


Original article

Crystallization Behavior of High Oleic Sunflower Oil, Palm Super Olein, and Refined Pomace Oil Determined by DSC Cooling Thermograms

Didar Üçüncüođlu *

Çankırı Karatekin University, Faculty of Engineering, Food Engineering Department, Çankırı, Türkiye

Abstract

High oleic sunflower oil (HOSO), palm super olein (PSO), and refined pomace oil (RPO) are widely used in food processing. This study compared their crystallization behavior under identical differential scanning calorimetry (DSC) conditions. Thermal transitions were measured between +30 °C and -80 °C. Distinct cooling thermograms were obtained for each oil. Two crystallization peaks were detected for HOSO with onset temperatures of -18.09 °C and -40.49 °C and enthalpy values of 0.72 and 47.28 J.g⁻¹. Three peaks were detected for RPO at -12.72 °C, -30.77 °C, and -45.51 °C with enthalpy values of 2.43, 0.39, and 11.76 J.g⁻¹. PSO showed a dominant crystallization event at 0.08 °C with an enthalpy of 23.64 J.g⁻¹ and two additional transitions at -22.24 °C and -54.98 °C. DSC cooling thermograms differentiated the refined oils based on crystallization onset temperature and enthalpy values. Results indicate that DSC cooling thermograms can support characterization and authenticity assessment of refined edible oils.

Keywords: Palm Super Olein Oil, High Oleic Sunflower Oil, Pomace Oil, DSC, Cooling Thermograms, Crystallization

Received: 03 September 2025 * **Accepted:** 20 February 2026 * **Published:** 27 May 2026

Corresponding author:

Didar Üçüncüođlu is an Assistant Professor in the Department of Food Engineering, Faculty of Engineering at Çankırı Karatekin University in Çankırı, Türkiye. Her research interests include Food Science, Quality Control Inspections and Edible oil Technology.
Email: didaru@karatekin.edu.tr

INTRODUCTION

Edible vegetable oils are essential in global food systems. They are widely used for frying, baking, emulsified foods, spreads, and confectionery, and they act as carriers of fat-soluble bioactive compounds and flavors (Kanekanian, 2005). Global vegetable oil production is dominated by palm, soybean, rapeseed, and sunflower oils (Murphy, 2025). In the 2024/25 season, palm oil production exceeded 79 million tonnes. Soybean oil reached nearly 69 million tonnes, rapeseed oil approximately 34 million tonnes, and sunflower oil 20 million tonnes according to the United States Department of Agriculture (USDA, 2024). Olive oil production is considerably smaller but remains nutritionally and culturally significant. Global output reached 2.8 million tonnes according to the Food and Agriculture Organization of the United Nations and the International Olive Council (FAO, 2024; IOC, 2023). Most edible oils are refined before entering the market to ensure oxidative stability, safety, and acceptable sensory characteristics (Čmolík & Pokorný, 2000). Refining typically includes degumming, neutralization, bleaching, and deodorization. These processes remove free fatty acids, pigments, and undesirable compounds and improve shelf life. Fatty acid composition and thermal stability strongly influence the selection of oils for specific food applications (Kanekanian, 2005). Palm super olein (PSO) is widely used in frying due to its resistance to oxidative degradation at elevated temperatures (Erickson, 2007). High oleic sunflower oil (HOSO) is preferred in snack and reformulated products because of its oxidative stability and high monounsaturated fatty acid content (Murphy, 2025). Refined pomace oil (RPO), derived from olive residues, has gained industrial relevance as a cost-efficient oleic-rich alternative with functional stability comparable to olive-based oils (Gharby & Charrouf, 2022).

