

The freezing point of raw and heat treated sheep milk and its variation during lactation

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Abstract

The freezing point of milk is an important indicator of the adulteration of the milk with water, but heat treatment may also affect its value. The aim of this study was determine freezing point of raw and heat treated sheep milk and its variation during lactation. The freezing point was determined in 42 bulk tank raw sheep milk samples and 42 pasteurized milk samples collected during lactation of sheep at one ecofarm in Moravian Walachia (Valašsko in the Czech Republic). The freezing point was determined in accordance with the standard ČSN 57 0538 using a thermistor cryoscope. The average freezing point of raw milk was -0.617 ± 0.052 °C, with a range from -0.560 to -0.875 °C. The freezing point was lower in the first months of lactation and increased at the end of lactation. The freezing point correlated ($r = 0.8967$) with the content of total non-fat solids. The average freezing point of sheep milk pasteurized at 65 °C for 30 min was -0.614 ± 0.053 °C, with a range from -0.564 to -0.702 °C. The median of freezing point differences between raw and pasteurized milk was 0.004 °C. Our study extends data about physico-chemical properties of sheep milk and registers for the first time specific changes in the freezing point value of sheep milk by heating.

Cryoscopy, physico-chemical properties, pasteurization

Information on the composition and features of sheep milk is crucial to the production of high quality sheep dairy products and to cheese production in particular (Bencini 2002). Sheep milk has a higher content of solids than goat and cow milk but, on the other hand, it has a lower freezing point (Park et al. 2007). The milk composition and features vary with the stage of lactation, nutrition, breed, and animal health status (Park et al. 2007; Kuchtik et al. 2008). Freezing point (FP) is an important feature of milk. Of the milk constituents, lactose and chlorides, i.e. substances that form solution in milk, are the major contributors to FP (Fox and McSweeney 1998). Their contents fluctuate depending on, beside the factors mentioned above, the water intake, heat stress, season and CO₂ content in milk (Rohm et al. 1991; Antunac et al. 2001; Slaghuis 2001). The effect of protein content is not important, although some authors mention it (Macek et al. 2008), and milk adulteration with extraneous water, added either intentionally or accidentally (Zee et al. 1982; Buchberger 1996). The freezing point of raw sheep milk is lower than those of cow and goat milk (Maurer and Schaeren 2007) because of the higher solids and non-fat solids contents in sheep milk. The average reported sheep milk FP is -0.570 °C (Park et al. 2007). Freezing point value of heat-treated milk is dependent on the value of FP of raw milk and the temperature of heat treatment, which causes an increase in FP in the range from 0.001 to 0.009 °C as observed by Kessler (1984) and Singhal et al. (1997). Rohm et al. (1991) have reported a freezing point about 0.002 °C depending on the temperature and heating time: the increase is a result of the changes in the calcium phosphate complex and carbon dioxide pressure.

The aim of this study was to determine freezing points of raw and pasteurized sheep milk and their variation during lactation and to assess the effect of pasteurization on freezing points.

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Materials and Methods

Raw and pasteurized sheep milk samples were collected on an ecofarm in Moravian Walachia in the Czech Republic. About 100 ewes of Lacaine cross breed, East Friesian and Improved Walachian sheep are bred on this farm. Raw sheep milk was sampled twice monthly from May to November 2011. Unpreserved milk samples were transported in an insulated cool box at 6 °C. In total, 42 samples of raw sheep milk (3 per month) and 42 samples of the same sheep milk after pasteurization (3 per month) were collected. Milk was pasteurized at 63 °C for 30 min on the farm.

The average composition (in %) of raw milk was the following: protein 6.73 ± 1.689 , fat 8.68 ± 2.046 , lactose 4.58 ± 0.715 , solids 20.31 ± 3.393 and non-fat solids 12.06 ± 1.002 . The average composition (in %) of pasteurized milk was the following: protein 6.82 ± 1.109 , fat 8.71 ± 2.024 , lactose 4.62 ± 0.612 , solids 20.53 ± 3.343 , and non-fat solids (NFS) 12.18 ± 1.131 . The milk composition was determined in raw and pasteurised milk in accordance with the standard ČSN 570536 (1999) using a Bentley 2500 infrared analyzer (Bentley Instruments, Minnesota, USA) in the laboratory for milk analysis, Brno.

The freezing point of milk was determined in accordance with the standard ČSN 57 0538 (1998) "Determination of freezing point of milk by the cryoscopic method using" a Funke Gerber Cryostar Automatic 7160 (Funke Gerber, Germany). The thermistor cryoscope was calibrated on a regular basis using standard solutions FB -0.408 °C and -0.600 °C, and Lactrol reference solution (-0.512 °C).

The measurement uncertainty was 0.61%: it is a combined uncertainty at a confidence level $U = 95\%$ for a coverage factor $k = 2$, which indicates approximately 95% confidence level. Basic statistical analyses were performed using Unistat 5.1 software – the significance of differences P and median differences between FP raw and pasteurized milk (Unistat Ltd. 1998).

Results

The monthly average FP values of raw sheep milk during the study period varied from -0.584 °C to -0.724 °C and the average FP for the whole study period was -0.617 °C \pm 0.052 °C (Table 1).

