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*Review Article*

# Differential Scanning Calorimetry (DSC) for Edible Oil Authentication

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**Abstract:** Edible oils greatly contributed to the large profits in many food industries because of their household usage for cooking and food preparations. However, the source of oil products raised the concerns of authorities and consumers. The authentication of oil is anticipated to be a crucial and essential undertaking for identifying foreign substances in edible oils. This is vital for safeguarding consumers' rights to confidently use any food product without concerns about its ingredients. For this issue, differential scanning calorimetry (DSC) is employed as the analytical tool to analyze the oils. DSC provides specific thermal profiles including cooling and heating curves for each oil sample. Several methods have been devised to adulterate oils with various substances, underscoring the need to distinguish between authentic edible oils and those that may have been adulterated. Adulterants are possibly derived from various types of constituents including replacing similar with cheaper or with low quality. Each oil composition demonstrates distinct thermal profiles that can be identified through DSC. Thus, this paper aims to review the use of DSC as an analytical method for edible oil authentication.

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## 1. Introduction

The authentication of edible oils is of paramount importance given the increasing demand for food. Manufacturers and investors, driven by economic considerations and the desire to reduce production costs, may be inclined to substitute certain ingredients with cheaper alternatives, thereby compromising the originality of the food. This trend is further exacerbated by economic burdens and escalating manufacturing costs. Instances of food misdescription, such as the substitution of a comparable but less costly ingredient or the over-declaration of a quantitative ingredient, are prevalent (Giménez *et al.*, 2010). Consequently, there is a pressing need for the authentication of edible oils to empower consumers and buyers in making informed choices, ensuring they select genuine oils without doubt or hesitation. Edible oils have been used for various purposes such as cooking, food preparations, and industrial food manufacturing. However, they are one of the most adulterated food products (Sudhakar *et al*., 2023).

Differential Scanning Calorimetry (DSC) is a technique used for analyzing the thermal properties of substances and involves measuring the heat flow associated with phase transitions or reactions as a function of temperature. This method typically consists of a sample chamber and a reference chamber, both subject to the same temperature conditions. By detecting the heat changes that occur in the sample compared to the reference, DSC provides valuable insights into the thermal behavior and characteristics of materials, including edible oils. DSC is the preferred thermal analysis method for analyzing oils and fats, especially in investigating oil authenticity as a quality control measure. As a function of temperature, this thermo-analytical approach calculates the difference between the amount of heat used to raise the temperature of a sample and a suitable reference material (Nur Azira & Amin, 2016). DSC includes information on the melting and crystallization of oils, which is affected directly by their physicochemical properties such as fatty acid, triglyceride (TAG) and chemical structure (Embaby *et al*., 2022)

The fundamental principle of DSC revolves around the measurement of the heat required to increase the temperature of a sample relative to a reference specimen. This technique relies on maintaining a nearly constant temperature for both the reference and sample during the experiment. To achieve this, a temperature program is devised, ensuring that the temperature of the samples increases linearly over time. DSC offers several strengths as a thermal analysis method. One of its key advantages is its ability to precisely quantify heat changes associated with phase transitions or reactions, providing detailed information about the thermal behavior of materials, including oils and fats. The technique is particularly well-suited for analyzing these substances, making it a valuable tool in quality control measures for assessing oil authenticity (Islam *et al*., 2022; Islam *et al*., 2023).

However, it's essential to note certain limitations of DSC. While it excels in detecting phase transitions and reactions, DSC may not provide a complete picture of complex sample compositions or interactions. Additionally, factors such as sample size, heating rate, and experimental conditions can influence the DSC results, requiring careful consideration and standardization in experimental setups. Despite these limitations, DSC remains a widely utilized and effective method for investigating thermal properties in various materials. The application of DSC in the field of oils and fats has been useful due to the increasing interest in the authentication of oils and the identification of adulteration (Indriyani *et al.*, 2016a; Li *et al.*, 2016).

In addition to DSC, researchers also developed various instrumental analytical methods for the detection of adulteration and authentication of edible oils. Methods used to detect the adulteration of edible oil include liquid chromatography-mass spectrometry (Sumara *et al.*, 2023), Fourier transform infrared (FTIR) spectroscopy (Bunaciu *et al.*, 2024; Ye & Meng, 2022), Raman spectroscopy (Didham *et al.*, 2020), nuclear magnetic resonance (NMR) (Dogruer *et al.*, 2021), ultrasound inspection (Jimenez *et a*l., 2024) and PCR (Jing *et al.*, 2024). The selection of a particular method for detecting adulteration depends on various factors, including practicality, budget constraints, and the specific requirements of the analysis. Each method comes with its own set of advantages and disadvantages.