Differential scanning calorimetry (DSC) is widely used to monitor thermal transitions in fats and oils. Onset temperature, peak temperature, and enthalpy values are commonly used to describe crystallization behavior (Chiavaro, 2014; Islam et al., 2022). Crystallization involves nucleation, crystal growth, and polymorphism, which influence physical properties such as texture and stability (Tan & Che Man, 2000). DSC thermograms are frequently used as thermal fingerprints in authenticity studies, adulteration detection, and oil classification (Islam et al., 2022). Fatty acid and triacylglycerol compositions of edible oils have been extensively reported, and DSC is often used to support oil authentication studies (Islam et al., 2022). Previous research has mainly focused on virgin olive oils or a limited range of seed oils (Chiavaro et al., 2008; Cerretani et al., 2012). Thermal fingerprint data for commonly used refined edible oils remain limited. Refining removes minor components and reduces compositional variability, which complicates differentiation of refined oils. Industrial users frequently handle refined oils without detailed compositional information. Standardized DSC crystallization fingerprints are required for authenticity assessment and comparative characterization of refined oils in industrial practice. This study hypothesized that refined oils with different fatty acid compositions exhibit distinct and reproducible crystallization fingerprints under identical DSC cooling conditions.

DSC cooling thermograms of HOSO, PSO, and RPO were generated across a broad temperature range. Crystallization onset temperatures and enthalpy changes were used as quantitative descriptors of thermal behavior. The aim of this study was to compare the crystallization behavior of HOSO, PSO, and RPO under identical DSC conditions and to determine their distinctive thermal profiles based on cooling thermograms.

MATERIALS and METHODS

Materials

Three edible oils were investigated: high oleic sunflower oil (HOSO), palm super olein (PSO), and refined pomace oil (RPO). These abbreviations are used throughout the manuscript. The refined pomace oil was obtained from Moliveys, Yavuzlar Group (Ayvalık, Balıkesir, Türkiye). The refined palm super olein and the refined high-oleic sunflower oil were supplied by Besler Gıda ve Kimya San. ve Tic. A.Ş. (Ülker Group, İstanbul, Türkiye).

Methods

The thermal behavior of oils was monitored using a differential scanning calorimeter (DSC Q20, TA Instruments Inc., New Castle, DE, USA) at the Instrumental Analysis Laboratory, Department of Food Engineering, Hacettepe University. Approximately 8 ± 1 mg of each liquid oil sample was weighed into aluminum pans (TA Instruments, Cat. No. 900793.901) and hermetically sealed with lids (TA Instruments, Cat. No. 901684.901) using the sealing press. An empty sealed pan was used as the reference. The DSC instrument was calibrated with indium (melting point 156.6 °C; $\Delta H_f = 28.45$ J.g⁻¹). High-purity nitrogen (99.9%) was used as purge gas at a flow rate of 50 mL.min⁻¹. The applied temperature program, adapted from Chiavaro et al. (2008) with slight modifications, was as follows: (i) the samples were heated from room temperature to 30 °C within 2 min and held isothermally for 3 min, (ii) cooled from 30 to -80 °C at a rate of 5 °C.min⁻¹ and maintained at this temperature for 3 min, and (iii) reheated to 30 °C at the same rate (5 °C.min⁻¹). Crystallization thermograms were obtained in approximately 30 min.

In the interpretation of DSC thermograms, several characteristic parameters were evaluated. The onset temperature (T_{onset}) is the point where the heat flow signal starts to deviate from the baseline, marking the beginning of the phase transition. The end temperature (T_{offset}) corresponds to the point where the curve returns to the baseline, indicating the end of the transition. The transition temperature range (ΔT) was calculated as the difference between these two values (Eq. 1). This equation defines the width of the transition region.

$$\Delta T = T_{\text{offset}} - T_{\text{onset}}$$

Equation (1)

The peak temperature (T_{max}) is the temperature at which the maximum heat flow (dQ/dT) occurs during the transition (**Eq. 2**).

$$T_{max} = \arg \max_T \left(\frac{dQ}{dt} \right) \quad \text{Equation (2)}$$

The enthalpy change (ΔH) reflects the total energy involved in the phase transition. It was obtained by integrating the heat flow signal over the temperature range of the transition and normalizing to the sample mass (**Eq. 3**). Here, m is the sample mass (g), and dQ/dT is the measured heat flow (mW). This integration gives the area under the transition peak, corresponding to the energy absorbed during melting or released during crystallization, expressed in $J \cdot g^{-1}$.

$$\Delta H = \frac{1}{m} \int_{T_{onset}}^{T_{offset}} \frac{dQ}{dt} dT \quad \text{Equation (3)}$$

All DSC parameters were calculated using Universal Analysis software V4.5A (TA Instruments). The obtained values were verified according to the definitions described above. Each measurement was performed in triplicate (n:3). Results were expressed as mean \pm standard deviation. Replicate measurements were conducted to demonstrate measurement repeatability and analytical precision. Statistical comparison of peaks was not applicable because crystallization transitions differed in number, temperature, and intensity among oils.

