Freezing point of raw and heat-treated goat milk

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ABSTRACT: The freezing point (FP) was established in 48 bulk tank samples of raw and 48 samples of pasteurized goat milk that were collected in the course of lactation. Alongside, non-fat solids (NFS) content was monitored. Milk freezing point measurements were carried out using the thermistor cryoscope method in compliance with the standard CTS 570538 (1998). The mean freezing point of raw milk was found to be in an interval of –0.5513 ± 0.0046°C, variation ranged from –0.5466°C to –0.5567°C, with higher values in the spring months and a drop at the end of lactation. FP corresponded to the NFS content. The average freezing point of goat milk heat-treated on the farm to the temperature of 72°C over a period of 20 s was –0.5488 ± 0.0046°C, pasteurisation brought an average increase in FP by 0.0025°C.

Keywords: cryoscopy; milk; goats; pasteurization; freezing point

Goat milk production accounts for only 2% of the milk produced worldwide (Haenlein, 2002). In spite of this fact, it plays a very important role as far as the economy of some countries is concerned. The principal European producers are situated along the Mediterranean Sea. Greece, France and Portugal produce 50% of goat milk in the whole of the European Union (Morgan et al., 2003).

Freezing point belongs to the key properties of milk. Many factors affect it. From among the main milk constituents, lactose and chlorides mostly influence FP. If combined, they account for 75–80% of the final FP. The remaining 20–25% of the FP value is affected by other milk constituents – calcium, magnesium, lactates, phosphates, citrates, urea, etc. (Fox and McSweeney, 1998). Milk fat globules, casein micelles and whey proteins play a negligible role in the reduction of milk FP (Bhandari and Singh, 2003). Kessler (1984) also stated that milk fat exerted no effect on milk FP and milk proteins displayed a minimum action as far as the value of freezing point is concerned.

When compared to cow milk FP, goat milk FP is lower. For instance, Szijarto and van de Voort (1983) reported its value –0.5527°C. Variation in the FP values in cow and goat milk is caused by a higher content of non-fat solids in goat milk (Alichanidis and Polychroniadou, 1995).

The freezing point of milk is determined by the proportion of milk constituents in a true solution and whose content in milk is affected by a number of factors: e.g. breed, stage and number of lactation, occurrence of subclinical mastitis, nutritional deficiencies, water intake, weather conditions, thermal stress, seasonal influences, presence of CO_2 in milk (Rohm et al., 1991; Wiedemann et al., 1993; Antunac et al., 2001; Slaghuis, 2001). FP values also vary in goats from different regions (Espie and Mullan, 1990).

Water content has a great impact on FP (Szijarto and van de Voort, 1983). Adulteration with water can be wilful or it can be caused by negligence, especially by technological imperfections in the primary production. The main causes of milk adulteration in the primary production remain the imperfections in the structure of the milking equipment or in the equipment sanitation. In these situations, residual and condensed water passes into the milk (Zee et al., 1982).

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The FP value of heat-treated milk depends on the FP value of raw milk and on changes in the milk FP in the process of heat treatment. Heat treatment causes an increase in the FP of pasteurized milk ranging from 0.001 to 0.009°C as opposed to the FP values of raw milk (Kessler, 1984; Singhal et al., 1997). Rohm et al. (1991) ascribed the increase in milk FP to a change in the calcium phosphate complex and to a change in the pressure of carbon dioxide. This shift is represented by approximately 0.002°C depending on the temperature and duration of heat treatment. The following influences can also come into action: milk adulteration e.g. with residual water, loss of salts caused e.g. by the formation of milk stone deposits, changes in milk acidity e.g. due to the formation of lactic acid while fermenting lactose and gas content in milk (Kessler, 1984). During the production, handling and processing of milk, small quantities of water may adulterate milk. This may result from washing and sterilising procedures or from careless practices. Therefore it is of practical importance to know the extent of variations in freezing point results and the upper tolerance limit for determination of extraneous water in factory milk (Tucker and Madsen, 1970).

The objective of this study was to determine the FP of raw and pasteurized goat milk, its changes in the course of lactation and the assessment of the effect of pasteurization on FP.