The effective assessment of the substantial amount of information generated through these multiple analysis techniques is made possible by employing chemometric techniques. In this way, chemometrics complements the analytical methods, offering a comprehensive approach to evaluating edible oils as well as contributing to both the efficiency and reliability of the assessment process. In short, this paper aims to review the use of DSC as an analytical method for edible oil authentication. In addition, this paper will also elaborate on some applications of chemometric techniques in combination with DSC for edible oil authentication.

## 2. Authentication of Edible Oils Using Differential Scanning Calorimetry (DSC)

Oil authentication through the detection of adulterants, geographical origin and falsely labeled products is debatable among sellers, traders and consumers. This incident has drawn the public attention since it brings serious consequences to society. Therefore, the establishment of rapid methods for the authentication and validation of edible oils is necessary and DSC is one of them. DSC applications in research fields are not rare instead it is reliable and very promising, especially in oil authentication studies (Table 1).

### *2.1 Differential Scanning* Calorimetry (DSC*) Studies on Adulterations of Edible Oils.*

Detection of adulterants is one of the ways to assess the authenticity of edible oils. In this context, all foreign substances either edible or not will be considered adulterants if it is not the original composition of the oil products. Many experiments on the authentication of olive oils have been conducted, according to researchers. Olives are widely grown in the Mediterranean region, and olive oils are the most common source of dietary fats in Mediterranean countries. Due to its higher price as compared to other edible oils, olive oils are often adulterated with inexpensive oils for the sake of greater profitability (Jafari *et al.*, 2009). The efficacy of physiochemical indices combined with fatty acid (FA) profiles in categorizing and detecting the purity of Iranian olive oils was discussed in this paper. Thus, the relationship between the thermal reaction of oil samples and their FA structure, the crystallization and melting profiles of soybean, sunflower, canola, and genuine olive oils was investigated. The information gathered would be used to determine if certain seed oils can be mixed with olive oils as adulterants. As previously stated, the rate of cooling or heating affects the form and number of thermal curve peaks. However, since all studies were carried out at the same rate using the DSC instrument, the influence of this element may be overlooked in this research, which focuses on the separation of oil samples in terms of FA and triacylglycerol composition. From the DSC, the presentation of peaks in the cooling and melting curves is significant as it represents the composition of FA and TAG composition in each edible oil which can be used as the key point to distinguish pure and adulterated ones.

Besides, the usage of DSC in avocado oil (AO) authentication is also promoted (Indriyani *et al.*, 2016a). Avocado oil was treated with various concentrations of refined bleached deodorized palm super olein (RBDPSO). In general, the thermogram profiles of AO and RBDPSO had two and three principal peaks, respectively, due to variations in their fatty acid and TAG compositions (Tan & Che Man, 2000). As RBDPSO is introduced to AO, the temperature of both transitions is shifted downward, improving crystallization, melting enthalpy, and developing all processes over a broader temperature spectrum. In a broad sense, DSC is capable of providing very valuable outcomes in authentication studies.

Other than the edible oils mentioned above, camellia oil has also been monitored for adulteration in oil authentication. Camellia oil, also known as oil-tea, tea tree oil, or tea seed oil, is an edible vegetable oil squeezed or extracted from the seed of the camellia plant (Camellia oleifera Abel). Because of the high economic value of camellia oil (CMO), dishonest merchants took advantage of the ability to blend inexpensive vegetable oils or non-edibles with CMO. In light of this occurrence, DSC was used to detect CMO adulteration with specific vegetables (Li *et al.*, 2016). Five vegetable oils namely sesame oil (SSO), sunflower oil (SFO), peanut oil (PNO), corn oil (CO) and canola oil (CNO) each with six different concentrations were added to CMO. It is interesting to find that DSC showed satisfactory results. From the cooling curves, the overall line shape of the exothermic peak of adulterated CMO were altered which showed significant differences in comparison with pure CMO. The addition of the five vegetable oils to CMO decreased the peak height of crystallization as the concentration of adulteration increased. Similarly, obvious differentiation could also be observed in heating curves.