Figure 1 shows the experimental workflow and the DSC cooling procedure applied to the investigated oils. The schematic summarizes the oil types and the temperature program used to obtain cooling thermograms under identical experimental conditions.

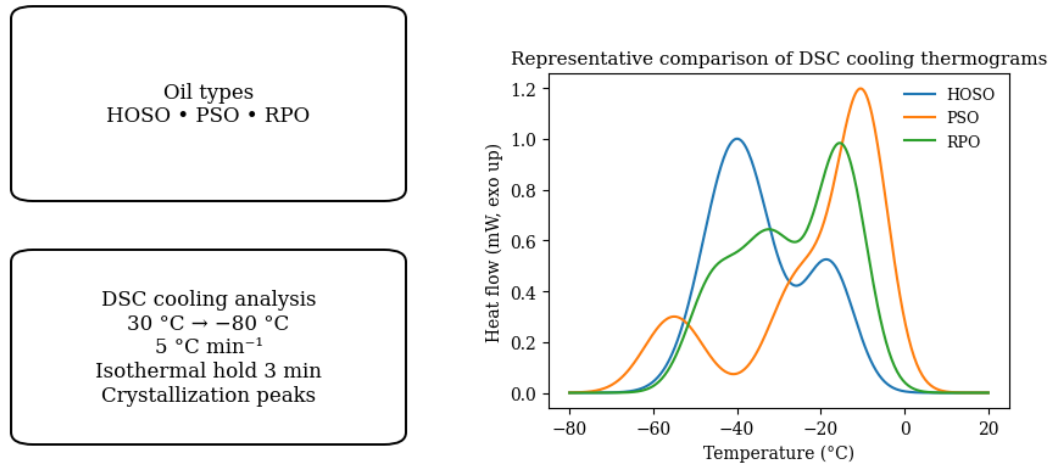


Figure 1. Schematic overview of DSC cooling workflow used to differentiate the studied oils

Graphical representation of the experimental design and DSC cooling procedure applied to the oil samples. The investigated oil types and the applied cooling program are summarized together with an illustrative example of cooling thermograms

RESULTS and DISCUSSION

The thermal behavior of the investigated oils was evaluated using cooling thermograms obtained by differential scanning calorimetry. Cooling was performed from 30 °C to -80 °C at 5 °C.min⁻¹. Distinct crystallization patterns were obtained for PSO, HOSO, and RPO (Fig. 2-4). Quantitative parameters extracted from the thermograms are summarized in Table 1.

The cooling thermogram of refined palm super olein is presented in Figure 2. Three crystallization peaks were detected. The main transition was observed at -2.34 °C with an onset temperature of 0.08 °C and an enthalpy of 23.64 J.g⁻¹. A second transition was detected at -27.17 °C with an onset temperature of -22.24 °C and an enthalpy of 1.27 J.g⁻¹. A third transition was detected at -59.18 °C with an onset temperature of -54.98 °C and an enthalpy of 1.50 J.g⁻¹. The crystallization behavior of high-oleic sunflower oil is illustrated in Figure 3. Two exothermic transitions were identified during cooling. The dominant crystallization event was detected at -43.99 °C with an onset temperature of -40.49 °C and an enthalpy of 47.28 J.g⁻¹. A secondary transition was detected at -21.40 °C with an onset temperature of -18.09 °C and an enthalpy of 0.72 J.g⁻¹. Distinct crystallization events were obtained for refined pomace oil, as presented in Figure 4. The first transition was detected at -16.13 °C with an onset temperature of -12.72 °C and an enthalpy of 2.43 J.g⁻¹. A weaker intermediate transition was detected at -37.21 °C with an onset temperature of -30.77 °C and an enthalpy of 0.39 J.g⁻¹. The most intense transition was detected at -54.20 °C with an onset temperature of -45.51 °C and an enthalpy of 11.76 J.g⁻¹.

