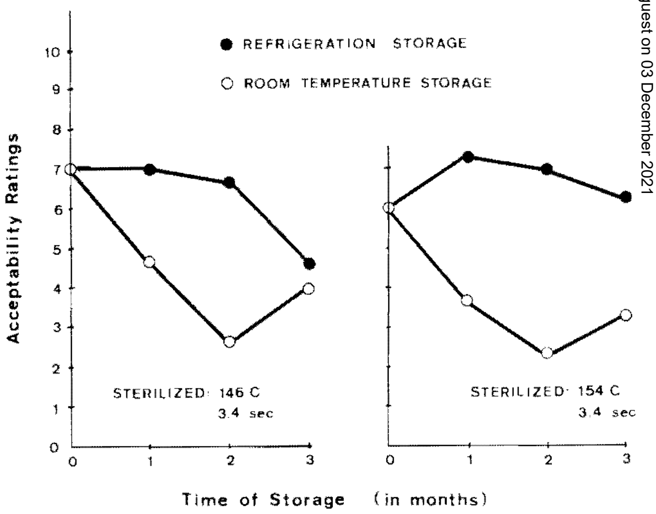

Figure 4. *Mean scores of acceptability ratings for the milks sterilized at 146 and 154°C for 3.4 s and stored at room and refrigeration temperatures.*

during storage appears to be important in contributing to off- flavor of UHT milk. If we consider the additive or synergistic effect of carbonyl compounds in milk (*3*), the effect of these aldehydes on the flavor quality can become even more significant. Results of our 5-member experienced panel indicated that these increases in aldehyde concentrations were closely related to the rapid decrease in product acceptability (Fig. 4). The decrease in acceptability ratings during storage was mainly due to increase in the intensity of stale flavor. The samples stored at room temperature were rated slightly stale after 1 month, and rated definitely stale on further storage. For samples stored at refrigeration temperature, however, acceptability ratings remained high with few increases in the initial aldehyde concentrations.

ACKNOWLEDGMENT

We thank D. C. Westhoff for providing the UHT milk used for this research and for his cooperation throughout the study. We also acknowledge the University of Maryland for the use of its facilities and DASI Industries, Inc. for the use of the UHT processing equipment.

REFERENCES

1. Bassette, R., and G. Ward. 1975. Measuring parts per billion of volatile materials in milk. J. Dairy Sci. 58:428-429.
2. Day, E. A. 1966. Milk lipids in dairy products. *In* Flavor chemistry, ACS Publication No. 56, pp. 94-120.
3. Day, E. A., D. A. Lillard, and M. W. Montgomery. 1963. Autoxidation of milk lipids. III. Effect on flavor of the additive interactions of carbonyl compounds at subthreshold concentrations. J. Dairy Sci. 46:291-294.
4. Early, R. R., and A. P. Hansen. 1982. Effect of process and temperature during storage on ultra-high temperature steam-injected milk. J. Dairy Sci. 65:11-16.
5. Hostettler, H. 1972. Appearance, flavor, and texture aspects. pp. 6-34. *In* International Dairy Federation. IDF monograph on UHT milk, Brussels, Belgium.
6. Jeon, I. J., E. L. Thomas, and G. A. Reineccius. 1978. Production of volatile flavor compounds in ultrahigh temperature processed milk during aseptic storage. J. Agric. Food Chem. 26:1183-1188.
7. Keenan, T. W., and R. C. Lindsay. 1968. Evidence for a dimethylsufide precursor in milk. J. Dairy Sci. 51:112-114.
8. Kinsella, J. E., S. Patton, and P. S. Dimick. 1967. The flavor potential of milk fat. A review of its chemical nature and biochemical origin. J. Amer. Oil Chem. Soc. 44:449-455.
9. Kirk, J. R., T. I. Hedrick, and C. M. Stine. 1968. Gas chromatographic study of flavor deterioration in high-temperature short-time fluid sterile milk. J. Dairy Sci. 51:492-497.
10. Mehta, R. S., and R. Bassette. 1978. Organoleptic, chemical, and microbial changes in ultra-high-temperature sterilized milk stored at room temperature. J. Food Prot. 41:806-810.
11. Mehta, R. S., and R. Bassette. 1979. Volatile compounds in UHT-sterilized milk during fluorescent light exposure and storage in the dark. J. Food Prot. 42:256-258.
12. Mehta, R. S., and R. Bassette. 1980. Effects of carton material and storage temperature on the flavor of UHT-sterilized milk. J. Food Prot. 43:392-394.
13. Nahra, J. E., and D. C. Westhoff. 1980. Direct sterilization of heat-sensitive fluids by a free-falling-film sterilizer. Food Technol. 34(9):49-55, 57.
14. Scanlan, R. A., R. C. Lindsay, L. M. Libby, and E. A. Day. 1968. Heat-induced volatile compounds in milk. J. Dairy Sci. 51:1001-1007.

Davis and Anderson, *con't. from p. 949*

6. Crespo, F. L., and H. W. Ockerman. 1977. Effect of heat on sarcoplasmic proteins of light and dark avian muscle tissue. J. Food Prot. 40:174.
7. Crespo, F. L., and H. W. Ockerman. 1977. Effect of heating avian muscle tissue on solubility of nitrogen fractions and pH values of breast and leg muscle. J. Food Prot. 40:178.
8. Hamm, R., and F. E. Deatherage. 1960. Changes in hydration solubility and protein charges of muscle proteins during heating of meat. Food Res. 25:587.
9. Heidelbaugh, N. D., and J. H. Graves. 1968. Effects of some techniques applicable in food processing on the infectivity of foot and mouth disease virus. Food Technol. 22:120.
10. Layne, E. 1957. Spectrophotometric turbidimetric methods for measuring proteins. p. 450. *In* S. P. Colowick, and N. O. Kaplan. (eds.) Methods in enzymology, Vol. 3. Academic Press, Inc., New York.
11. Lee, Y. B., D. A. Rickansrud, E. C. Hagberg, and E. J. Briskey. 1974. Application of SDS-acrylamide gel electrophoresis for determination of the maximum temperature to which bovine muscles have been cooked. J. Food Sci. 39:428.
12. McLaughlin, J. V., and G. Goldspink. 1963. Post-mortem changes in the color of pig longissimus dorsi muscle. Nature 198:584.
13. Trautman, J. C. 1966. Effect of temperature and pH on the soluble proteins of ham. J. Food Sci. 31:409.
14. USDA-APHIS. 1982. Code of Federal Regulations. Title 9. Part 94.9(b)(1)(ii)(b), 304; Part 94.12 (b)(1)(ii)(B), 307.
15. USDA-FSIS. 1982. Code of Federal Regulations. Title 9. Part 318.10 (c)(1)(i), 218.