Furthermore, the sole use of DSC in edible oil authentication has also been demonstrated in a study examining fraudulent sesame oil (Fahimdanesh *et al.*, 2014). Three separate concentrations of sunflower oil and corn oil were combined with sesame oil in this study. According to DSC melting criteria, as the proportion of corn oil and sunflower oil in sesame oil increases, the peak of the sesame oil shifts to lower temperatures, and temperatures begin to rise.

Despite using cheaper vegetable oils and non-edible oils, some fraudsters also added animal fats to other vegetable oils. Initially, the concept behind combining animal fats like lard and tallow was to make unique products (Marikkar *et al.*, 2012). Adding animal fats to plant oils, however, appeared unacceptable due to religious restrictions and a negative nutritional view of animal fat intake. Adulterants in sunflower oil (SFO) such as lard (LD), beef tallow (BT), and chicken fat (CF) are identified using DSC heating thermograms (Marikkar *et al.*, 2012). SFO was combined with different amounts of animal fats ranging from 1 to 20% (w/w). The area of the DSC melting curve of SFO between 0 and 50°C was found to be vulnerable to TAG compositional changes due to the different animal fats, according to the findings. Changes caused by BT could be seen in thermal profiles as early as 2% of adulteration, while changes caused by LD could only be seen after 8% of adulteration. Furthermore, based on the characteristic shape, height, and location of the contaminant of peaks, it is possible to distinguish lard contamination from other animal fat contaminations in SFO.

When it comes to non-halal ingredients like lard, food manufacturers and producers can be considered an adulterant in edible oils. In line with Muslim and Jewish societies, the detection of unlawful ingredients specifically pork and lard in the food becomes a major concern. This incident is globally appealing since Muslim and Jewish markets have developed progressively because of the increasing awareness of their needs (Mansor *et al.*, 2012). It is also mentioned that in fats and oils, the use of lard (LD) as an adulterant is common. The authors used the DSC in the identification of lard (LD) in virgin coconut oil (VCO) by demonstrating unique thermal profiling for each oil in this case. VCO was blended with eight separate concentrations of LD. The obtained results demonstrated DSC's ability to recognize shifts in the cooling and heating thermograms. A slight difference was observed in adulterated VCO based on the heating thermogram, in which as the concentration of LD increased, a smaller shoulder peak emerged in the main peak, which eventually smoothed out to the major peak. On the other hand, a cooling thermogram revealed more distinct differences among the adulterated VCO. From the cooling curves, there were two major peaks developed that are named as peak C and peak D. The peak C increased when the LD concentration increased, in contrast, peak D decreased as the concentration of LD increased. The results from DSC can serve as good evidence for detecting LD adulteration in VCO.

The topic of lard adulteration in edible oils was also addressed. The content of lard stearin (LS) in canola oil (CaO) was detected and quantified using DSC (Marikkar & Rana, 2014). Similar to the study mentioned before, pure CaO was adulterated with different concentrations of LS ranging from 5 to 15% (w/w) for the spike samples preparation. The results revealed that the characteristic contaminant peaks were already formed in cooling and heating curves when CaO was adulterated with 5% (w/w) of LS, which can be used as an indicator to detect those adulterants in adulterated CaO. This occurrence illustrated that the increased concentration of LS would influence the size of contaminants' peaks. Based on cooling and heating curves, many changes occurred when pure CaO was adulterated with LS as the result of TAG compositional changes. The major contributor to the form of the cooling and heating curves was thought to be TAG compositional shifts. However, DSC proved to be a far more effective method for detecting fat, with a detection limit of 1%. Furthermore, DSC does not necessarily require any sample pre-treatment which allows for quick analysis (Goggin & Murphy, 2018; Malvis *et al*., 2019). In other words, the employment of DSC for oil authentication was reliable and significant.