MATERIAL AND METHODS

Milk samples were collected on a goat farm in the South Moravian Region, Czech Republic. The farm has 75 goats of the White Shorthaired breed in the 1st to 8th lactation, with an average yield of 2–3 l a day, 600–800 l a year. In the period from mid-May to the beginning of November, the goats were fed a summer diet including grazing, 0.5 kg of hay and up to 1 kg of cereals (barley, triticale, oats), vitamin mineral mixture and salt. In the remaining period, they were fed a winter diet: 3 kg of grass hay, 1 kg of ensiled beet, 1 kg of hay and up to 1 kg of cereals, vitamin mineral mixture and salt.

Milking equipment was used for daily milking, once a day. After milking, the milk was cooled down to the temperature of 4–6°C. It was then kept at this temperature over a period of 12–24 hours until heat treatment by HTST method (temperature of 72°C for 20 s). The sample collection was performed after weaning kids in the period April–November 2005 in regular time intervals. Altogether, 48 samples of raw and 48 samples of pasteurized goat milk were collected. The samples, cooled to the temperature of 4–6°C, were transported so as to prevent their warming to more than 8°C. They were not preserved and following their laboratory reception, they were immediately analyzed.

The freezing point determination was carried out in compliance with the standard CTS 570538 (1998), Determination of Freezing Point in Milk – a Cryoscope Method, using a Cryostar Funke Gerber Automatic 7160 (Funke Gerber, Germany) cryoscope. The machine was always calibrated at the measuring day with standardized solutions of FP values –0.408°C and –0.600°C, and the calibration was verified by Lactrol Reference Solution (–0.512°C). The uncertainty of measurement was 0.61%. It is a combined type, at the level of probability U = 95% for distribution coefficient k = 2. FP measurements were performed in 48 samples of raw milk and 48 samples of milk pasteurized on the farm at the temperature of 72°C for 20 s.

Non-fat solids content (NFS) was determined by the analysis of the composition of raw and pasteurized milk in compliance with the standard CTS 570536 (1999) Determination of Milk Composition with Mid-infrared Analyzer, using a Bentley 2500 (Bentley Instruments, Minnesota, USA) analyzer.

Table 1. Average values of freezing point of raw goat milk in the course of lactation (°C)

Month	4	5	6	7	8	9	10	11	Lactation
$\bar{x}$	–0.5478	–0.5454	–0.5507	–0.5567	–0.5545	–0.5513	–0.5514	–0.5528	–0.5513
SD	0.0036	0.0027	0.0057	0.0023	0.0035	0.0022	0.0042	0.0025	0.0046
v_x	0.6506	0.4946	1.0312	0.4189	0.6325	0.3918	0.7538	0.4523	0.8372
Min	–0.5510	–0.5500	–0.5590	–0.5580	–0.5590	–0.5540	–0.5570	–0.5560	–0.5590
Max	–0.5420	–0.5420	–0.5450	–0.5540	–0.5500	–0.5490	–0.5450	–0.5500	–0.5490

$\bar{x}$ = average; SD = standard deviation; v_x = variation coefficient; min = minimum; max = maximum

Table 2. Average non-fat solids content in raw goat milk in the course of lactation (%)

Month	4	5	6	7	8	9	10	11	Lactation
$\bar{x}$	7.58	7.64	7.84	8.17	8.04	7.88	8.01	8.14	7.92
SD	0.04	0.08	0.07	0.02	0.01	0.14	0.20	0.08	0.22
v_x	0.53	1.06	0.91	0.22	0.39	1.80	2.55	1.01	2.78
Min	7.52	7.48	7.70	7.85	7.77	7.70	7.61	8.07	7.48
Max	7.63	7.75	7.89	8.17	8.04	8.07	8.18	8.26	8.26

$\bar{x}$ = average; SD = standard deviation; v_x = variation coefficient; min = minimum; max = maximum

Basic statistical processing was done using Unistat Software, version 5.1 (Unistat, 1998). Seasonal influences and effects of heat treatment of milk on FP were evaluated, and the correlation between FP and NFS was established.

