

Differential Scanning Calorimetry Analysis of Goat Fats: Comparison of Chemical Composition and Thermal Properties

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Abstract The physical–chemical properties, fatty acid composition and thermal properties of goat subcutaneous (SF), tallow (TF) and intestinal (IF) fats were determined. SF differed from other fat types with respect to its lower melting (41.6 °C), lower saponification (190.3 mg KOH/g) and higher iodine (40.4) values as compared to those of other fats. Goat fat types contained palmitic acid (C_{16:0}), stearic acid (C_{18:0}), oleic acid (C_{18:1 ω 9}) and linoleic acid (C_{18:2 ω 6}) as the major components of the fatty acid composition (23.06–23.52, 22.95–39.03, 21.94–36.16 and 1.96–2.22%, respectively). A differential scanning calorimetry (DSC) study revealed that two characteristic peaks were detected in both crystallization and melting curves. Major peaks (T_{peak}) of TF and IF were similar and determined as 34.02–35.24 and 9.95–10.72 °C, respectively for the crystallization peaks and 15.11–18.26 and 50.70–52.76 °C, respectively for the melting peaks in the DSC curves; but those of SF (27.14 and 4.36 °C for crystallization peaks and 8.39 and 44.93 °C for melting peaks) differed remarkably from those of other fat types.

Keywords Goat fats · Chemical composition · Fatty acid profile · Differential scanning calorimetry

Introduction

The physical and chemical characteristics of fat have been an important aspect of the evaluation, classification, and

sensory properties of meat. For instance, soft fat is a critical factor decreasing the grade of a carcass in meat grading in some countries [1]. Soft fat is characterized by its low melting point, which results in low processing properties. On the other hand, a low melting point of fat in foods has been generally considered to relate positively to the sensory properties, especially mouth feel [2].

In goat and other muscle foods, the melting properties of fat have generally been analyzed by the glass-capillary tube method [1, 3, 4]. However, DSC is a common acceptable method for determining the crystallization and melting characteristics of fats. DSC can be used for the determination of the phase transitions of fats [5, 6].

In countries such as Turkey which has a great animal fat capacity, the fat is not used effectively in the food or confectionary industries. However, cocoa, butter, palm, coconut and palm kernel oils are imported and used as edible oils whose usage in food and confectionary products has been increasing steadily over the last few years [7]. It is therefore important to investigate the suitability of other fat sources such as edible goat fats as replacements for more expensive fats and oils in the food and confectionary industries.

The relationship between fatty acid composition and the DSC properties of a fat has been pointed out in the literature [8, 9]. The fundamental differences between fat types with respect to their fatty acid composition could be a basis for the detection of these fats in some further processed meat products using a melting thermogram of DSC. Therefore, the physical characteristics of fats such as their melting temperatures should be investigated based on their fatty acid composition. In addition, the chemical and thermal data to be obtained in this way may be beneficial to future research, which will be required for the nutritional, energy and technological properties of goat fats to develop feed or food

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products containing these fat types for agricultural (as a soap stock in animal feed) or food production purposes, respectively. However, there are a very limited number of studies in which goat fat types were analyzed and compared with each other in respect to their physical–chemical properties and fatty acid composition. Furthermore, no study has appeared which evaluates the thermal properties of goat fats, to compare them with each other with regard to these properties and to relate these thermal properties to their chemical and fatty acid composition. Therefore, this study was undertaken to investigate the potential use of crystallization and melting curves of DSC for the evaluation of the chemical and physical properties of goat fats and present the basic physical–chemical and compositional differences between the goat fat types studied.