### *2.2 Differential Scanning Calorimetry (DSC) Studies of Geographical Origin of Edible Oils.*

The geographical origin of edible oils is also being considered when authenticating them. Thus, traceability of the original geographical regions of edible oils is conducted. The application of DSC in this section will be based on the geographical sources of olive oils. Traceability of extra virgin olive oils (EVOOs) is currently being improved by manufacturers to prevent mislabeling of geographical origin and olive varieties of the commodity and, as a result, to provide accurate information to consumers (Kotti *et al*., 2009). As a result, in this case, the use of DSC for EVOO discrimination based on cultivar and geographical origin is recommended (Chiavaro *et al.*, 2010). Thirteen monovarietal EVOO samples were obtained from drupe cultivars grown in two separate Italian regions, respectively Abruzzo and Apulia, for this research. Drupes for oil production were hand-picked in 2007 and refined in industrial mills continuously. The outcomes of this research relied upon the cooling thermograms and thermal properties. The thermal properties of the three deconvoluted peaks (area, onset and offset transition temperatures, transition range, and peak temperature) revealed a strong association between major and minor components (diacylglycerols, free fatty acids) and oxidative stability indices. The study of thermal properties obtained by cooling transformation does not affect sample discrimination based on geographical provenience. Nonetheless, Ton, Toff, and the peak areas of the two deconvoluted transitions that peaked at the lowest temperatures, as well as Toff of the deconvoluted peak that peaked at the maximum temperature, distinguished oil samples according to their geographical origin. These results demonstrated that the DSC cooling transformation greatly aided in the differentiation of oil samples from various geographical origins.

Another study used a similar method to differentiate EVOOs according to geographical origin (Chiavaro *et al.*, 2011). Fifty-three commercial EVOO samples from various geographical origins were examined. To be more precise, 43 of them were collected from olives grown in Italy, 10 from Croatia, five from the Istria peninsula, and one from another part of the region (named Croatia Centre). Some thermal properties obtained from DSC analysis, in particular those obtained by peak 3, which peaks at the highest temperature area of cooling thermograms, can differentiate not only among samples from different Italian regions but also grouping samples according to different varieties. By comparing samples from various Italian regions and Croatia, however, the distinctions were more subtle (Istria and another zone of the country). In conclusion, it should be noted that the discriminant can be obtained by analyzing only some regions of cooling profiles rather than the whole cooling profiles.

Apart from that, the same approach of employing DSC in distinguishing EVOO was also attempted in another study (Kotti *et al.*, 2009). This analysis aims to investigate the cooling and heating profiles of EVOO from two Tunisian varieties (Chetoui and Chemlali) to see whether DSC can be used to distinguish samples based on the cultivar–environment relationship relating their thermal properties to TAG and FA composition. Seven EVOO samples were obtained from drupes of Chetoui and Chemlali cultivars grown in various regions of Tunisia during sample preparation. The method of oil extraction was also similar to that used in previous studies (Chiavaro *et al.*, 2010). DSC heating profiles were more persuasive in detecting discrimination among the cultivar and geographical origin in comparison to cooling profiles, as the difference was detectable not only between samples from Chetoui and Chemlali cultivars but also between oil samples from the same cultivar and different geographical regions, as shown by DSC thermograms, as the differentiation was detectable not only between samples from Chetoui and Chemlali cultivars but also between oil samples from the same cultivar and different geographical regions both for exotherm and endotherm events. Nonetheless, DSC heating profiles combined with cooling profiles have proven to be a useful tool for distinguishing EVOO from the same fruit variety with diverse regional origins.

### *2.3 Differential Scanning Calorimetry (DSC) Studies on Profile Variation of Edible Oils.*

The application of DSC in the identification of profile variation in edible oils provides a comprehensive understanding of their thermal characteristics. This information is crucial for quality control, authenticity verification, and ensuring the integrity of edible oil products in the food industry. The investigation into avocado oils from distinct cultivars showcased significant variations in physicochemical attributes such as fatty acid composition, oxidative stability, and thermal behavior. Employing a combination of traditional analytical methods and advanced techniques, such as DSC and PCA, facilitated a comprehensive understanding of the distinctive qualities inherent in avocado oils from these specific cultivars (Indriyani *et al.*, 2016b).