Quantitative crystallization parameters derived from the cooling thermograms are presented in Table 1. Clear differences were observed in crystallization onset temperatures and enthalpy values among the investigated oils. In palm super olein, crystallization began at 0.08 °C and the main transition occurred at -2.34 °C with an enthalpy of 23.64 J.g⁻¹. In high-oleic sunflower oil, the dominant crystallization event occurred at -43.99 °C with the highest enthalpy value of 47.28 J.g⁻¹. Refined pomace oil showed three transitions distributed across a wide temperature range, with a dominant low-temperature transition at -54.20 °C and an enthalpy of 11.76 J.g⁻¹.

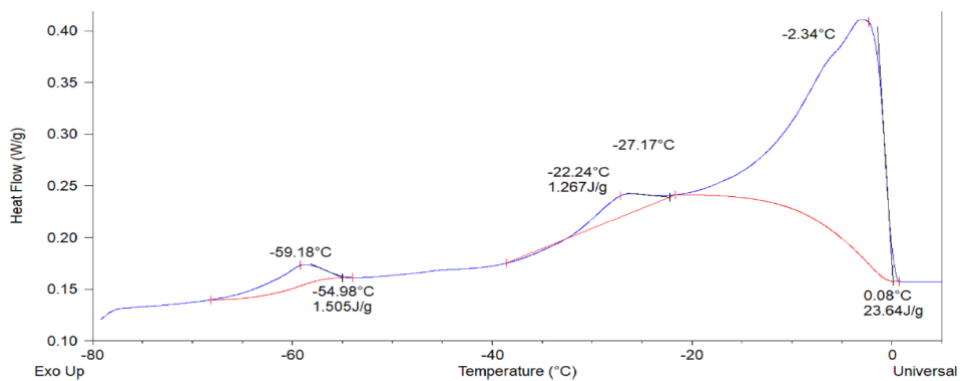


Figure 2. DSC cooling thermogram of refined palm super olein

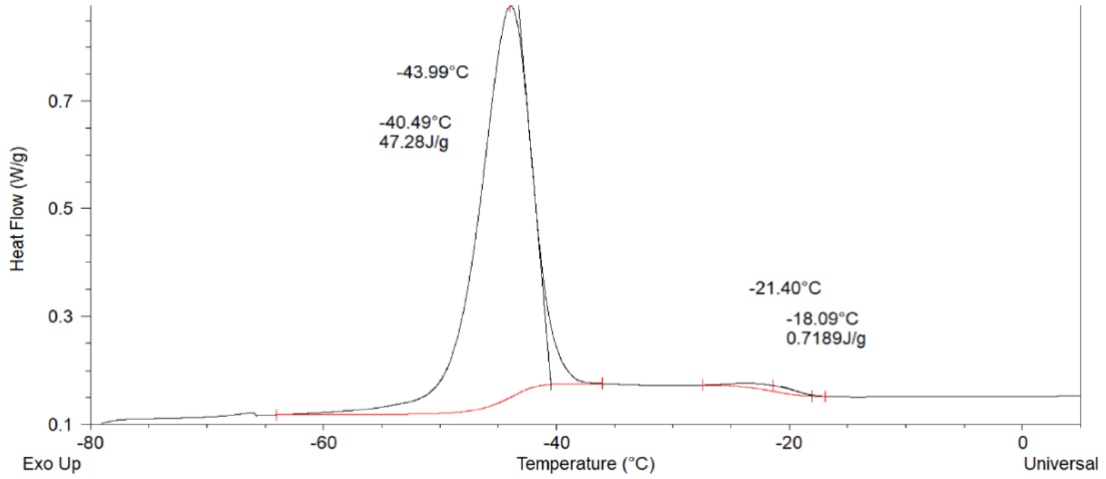


Figure 3. DSC cooling thermogram of refined high-oleic sunflower oil

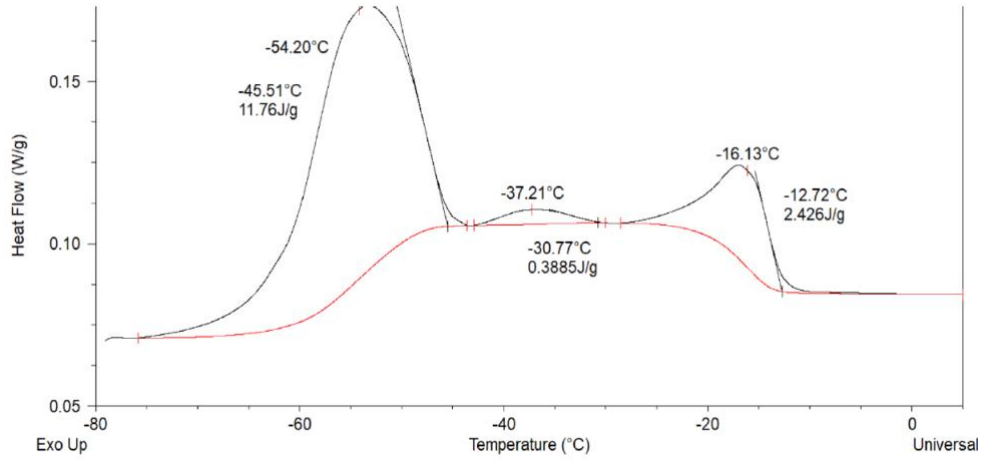


Figure 4. DSC cooling thermogram of refined pomace oil

Table 1. Differential scanning calorimetry parameters of refined palm super olein, high-oleic sunflower oil and refined pomace oil

Oil Type	Peak	Tonset (°C)	Tmax (°C)	ΔH (J.g ⁻¹)
PSO	1	0.08 ± 0.21	-2.34 ± 0.32	23.64 ± 0.88
PSO	2	-22.24 ± 0.41	-27.17 ± 0.55	1.27 ± 0.24
PSO	3	-54.98 ± 0.73	-59.18 ± 0.81	1.50 ± 0.31
HOSO	1	-18.09 ± 0.36	-21.40 ± 0.48	0.72 ± 0.18
HOSO	2	-40.49 ± 0.62	-43.99 ± 0.77	47.28 ± 1.12
RPO	1	-12.72 ± 0.29	-16.13 ± 0.44	2.43 ± 0.33
RPO	2	-30.77 ± 0.51	-37.21 ± 0.69	0.39 ± 0.11
RPO	3	-45.51 ± 0.67	-54.20 ± 0.95	11.76 ± 0.74

Values are expressed as mean ± standard deviation of three independent measurements (n:3)

Table 2. Fatty acid composition of the studied oils reported in the literature (%)