Materials and Methods

Samples

Subcutaneous, tallow and intestinal fat adipose tissues were obtained from Turkish hair goat (*Capra hircus*, 30 months of age) fed at the Konet Slaughterhouse and Meat Products Processing Co., Konya, Turkey. Subcutaneous fat tissue samples (SF) were obtained from the areas under the hide and carcass surfaces. The outer layer of the fat tissue was used for samples. Tallow samples (TF) were collected from the fat tissues surrounding tripe. Intestinal fat tissue (IF) was sampled from the fatty tissues holding the intestines together. After slaughtering the animals, all the fat tissue samples were immediately transferred to the laboratory in ice boxes. The samples were kept in a deep freeze at a temperature of $-20\text{ }^{\circ}\text{C}$ during the preparation of the fat samples. After being kept in a refrigerator ($4\text{ }^{\circ}\text{C}$) until constant temperature had been reached, the fatty tissues were ground twice through a 3-mm plate. The fat samples were prepared by melting the related adipose tissues at $60\text{ }^{\circ}\text{C}$ in a water bath. Then, the samples were strained through a porous cloth and used without further purification. The analyses were conducted on the extracted fat samples. These fat samples were transferred into glass containers and stored at $-20\text{ }^{\circ}\text{C}$ until analysis.

Proximate Analysis

Moisture, fat and total nitrogen contents of the fat tissues were determined using standard methods of the AOAC [10].

Melting Point

Capillary tube methodology was used to determine the melting point (MP) of the fat samples. Samples were

allowed to enter from the bottom capillary tubes and rise to a height of 10 mm and then frozen. Frozen samples were attached near to the bulb-end of a mercury thermometer and subsequently submerged in a temperature-controlled water bath, the temperature of which could be increased at a rate of $1\text{ }^{\circ}\text{C}/\text{min}$. The MP was recorded as the temperature at which the fat became transparent [11].

Saponification Number

Saponification (SN) values of the fat samples were determined as outlined in Ref. [11]. The fat samples were saponified with a 0.5-N KOH solution by boiling 60 min on a hot plate and attached to a water-cooled reflux condenser. The saponified solution was titrated with 0.5 N HCl until the brilliant color point. The same procedure was repeated for a blank. The SN was calculated as follows:

$$\begin{aligned} \text{SN (mg KOH / g fat)} \\ &= \frac{[\text{HCl (ml) for sample} - \text{HCl (ml) for blank}] \times \text{N (0.5)} \times 56.1}{\text{sample amount (g)}} \end{aligned}$$

Iodine Values

The Wijs method was used to determine the iodine values (IV) of the fat samples [11]. A fat sample (0.6 g) was dissolved in a flask containing 15 ml CCl_4 , followed by the addition of 25 ml of Wijs solution. After being covered and agitated, the flasks were kept in a dark place for 1 h. Then 20 ml of 10% of KI, 150 ml of distilled water and 1 ml of starch solution were added to the flask. The solution in the flask was subsequently titrated with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the point it became colorless. The same procedure was repeated for the blank. The IV was calculated as follows:

$$\begin{aligned} \text{IV} \\ &= \frac{[\text{Na}_2\text{S}_2\text{O}_3 \text{ (ml) for sample} - \text{Na}_2\text{S}_2\text{O}_3 \text{ (ml) for blank}] \times 1.269}{\text{sample amount (g)}} \end{aligned}$$

Energy Values

The energy values of the fat samples were determined using a bomb calorimeter (IKA, C200, Germany). Approximately $0.1\text{--}0.2 \pm 0.0001$ g of the fat sample was weighed into a burning crucible placed in the decomposition vessel of the instrument. To optimize the combustion process, the decomposition vessel was filled with pure oxygen (99.95%) until the pressure of oxygen atmosphere reached a maximum of 30 bar. The fat sample was ignited by a cotton thread as an ignition aid. The temperature increase in the calorimeter system was measured and the specific calorific value of the fat sample was calculated as follows [12]:

$$H_0 = [C \times \Delta T - (Q_{\text{External1}} + Q_{\text{External2}})]/m$$

where m was the weight of fat sample, C was the heat capacity (C -value), ΔT was the calculated temperature increase of the water in the inner vessel of the measuring cell, $Q_{\text{External1}}$ was the correction value for the heat energy generated by the cotton thread an ignition aid and $Q_{\text{External2}}$ was the correction value for the heat energy from other burning aids.