RESULTS AND DISCUSSION

As Table 1 shows, the calculated monthly average values of freezing points of raw goat milk ranged in the course of lactation from –0.5454°C to –0.5567°C. The average value of FP over the whole period of monitoring was –0.5513 ± 0.0046°C, i.e. higher than that reported by Szijarto et al. (1983). They presented the average yearly value of FP of raw goat milk in the Ontario province to be –0.5527°C, which is, however, in agreement with the range of –0.551 to –0.548°C established by Petrova et al. (2001).

The results document seasonal changes in FP of raw goat milk that are caused by lactation, weather changes and seasonal nutritional variance. The minimum detected value of FP of the whole set of measurements (n = 48) was –0.5590°C (month 8) and the maximum –0.5420°C (in months 4 and 5). At the beginning of the monitored period, the average value of FP was –0.5478 ± 0.0036°C (month 4), month 5 brought another increase (–0.5454 ± 0.0027°C), probably due to transition from winter diet to grazing. In the following period, there was a reduction of FP, the lowest values being found in the summer season (–0.5567 ± 0.0023°C and –0.5545 ± 0.0035°C). The effect of high environmental temperatures and organism dehydration can also be the causes of this FP reduction. Bjerg et al. (2005) reported a reduction in FP of milk due to short-term dehydration. In autumn, in months 9 and 10, the detected values were –0.5513 ± 0.0022°C and –0.5514 ± 0.0042°C. The grazing season terminated by the beginning of month 11 and winter diet set in. Lower milk yield and invariable feed rations in month 11 contributed to lower values of FP in milk (–0.5528 ± 0.0025°C).

Our findings of FP changes in raw goat milk in the course of lactation correspond to those presented by Antunac et al. (2001), namely the fact that there are maximum FP values at the beginning of lactation and lower ones at the end of lactation. These authors also assessed the impact of the stage and number of lactation on FP values and they stated that in the course of the first three lactations FP was from –0.540 to –0.548°C, and there was a higher variation during the 4^{th} and 5^{th} lactation (–0.548 to –0.574°C).

FP values follow the trends of non-fat solids content (NFS). As Table 2 documents, NFS content in raw goat milk ranged from 7.58 to 8.17%, the average value being 7.92 ± 0.22%. The lower NFS content in milk at the beginning of the monitored period corresponded to the increased FP. By

Table 3. Average values of freezing point of pasteurized goat milk in the course of lactation (°C)

Month	4	5	6	7	8	9	10	11	Lactation
$\bar{x}$	–0.5400	–0.5452	–0.5480	–0.5520	–0.5525	–0.5507	–0.5506	–0.5512	–0.5488
SD	0.0014	0.0033	0.0019	0.0031	0.0020	0.0022	0.0038	0.0022	0.0046
v_x	0.2619	0.5952	0.3475	0.5490	0.574	0.3923	0.6803	0.4022	0.8451
Min	–0.5420	–0.5500	–0.5480	–0.5550	–0.5560	–0.5540	–0.5550	–0.5540	–0.5560
Max	–0.5360	–0.5420	–0.5440	–0.5490	–0.5500	–0.5480	–0.5450	–0.5480	–0.5540

$\bar{x}$ = average; SD = standard deviation; v_x = variation coefficient; min = minimum; max = maximum

Table 4. Average non-fat solids content in pasteurized goat milk in the course of lactation (%)

Month	4	5	6	7	8	9	10	11	Lactation
$\bar{x}$	7.48	7.84	7.88	7.88	7.93	7.89	7.99	8.17	7.93
SD	0.01	0.17	0.05	0.01	0.21	0.16	0.40	0.06	0.25
v_x	0.21	2.14	0.59	0.09	2.71	1.99	0.13	0.70	3.11
Min	7.46	7.66	7.82	7.88	7.75	7.73	7.19	8.09	7.19
Max	7.50	8.06	7.94	7.89	8.35	8.07	8.60	8.21	8.60

$\bar{x}$ = average; SD = standard deviation; v_x = variation coefficient; min = minimum; max = maximum

the end of lactation, the content of NFS grew (Table 2), which corresponds to the reduced values of FP. The peak NFS content value was detected in summer (month 7), which is consistent with the lowest FP value found.