## **3. Differential Scanning Calorimetry (DSC) in Combination with Chemometric Analysis**

Chemometric analysis involves the application of statistical and mathematical methods to chemical data, enabling the extraction of meaningful information and patterns. In the context of authentication studies, combining chemometric analysis with Differential Scanning Calorimetry (DSC) becomes crucial for a comprehensive understanding of edible oils. To enhance the efficacy of DSC in authentication studies, researchers often integrate chemometric analysis into their methodologies. This combination allows for a more sophisticated interpretation of the data generated by DSC. Chemometric techniques, including multivariate analysis and pattern recognition, enable researchers to discern subtle variations in thermal profiles that may be indicative of specific oil types or adulterations. The relevance of this combination lies in its capacity to provide a more nuanced and accurate assessment of the authenticity of edible oils, a task that may be challenging when relying solely on individual thermal profiles. In the analysis of fats and oils, researchers have frequently applied combination techniques to ease data interpretation, as illustrated by numerous studies in Table 1. Several chemometric approaches, including Principal

Component Analysis (PCA) (Huang *et al*., 2023; Indriyani *et al*., 2016b; Rajagukguk *et al*., 2023), Partial Least Square Regression (PLSR) (Gonzalez-Ortega *et al*., 2024; Pereira *et al*., 2023), Stepwise Multiple Linear Regression (SMLR) (Indriyani *et al*., 2016b), and Linear Discriminant Analysis (LDA) (Chatziantoniou *et al*., 2014; Godoy *et al*., 2020) have been combined with DSC.

The integration of DSC and chemometric analysis proves to be a robust approach for authenticating edible oils. DSC capturing heat flow associated with phase transitions during temperature changes unveils distinctive thermal profiles reflecting specific transitions like melting points or crystallization in edible oils. Preprocessing steps like baseline correction and normalization enhance the interpretability of complex DSC data. Subsequently, chemometric techniques are applied to extract meaningful information and identify patterns. Calibration with known samples enables the establishment of correlations between observed DSC patterns and unique characteristics of each oil type, creating a valuable model for authenticating and classifying unknown samples in quality control for the food industry. This approach is particularly effective in detecting adulteration or fraudulent practices, ensuring consumers receive authentic and accurately labeled edible oils.

The described integration of chemometric analysis with DSC is showcased as a powerful tool for discerning alterations in the properties of edible oils, as demonstrated in both flaxseed oil and butter adulteration studies. In the flaxseed oil study, DSC analyzed melting phase transition curves, revealing linear correlations between refined rapeseed oil concentration and DSC parameters. Various chemometric approaches, including Classification Models (LDA, Adaptive Regression Splines - MARS, Support Vector Machine - SVM, Artificial Neural Networks - ANNs) and Regression Models (Multiple Linear Regression - MLR, MARS, SVM, ANNs, and PLS), were effectively applied to assess DSC efficacy in detecting adulterations. Results highlighted the LDA model's high accuracy (99.5%) in classification and the ANN model's superior correlation (R = 0.996) in regression analysis (Islam *et al*., 2023).

Similarly, the butter adulteration study by Gonzalez-Ortega *et al*. (2024) utilized DSC with chemometric analysis (PCA and PLSR) to detect fat adulteration. Discriminant analysis using K-Nearest Neighbors achieved an accuracy rate exceeding 92.1% in differentiating authentic from adulterated samples, while hierarchical cluster analysis (HCA) discriminated between palm stearin and coconut oil at concentrations exceeding 5% (w/w). This integrated approach demonstrated the high sensitivity of DSC in detecting small alterations in butter's melting pattern due to fat adulteration, showcasing its efficacy in quality control within the food industry. Collectively, these findings underscore the practical utility of combining DSC with chemometric analysis for comprehensive and accurate assessments in the authentication and quality control of edible oils and butter products.