Fatty Acid Analysis

Fat samples were methylated by the boron trifluoride–methanol method of Yazıcıoğlu and Karaali [13] and the FAME obtained were analyzed using a gas chromatograph (Shimadzu, GC-2010, Shimadzu Instrument Ltd., Kyoto, Japan) fitted with an FID detector. A polar silica fused capillary column TR-CN100 (0.25-mm i.d., 60-m length and 0.20- μm film thickness; Teknokroma Ltd., Barcelona, Spain) was used at a pressure of 200 kPa. The temperature of the column was programmed to increase to 90 °C for 7 min and 240 °C at 5 °C/min for 15 min. The temperature of the injector and detector was maintained at 260 °C. Nitrogen was used as carrier gas at a flow rate of 1.51 ml/s. The fatty acids methyl esters were identified by comparing the retention time of the samples with appropriate fatty acid methyl ester standards, purchased from Supelco, USA. The relative percentage of the area was obtained by using the following equation: $\text{Area \% FA}_X = [A_X/A_R] \times 100$, where: FA_X = fatty acid to be quantified, A_X = area of the methyl ester X and A_R = total area of the chromatogram. Peak areas lower than 0.1% of the total area were not considered.

Thermal Analysis by DSC

The DSC system (Shimadzu DSC-50, Shimadzu Instrument Ltd., Kyoto, Japan) was used to record the crystallization and melting curves of the fat types. The instrument was calibrated with indium and mercury for heat flow as outlined in Ref. [14]. The fat samples (10 mg) were weighed into aluminum hermetically sealable pans and covers were crimped into place. An empty, hermetically sealed aluminum pan was used as a reference. The measuring cell was purged with nitrogen gas (30 ml/min) during analysis. The fat samples were subjected to the following temperature program: (a) heated to 75 °C to destroy the crystal structure; (b) cooled at a rate of 5 °C/min from 75 to –65 °C using liquid nitrogen; (c) heated from –65 to 75 °C at 5 °C/min. The crystallization and melting characteristics of each sample i.e., extrapolated onset temperature (T_o), maximum peak temperature (T_p) and

enthalpy (ΔH) were obtained using a Shimadzu TA-50I data processor.

Statistical Analysis

Statistical analysis was carried out by the General Linear Model (GLM) procedure using the SPSS [15] with one-way allocation. Significant differences between the groups were further analyzed using the Duncan's Multiple Range Test of the MSTATC program [16].

Table 1 Proximate composition of goat fat tissues and physical–chemical properties and fatty acid composition of goat fat types

Analyses	Fat tissue and fat types		
	Subcutaneous	Tallow	Intestinal
Proximate composition of fat tissues			
Moisture (%)	15.5 ± 1.3 ^a	9.03 ± 0.9 ^b	6.7 ± 0.4 ^c
Fat (%)	81.1 ± 1.3 ^b	86.4 ± 3.2 ^a	84.3 ± 1.8 ^{ab}
Total nitrogen (%)	3.75 ± 0.07 ^a	1.60 ± 0.00 ^b	1.65 ± 0.07 ^b
Physical and chemical properties of fat types			
MP (°C)	41.6 ± 0.9 ^b	48.6 ± 0.5 ^a	49.5 ± 1.1 ^a
SN (mg KOH/g fat)	190.3 ± 8.3 ^b	200.6 ± 6.9 ^b	221.2 ± 4.2 ^a
IV	40.4 ± 1.6 ^a	34.4 ± 1.4 ^b	31.0 ± 0.8 ^c
EV (kcal/100 g)	984 ± 11 ^a	984 ± 11 ^a	952 ± 14 ^b
Fatty acid composition of fat types (%)			
C _{12:0}	0.11 ± 0.01 ^a	0.11 ± 0.01 ^a	0.13 ± 0.01 ^a
C _{14:0}	3.06 ± 0.04 ^a	2.34 ± 0.01 ^b	2.51 ± 0.06 ^b
C _{14:1ω5}	0.90 ± 0.01 ^a	0.40 ± 0.16 ^b	0.72 ± 0.01 ^a
C _{15:0}	0.41 ± 0.00 ^a	0.24 ± 0.01 ^b	0.27 ± 0.01 ^b
C _{16:0}	23.06 ± 0.11 ^a	21.53 ± 0.75 ^a	23.52 ± 0.86 ^a
C _{16:1ω7}	1.88 ± 0.23 ^a	0.47 ± 0.13 ^b	0.50 ± 0.06 ^b
C _{17:0}	0.73 ± 0.00 ^a	0.62 ± 0.03 ^a	0.65 ± 0.08 ^a
C _{17:1}	1.72 ± 0.00 ^a	1.50 ± 0.04 ^b	1.72 ± 0.06 ^a
C _{18:0}	22.95 ± 0.30 ^b	38.18 ± 0.37 ^a	39.03 ± 0.50 ^a
C _{18:1ω9}	36.16 ± 0.39 ^a	23.51 ± 0.00 ^b	21.94 ± 0.55 ^b
C _{18:2ω6}	1.96 ± 0.08 ^a	2.22 ± 0.25 ^a	2.21 ± 0.47 ^a
C _{18:3ω3}	0.35 ± 0.01 ^a	0.15 ± 0.07 ^a	0.41 ± 0.24 ^a
C _{20:0}	0.24 ± 0.01 ^a	0.16 ± 0.01 ^a	0.24 ± 0.06 ^a
C _{20:1ω6}	0.96 ± 0.00 ^a	0.74 ± 0.30 ^a	0.89 ± 0.53 ^a
Σ SFA	50.55 ± 0.23 ^c	63.17 ± 0.40 ^b	66.34 ± 0.40 ^a
Σ MUFA	41.60 ± 0.62 ^a	26.60 ± 0.57 ^b	25.75 ± 1.09 ^b
Σ PUFA	2.30 ± 0.06 ^a	2.38 ± 0.33 ^a	2.62 ± 0.23 ^a
Σ UFA	43.90 ± 0.69 ^a	28.98 ± 0.90 ^b	28.36 ± 1.33 ^b
UFA/SFA ratio	0.87 ± 0.01 ^a	0.46 ± 0.01 ^b	0.43 ± 0.02 ^b