The correlation coefficient between the NFS content and FP is 0.9394. It is statistically highly significant (P = 0.01).

In practice, the determination of milk FP can be used to prove the adulteration of milk with water or, as the case may be, to determine the amount of added water (Bhandari and Singh, 2003). Based on their findings while establishing FP of goat milk in the Ontario province, Szijarto and van de Voort (1983) compiled a table for establishing the amount of added water depending on the FP value.

Table 3 presents the FP values measured in goat milk pasteurized on the farm. Similarly to the raw milk situation, higher FP values in pasteurized milk were detected in the spring months while lower ones in summer and by the end of lactation. Average FP values were −0.5488°C ± 0.0046°C, average monthly values ranged from −0.5400 to −0.5525°C. After the milk was pasteurized at the temperature of 72°C for 20 s, an increase in average FP by 0.0025°C was detected as compared to the FP values in raw milk. The differences in the FP values of pasteurized and raw goat milk in the individual months are presented in Figure 1.

Changes in FP of pasteurized milk correspond to changes in FP of raw milk. The differences between FP values of raw and pasteurized milk assessed by the paired t-test were found to be highly statistically significant (P = 0.01).

Literature data on the effect of heat treatment on FP of milk vary. It can be concluded from the above-mentioned results that changes in FP of pasteurized goat milk correspond to data on changes in FP of pasteurized cow milk. Kessler (1984) found that the FP of cow milk treated with mild pasteurization (30 s at 74°C) did not change but a high temperature of pasteurization (2.8 s at 85°C) increased the FP value by 0.002°C and prolonged pasteurization at the temperature of 95°C (303 s) increased FP by 0.001°C. Rohm et al. (1991) ascribed the increase in milk FP to a change inside the calcium phosphate complex and a change in the pressure of carbon dioxide. This modification is about 0.002°C depending on the temperature and length of heat treatment. Other authors say that the influence of the length of heat treatment on FP is even more significant – pasteurization increases milk FP by 0.006–0.009°C (Singhal et al., 1997). The increase in FP due to heat treatment is almost negligible as

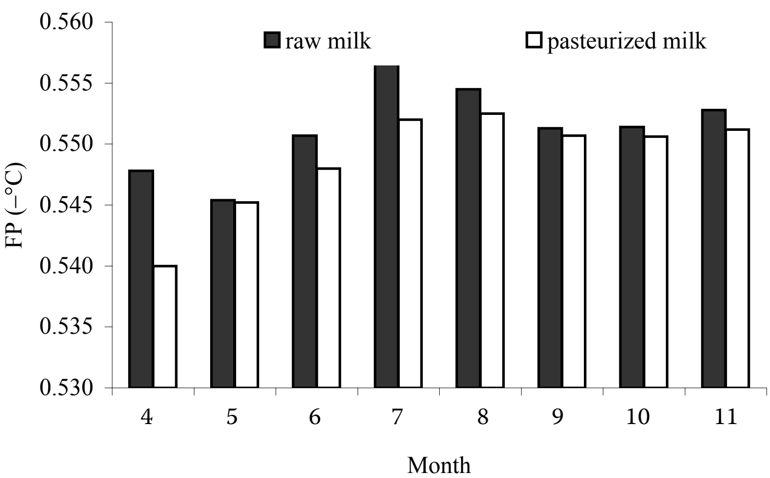

Figure 1. Freezing point of raw and pasteurized goat milk in the course of lactation

compared to the technological adulteration in the primary production and in the dairy plant.

The average value of NFS content in pasteurized milk was 7.93 ± 0.25% (Table 4). This value is by 0.01% higher than the value of NFS content in raw milk but the differences are not statistically significant. Owing to the fact that the detected content of NFS is not a clear proof of potential adulteration with water, the increase in FP is probably caused by changes in milk during heat treatment, as reported by Rohm et al. (1991). The correlation coefficient for FP and NFS in pasteurized milk is 0.4188, which is statistically significant ($P = 0.05$). The minimum value was found in month 4 (7.48 ± 0.01%), the maximum in month 11 (8.17 ± 0.06%).

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