Parameter	PSO	HOSO	RPO
C16:0	38.0-40.0	3.0-4.0	6.0-20.0
C18:0	3.0-5.0	2.0-3.0	0.3-3.5
C18:1	45.0	81.9-84.0	56.0-85.0
C18:2	12.0-13.0	5.0-9.0	3.0-21.0
ΣSFA	42.0-45.0	7.8-9.3	12.0-15.0
ΣMUFA	45.0	82.0-84.0	70.0-75.0
ΣPUFA	12.0-13.0	5.0-9.0	10.0-15.0

Literature data compiled from Che Man et al., 1999; Škevin et al., 2011; Romano et al., 2012; Bouaziz et al., 2010; Mateos et al., 2020; Wang et al., 2021; Lužaić et al., 2025

DSC cooling thermograms obtained in this study demonstrate that refined oils can be differentiated under identical thermal conditions. DSC is widely used to characterize thermal behavior of edible oils and to detect compositional differences between lipid systems (Chiavaro et al., 2008). Thermal transitions of vegetable oils are strongly influenced by fatty acid composition and triacylglycerol structure (Tomaszewska-Gras, 2013). Similar approaches have been applied in oil authentication and characterization studies (Islam et al., 2022; Ghanbari et al., 2023). Fatty acid composition ranges summarized in Table 2 support the crystallization behavior observed in the DSC thermograms.

Earlier crystallization observed in PSO is consistent with the high saturated fatty acid content reported for palm oil fractions (Che Man et al., 1999). Palm oil fractions contain higher-melting triacylglycerols that crystallize at higher temperatures (Tan & Che Man, 2000). Multiple crystallization regions have been described in palm oil systems and PSO blends (Ng et al., 2014). Fractionated crystallization behavior has also been reported using DSC analysis of palm oil fractions (Habi Mat Dian et al., 2006).