In each row, mean (\pm SD) values with different superscripts indicate significant differences ($P < 0.01$)

MP melting point, SN saponification value, IV iodine value, EV energy value, SFA saturated fatty acids = C_{14:0} + C_{15:0} + C_{16:0} + C_{17:0} + C_{18:0} + C_{20:0}, MUFA monounsaturated fatty acids = C_{14:1 ω 5} + C_{16:1 ω 7} + C_{17:1} + C_{18:1 ω 9} + C_{20:1 ω 6}, PUFA polyunsaturated fatty acids = C_{18:2 ω 6} + C_{18:3 ω 3}, UFA unsaturated fatty acids = MUFA + PUFA

Results and Discussion

Proximate Composition of Fat Tissues

Table 1 indicates that significant ($P < 0.01$) differences were found between the fat tissues, namely, subcutaneous, tallow and intestinal fat tissues with respect to their

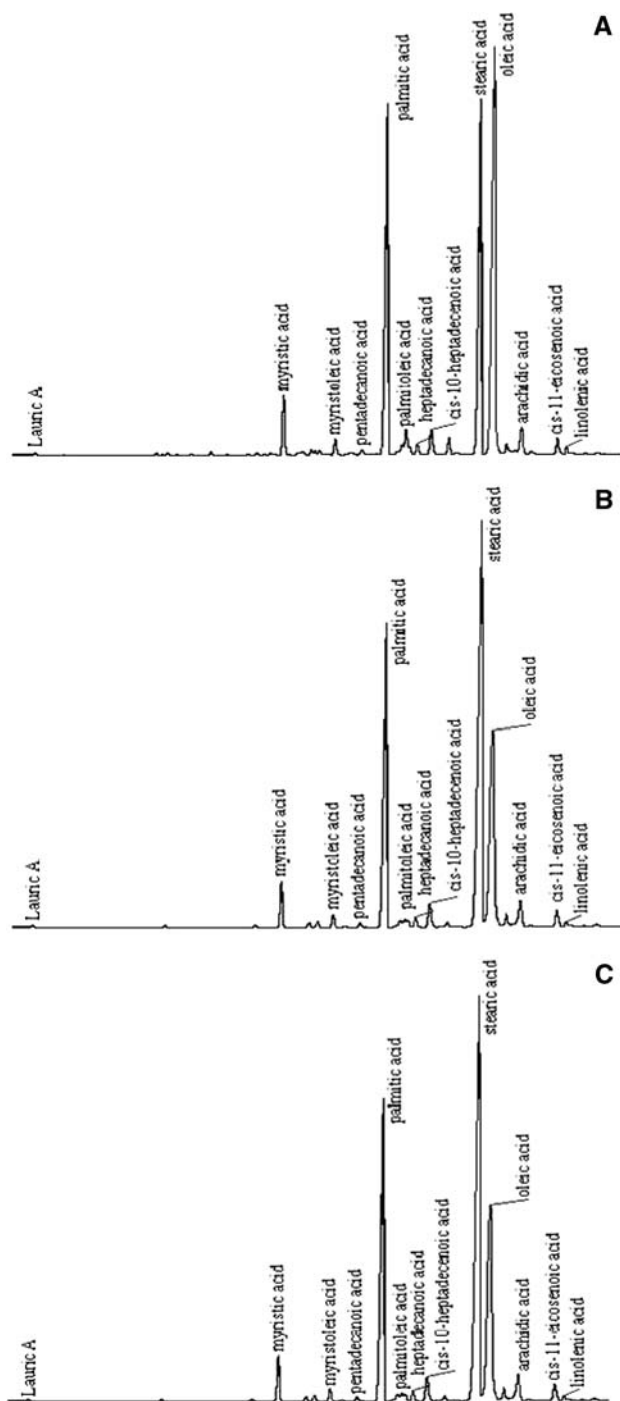


Fig. 1 Fatty acid chromatograms of subcutaneous (a), tallow (b) and intestinal (c) fats of goat

proximate composition. As can be seen in the table, the moisture and total nitrogen contents of the subcutaneous tissue were higher, but the fat content of this tissue was lower than those of tallow and intestinal tissues. Based on these results, the subcutaneous fat tissue could be regarded as a distinct fat tissue among the goat fat tissues.

Physical and Chemical Properties of Fat Types

The physical and chemical properties of goat fat types are also shown in Table 1. MP and SN values of subcutaneous fat (SF) were found to be lower than those of tallow fat (TF) and intestinal fat (IF), while the IV value of SF was higher than those of TF and IF, which indicated that SF was a distinct fat among the fat types with respect to these physical and chemical properties. As far as fats of animal origin are concerned, this exceptional trait of SF could be important for human nutrition and health.