At the beginning of the study period (month 5), the average FP was -0.584 ± 0.010 °C while in the summer (month 8), it decreased to -0.625 °C. At the end of lactation (month 10), FP decreased to -0.637 °C. Grazing season ended at the beginning of November (month 11). In those last days of lactation a significant decrease of FP (-0.724 °C) was recorded as a result of reduced milk production, which corresponded to the increased dry matter content. The average NFS content of raw sheep milk was $12.06 \pm 1.002\%$, ranging from 11.20 to 13.69%. Freezing point correlated ($r = 0.8967$) with the non-fat solids content ($P = 0.05$).

Table 1. Average freezing points of raw sheep milk during the study period (°C).

Statistical evaluation	Month*	5	6	7	8	9	10	11	Whole study period
\bar{x}		-0.584	-0.594	-0.572	-0.625	-0.586	-0.637	-0.724	-0.617
SD		0.006	0.026	0.011	0.025	0.006	0.021	0.108	0.052
min		-0.588	-0.619	-0.582	-0.672	-0.579	-0.669	-0.875	-0.875
max		-0.579	-0.560	-0.560	-0.578	-0.588	-0.565	-0.573	-0.560

\bar{x} - average, SD - standard deviation, min - minimum, max - maximum, *May–November

Table 2. Average freezing points of pasteurized sheep milk during study period (°C).

Statistical evaluation	Month*	5	6	7	8	9	10	11	Whole study period
\bar{x}		-0.581	-0.591	-0.568	-0.622	-0.571	-0.622	-0.720	-0.614
SD		0.014	0.013	0.007	0.020	0.006	0.018	0.048	0.053
min		-0.574	-0.577	-0.567	-0.624	-0.580	-0.625	-0.702	-0.702
max		-0.564	-0.564	-0.564	-0.592	-0.565	-0.591	-0.665	-0.564

\bar{x} - average, SD - standard deviation, min - minimum, max - maximum, *May–November

Table 2 displays the FP values determined in sheep milk pasteurized at 63 °C for 30 min which corresponds to pasteurization at 72 °C for 15 s (Regulation EC No 853/2004).

The average FP of pasteurized sheep milk per study period was $-0.614 \text{ °C} \pm 0.053 \text{ °C}$ and the average monthly FP ranged from -0.564 °C to -0.720 °C . The average NFS content of pasteurized sheep milk was $12.18 \pm 1.131\%$ and was by 0.01% higher than the NFS content of raw sheep milk, but the difference was not significant ($P > 0.05$).

The dynamics of FP changes in pasteurized milk during the lactation period follow the pattern of those in raw sheep milk. The differences between FPs of raw and pasteurized sheep milk determined by paired *t* test were highly significant ($P = 0.01$). After pasteurization, the average FP increased by 0.004 °C compared to raw milk (median of differences between FP raw and pasteurized milk).

Discussion

No literature data on the FP variation in sheep milk induced by heat treatment have been available, but from the above mentioned results it follows that the dynamics of the FP variation in the pasteurized sheep milk should follow the patterns observed for the pasteurized cow and goat milk.

The average FP of raw sheep for every month and for the whole study period corresponded to the findings of other authors. The following freezing points were found: -0.564 °C to -0.570 °C (Pavić et al. 2002), -0.568 °C (Petrova and Nedelchev 2002), -0.570 °C (Park et al. 2007), -0.6048 °C (Macek et al. 2008) and -0.560 °C to -0.610 °C (Keresteš 2008).

The different results document seasonal variation of FP of raw sheep milk depending on the stage of lactation, climate change and nutrition. The average value of FP in our study decreased in the summer, most probably as a result of high temperatures in the surrounding environment and short-term dehydration of the animals, as also reported by Bjerg et al. (2005). Early in month 11 when the grazing season ended with reduced milk production, FP value was the lowest, corresponding to a higher content of solids also reported by Kuchtík et al. (2008). Nevertheless, this minimal FP markedly influenced the average FP during lactation, and therefore, the average FP in our study is lower than reported by others (Pavić et al. 2002; Petrova and Nedelchev 2002; Park et al. 2007; Macek et al. 2008; Keresteš 2008). Nevertheless, the average FP of -0.601 °C calculated for the period of mature milk production (months 5 to 10) is consistent with their data. In practice, the determination of milk FP is helpful primarily in detecting milk adulteration with water (Bhandari and Singh 2003). For instance in Bulgaria, the limit of -0.585 °C was set for FP of sheep milk (Keresteš 2008).

Significantly higher contents of NFS raw sheep milk were found, clearly exceeding the 9.5% required by the standard STN 57 0510/1993 and the 11.45% reported by Pavić et al. (2002).

As the determined NFS content is not suggestive of milk adulteration with water, the increased FP seems to be a result of milk changes after heat processing, as reported by Rohm et al. (1991).

Kessler (1984) has found that FP of cow's milk remains the same after pasteurization at 74 °C for 30 s but increases by 0.002 °C after high pasteurization at 85 °C for 2.8 s. Other authors have found the heat treatment to have a more substantial impact on FP of milk: FP increases by 0.006 to 0.009 °C after pasteurization and by 0.023 °C after ultra high temperature (UHT) treatment as reported by Singhal et al. (1997). Anyway, the effect observed, i.e. increase in FP of pasteurized milk, is a result of milk protein response to the heat shock (Rohm et al. 1991), and, at the same time, of water removal from milk by evaporation, with the milk becoming thicker. Janštová et al. (2009) found FPs of cow and

goat milk to increase by 0.0023 °C and 0.0028 °C, respectively, as a result of pasteurization at 72 °C for 20 s in comparison with raw milk; a lower FP of sheep milk (by 0.003 °C) was expected, given the higher NFS content and lower FP of raw milk.

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