Delayed crystallization in HOSO is consistent with the thermal behavior of oils rich in oleic acid. High-oleic sunflower oil typically contains more than 80% oleic acid (Wang et al., 2021), as summarized in Table 2. Reduced saturated fatty acid content is associated with lower crystallization temperatures in vegetable oils (Tomaszewska-Gras, 2013). Similar relationships between fatty acid composition and crystallization behavior have been described in DSC studies of edible oils (Islam et al., 2022).

Crystallization across a broader temperature range in RPO agrees with the compositional variability reported in olive pomace oil (Mateos et al., 2020). The wider compositional ranges listed in Table 2 are consistent with the broader crystallization interval observed for RPO. Thermal studies of olive oils have shown broad crystallization ranges and multiple transitions linked to compositional heterogeneity (Cerretani et al., 2012). Changes in lipid composition during frying of refined olive

pomace oil have also been reported (Ben Hammouda et al., 2018). Similar compositional characteristics have been described in refined olive pomace oil (Ruiz-Méndez et al., 2022).

The relationship between fatty acid composition and crystallization behavior is well established. Higher saturated fatty acid content promotes earlier crystallization (Tan & Che Man, 2000). Higher monounsaturated fatty acid content delays crystallization and reduces the number of crystallization transitions (Tomaszewska-Gras, 2013). Distinct crystallization patterns obtained under identical DSC conditions indicate that cooling thermograms provide characteristic thermal fingerprints for refined oils.

Thermal differentiation of refined oils using DSC cooling thermograms has potential applications in quality control and industrial classification. Crystallization profiles may support verification of oil identity during production and storage. Thermal profiling has been proposed as a complementary approach for authenticity assessment and detection of compositional deviations in edible oils (Chiavaro et al., 2008; Islam et al., 2022). Differences observed among PSO, HOSO, and RPO indicate that DSC cooling thermograms can support classification of refined oils based on compositional characteristics.

Distinct crystallization fingerprints indicate that DSC cooling thermograms may support authenticity verification of refined edible oils. Reference thermograms generated under standardized conditions may enable screening of oil identity and detection of compositional deviations during storage, blending, or supply chain handling. DSC has previously been applied to identify compositional changes and blending effects in vegetable oils (Cerretani et al., 2012; Ghanbari et al., 2023). Deviations from expected crystallization behavior may therefore indicate blending or substitution in refined oils.

Conclusion

Crystallization behavior of HOSO, PSO, and RPO was characterized using DSC cooling thermograms. Distinct crystallization profiles were obtained under identical cooling conditions. PSO showed the earliest crystallization, with Tonset at 0.08 °C and a dominant peak at -2.34 °C with an enthalpy of 23.64 J.g⁻¹. Additional transitions were detected at -27.17 °C and -59.18 °C with enthalpy values of 1.27 and 1.50 J.g⁻¹, respectively. HOSO crystallized at lower temperature, with Tonset at -18.09 °C and a dominant peak at -43.99 °C with an enthalpy of 47.28 J.g⁻¹. RPO showed intermediate behavior, with Tonset at -12.72 °C and peaks at -16.13 °C, -37.21 °C, and -54.20 °C, with the lowest-temperature transition showing an enthalpy of 11.76 J.g⁻¹.

DSC cooling thermograms enabled differentiation of the investigated refined oils based on Tonset, Tmax, and ΔH. Refining processes apply standardized operations that remove impurities and reduce compositional variability among oils, which makes differentiation of refined oils challenging in industrial practice. The thermal profiles obtained in this study therefore provide comparative thermal data for characterization of refined edible oils under standardized DSC conditions.

Future research may extend this approach to adulteration detection studies based on DSC crystallization fingerprints.

Additional Declaration

Acknowledgements

I would like to thank the Instrumental Analysis Laboratory staff of the Department of Food Engineering, Hacettepe University, for providing access to DSC facilities and technical support during the experiments.

Author Contributions

The author performed all stages of the study.

Funding

This research was supported by the Çankırı Karatekin University Scientific Research Projects Unit (BAP) under Project No. MF080120B30.

Responsible Artificial Intelligence Statement

No generative AI tools were used in data generation, analysis, or interpretation.

Conflicts of Interest

The author declares no conflict of interest

Ethics Approval

This study does not require ethics committee approval as it does not involve any direct application on human or animal subjects.

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