The results for MP and SN values of TF and IF were similar to those of Swern [17] who determined these value to be 46.2 °C and 199 mg KOH/g fat, respectively for goat suet fats. SN of IF was found higher than those of SF and TF, which indicated that the molecular weight of IF was lower than those of other fat types. IV, a measure of the unsaturation degree of a fat, also varied between fat types. The IV of SF was higher than that of other fat types, which indicates that the unsaturation degree of SF was higher than those of the others. The IV results for TF and IF as goat suet fats were similar to those of Swern [17] who reported the IV of goat suet to be 33.5/100 g.

The energy values of fat types were determined and ranged between 952 and 984 kcal/100 g. However, no remarkable difference was observed between the energy values of fat types, which indicates that all fat types could provide a similar energy level for human metabolism.

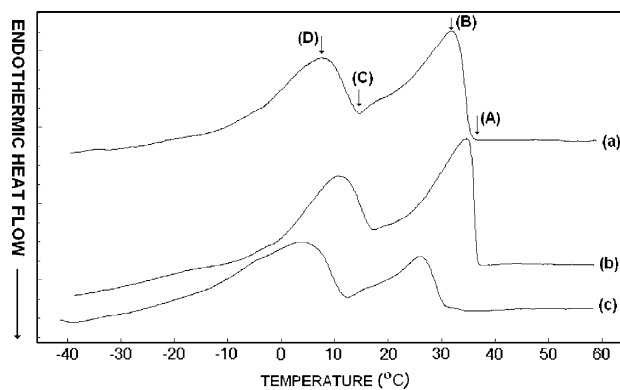


Fig. 2 DSC crystallization curves indicating endothermic heat flow for: goat (a) tallow; (b) intestinal and (c) subcutaneous fats. A, B, C, and D indicate ‘first onset temperature’, ‘first peak temperature’, ‘second onset temperature’, ‘second peak temperature’, respectively

Table 2 T_{onset} , T_{peak} and ΔH values obtained from DSC crystallization peaks of goat fat types

Fat types	First crystallization peak			Second crystallization peak		
	T_{onset} (°C)	T_{peak} (°C)	ΔH (J/g)	T_{onset} (°C)	T_{peak} (°C)	ΔH (J/g)
Subcutaneous	30.59 ± 0.16 ^b	27.14 ± 1.73 ^b	–	11.42 ± 0.28 ^b	4.36 ± 0.78 ^b	–
Tallow	37.02 ± 0.47 ^a	34.02 ± 0.74 ^a	–	16.13 ± 0.69 ^a	9.95 ± 0.44 ^a	–
Intestinal	37.53 ± 1.24 ^a	35.24 ± 1.00 ^a	–	16.69 ± 0.09 ^a	10.72 ± 0.23 ^a	–

In each column, mean (±SD) values with different superscripts indicate significant differences ($P < 0.01$)

Fatty Acid Composition of Fat Types

Chromatograms and fatty acid profiles of goat fat types are shown in Fig. 1 and Table 1, respectively. The fat types had similar amounts of palmitic acid ($C_{16:0}$); however, stearic ($C_{18:0}$) and oleic acid ($C_{18:1\omega9}$) contents of SF were different ($P < 0.01$) from those of TF and IF. SF fat had lower $C_{18:0}$, but higher $C_{18:1\omega9}$ contents than did TF and IF. In addition, SF was the fat type having the highest ($P < 0.01$) myristic ($C_{14:0}$), myristoleic ($C_{14:1\omega5}$), pentadecanoic ($C_{15:0}$) and palmitoleic acid ($C_{16:1\omega7}$) contents. Total saturated fatty acids (SFA) were lower ($P < 0.01$), but total monounsaturated (MUFA) and total unsaturated fatty acids (UFA) were higher in SF than those in TF and IF. On the other hand, no remarkable difference was found among these fat types with respect to the other fatty acids. According to the above results, SF was quite different from TF and IF because of the difference between these fat types in respect to their fatty acid composition.

The results for the fatty acid profile of goat subcutaneous fat were, to some extent, different from those reported by other researchers. Mahgoub et al. [18] reported that lauric acid, myristic acid, palmitic acid, palmitoleic acid, heptadecanoic acid, stearic acid, oleic acid, linoleic acid, linolenic acid and arachidic acid contents of goat subcutaneous fat were 0.84, 9.22, 20.69, 9.65, 5.28, 16.06, 27.40, 6.70, 0.35, and 0.40%, respectively. Differences between these values in the literature and those determined in our study could be attributed to the fact that the fatty acid composition is affected by breed, sex, age and nutritional conditions [7, 19]. On the other hand, the results for SFA, MUFA, PUFA, UFA contents and UFA/SFA ratio in goat subcutaneous fat in this study were similar to those of Mahgoub et al. [18] who determined these values to be 55.37, 37.38, 6.90, 44.63 and 0.82%, respectively.

DSC Analysis of Fat Types

Figure 2 shows the DSC crystallization curves of goat fat types. As can be seen in Fig. 2, two major exothermic peaks were observed between approximately -10 and 40 °C. T_{onset} , T_{peak} , and ΔH values of those peaks are shown in Table 2. As can be seen in the table, the lowest

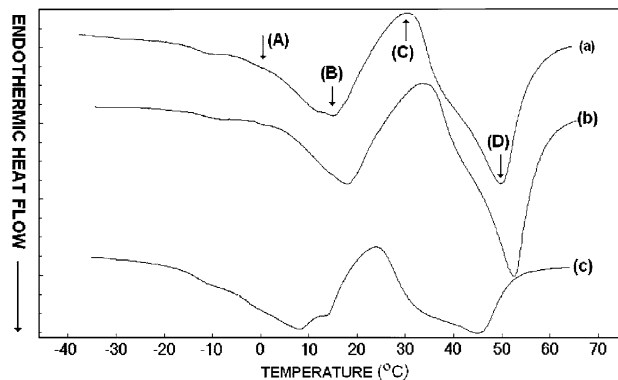


Fig. 3 DSC melting curves indicating endothermic heat flow for: goat (a) tallow; (b) intestinal and (c) subcutaneous fats. A, B, C, and D indicate ‘first onset temperature’, ‘first peak temperature’, ‘second onset temperature’, ‘second peak temperature’, respectively

values for T_{onset} and T_{peak} were determined in SF. The lowest values of T_{onset} and T_{peak} could be related to the highest percentage of oleic acid ($C_{18:1\omega9}$, 36.16%), the lowest percentage of stearic acid ($C_{18:0}$, 22.95%) and the highest unsaturated fatty acid ($\sum \text{UFA} = 43.90\%$) contents in SF. The differences between SF and other fat types, namely, TF and IF with respect to these values might be attributed to the physical properties of triglycerides in these fat types. Sato [20] reported that the fats and lipids present in natural resources are mixtures of different types of triglycerides, and therefore the complicated behavior of the crystallization of the real-fat systems could be to partly due to the physical properties of the triglycerides.

Figure 3 shows the DSC melting curves. The results showed that two major endothermic peaks were observed. On the other hand, no another peak was found to exist in the temperature region beyond -12.49 °C because the majority of the triglycerides present in goat fat types were high-melting ones. T_{onset} , T_{peak} and ΔH values of these peaks are shown in Table 3. As can be seen in the table, the lowest T_{onset} and T_{peak} values were determined for the first and second peaks of SF. The lowest ΔH value was also determined in the second peak of SF. Accordingly, the DSC results for SF were in line with the MP and IV values of goat fat types because SF had the lowest MP and highest IV values among the goat fat types (Table 1).

Table 3 T_{onset} , T_{peak} and ΔH values obtained from melting peaks of different goat fat types

Fat types	First melting peak			Second melting peak		
	T_{onset} (°C)	T_{peak} (°C)	ΔH (J/g)	T_{onset} (°C)	T_{peak} (°C)	ΔH (J/g)
Subcutaneous	-12.49 ± 0.71^c	8.39 ± 0.48^c	56.91 ± 0.24^a	25.30 ± 0.25^c	44.93 ± 0.09^b	50.77 ± 2.43^b
Tallow	0.57 ± 0.07^b	15.11 ± 0.13^b	46.20 ± 0.89^b	32.50 ± 0.11^b	50.70 ± 1.26^a	73.06 ± 1.30^a
Intestinal	4.27 ± 0.44^a	18.26 ± 0.46^a	47.78 ± 0.38^b	36.36 ± 0.57^a	52.76 ± 0.36^a	79.26 ± 0.85^a

In each column, mean (\pm SD) values with different superscripts indicate significant differences ($P < 0.01$)

The higher T_{onset} and T_{peak} of TF and IF as compared to that of SF could be attributed to their higher percentage of the saturated stearic acid ($C_{18:0} = 38.18$ and 39.03% , respectively) and SFA (\sum SFA = 63.17 and 66.34% , respectively). De Man [8] reported that usually highly saturated triglycerides demonstrate a higher melting point than those which are highly unsaturated. Differences between onset and peak temperatures were thought to be due to the physical properties of triglycerides. It was reported that the behavior of melting of the real-fat systems could be somewhat result from the physical properties of the triglycerides [20]. Accordingly, higher T_{onset} and T_{peak} values for TF and IF was thought to confirm that the main components in these fat types were triglycerides with saturated fatty acids, which melted at high temperatures. Hence, this group of triglycerides could be categorized as high-melting ones. On the other hand, DSC heating profiles of SF were found to show a low-melting endothermic peak at 44.93 °C. Therefore, this lower peak value could be a distinct property of SF for detecting and discriminating this fat type from TF and IF using melting chromatograms of DSC.

Conclusion

Tallow and intestinal fats are higher melting fats. Therefore, these fats could be categorized as solid glycerides which would be useful in any application where high-melting glycerides are required. In addition, these types of fats are similar to the hydrogenated fats with respect to their melting characteristics, which make them useful as a hardening fat in shortening and margarine formulations. The compositional and thermal properties of subcutaneous fat were different from those of the tallow and intestinal fats, which put subcutaneous fat into a distinct place in respect to some further technological processes where melting characteristics are of primary concern. In addition, the results of this study could be a basis for a further study where some food or fat adulteration will be assessed to monitor these goat fat types.

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