**Table 1.** Authentication analysis of edible oil by using DSC.

| **Issue: Adulteration** | | | | | |
|---|---|---|---|---|---|
| **Oils/Fats** | **Adulterant** | **Aim of study** | **Chemometric approach** | **Remarks** | **Reference** |
| Butter | Palm stearin and coconut oil | To propose an innovative method combining DSC with chemometric analysis for the detection of fat adulteration in butter. | Principal component analysis (PCA), Partial least squares regression (PLSR) and Hierarchical Cluster Analysis (HCA) | Limit of detection (LOD): 5% (w/w) | Gonzalez-Ortega *et al*., 2024 |
| Butter | Palm oil | To test the feasibility of the DSC technique coupled with PCA for the distinction of pure butterfat from adulterated. | PCA | LOD: 2% (w/w) | Tomaszewska-Gras, 2016 |
| Extra virgin olive oil (EVOO) | Canola oil, corn oil, soybean oil, peanut oil, sunflower oil | To discriminate against potential adulteration of common EVOO products in the Chinese market. | PCA | 100% ability to distinguish EVOO from non-EVOO | Huang *et al*., 2023 |
| Flaxseed oil | Refined Rapeseed Oil | To compare different chemometric models to detect adulteration of flaxseed oil with refined rapeseed oil using DSC. | 1) Classification models (Linear Discriminant Analysis, LDA Adaptive Regression Splines, MARS, Support Vector Machine, SVM, Artificial Neural Networks, ANNs);<br>2) Regression models (Multiple Linear Regression, MLR, MARS, SVM, ANNs, PLS) and<br>3) A combined model of Orthogonal Partial Least Squares Discriminant Analysis (OPLS-DA) | The ANN model showed the highest correlation between observed and predicted values (R = 0.996), while other models showed goodness of fit as following MARS > SVM > MLR. | Islam *et al*., 2023 |
| Avocado oil | Avocado oil-refined bleached deodorized | To discriminate between avocado oil and adulterated samples. | Partial Least Square (PLS) and Stepwise Multiple Linear Regression (SMLR) | - | Indriyani *et al*., 2016a |

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|---|---|---|---|---|---|
| | palm superolein | | | | |
| Camellia oil | Sesame oil, sunflower oil, peanut oil, corn oil and canola oil | To verify the adulteration of camellia oil. | SMLR | The detection limit of sesame oil, sunflower oil and corn oil was 10 %, peanut oil 20 %, and rapeseed oil 30 % respectively. | Li *et al.*, 2016 |
| Virgin Coconut Oil (VCO) | Lard | To detect the presence of lard in VCO. | SMLR | - | Mansor *et al.*, 2012 |
| Olive oil | Soybean, sunflower and canola oils | Detection of adulteration of olive oil with soybean, sunflower, and canola oils. | - | - | Jafari *et al.*, 2009 |
| Sesame oil | Corn oil and sunflower oil | To investigate fraud in sesame oil using DSC analysis. | - | - | Fahimdanesh *et al.*, 2014 |
| Sunflower oil | Lard, beef tallow, and chicken fat | Lard, chicken fat, and beef tallow concentrations in sunflower oil. | - | Can detect below 20% (w/w) level of lard and beef tallow in sunflower oil. | Marikkar *et al.*, 2012 |
| Canola oil | Lard stearin | To detect and quantify lard stearin content in canola oil. | - | - | Marikkar and Rana, 2014 |
| **Issue: Geographical origin** | | | | | |
| **Oils/Fats** | | **Aim of study** | **Chemometric approach** | | **Reference** |
| EVOO | | To find and mark differences and similarities that allow to cluster the studied samples according to their thermodynamic characteristics. | PCA | - | Mallamace *et al.*, 2017 |
| EVOO | | Identifying the varietal and geographical origins of Greek extra virgin olive oil | Linear Discriminant Analysis (LDA) | - | Chatziantoniou *et al.*, 2014 |
| | | Discrimination EVOO of different cultivars and geographical origin | - | - | Chiavaro *et al.*, 2010 |

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| | To discriminate among extra virgin olive oils based on cultivar–environment interaction. | - | - | Kotti *et al*., 2009 |
| Sesame oil | To conduct thermal characterization of sesame seeds and oils from various geographical origins (Ethiopia, India, Nigeria, Sudan, Turkey), different methods of extraction (hexane and cold-pressing), and different types of derived products (halva and tahini). | PCA | The origin of the seeds does not influence the melting profile of sesame oil. | Rajagukguk *et al*., 2022 |
| **Issue: Profile variation** | | | | |
| **Oils/Fats** | **Aim of study** | **Chemometric approach** | | **Reference** |
| Raspberry seed oils | To investigate the melting-crystallization behavior by discriminating marketed cold-pressed raspberry oil with a freshly prepared sample. | PCA | Two groups of oils were distinguished. | Rajagukguk *et al*., 2023 |
| Avocado oil (AO) | To determine the oil properties of different Indonesian avocado varieties. | PCA | Avocado oils from three different cultivars had different melting and crystallization profiles. | Indriyani *et al*., 2016b |

## 4. Conclusion

Throughout the century, studies and research have been abundantly conducted by researchers regarding edible oil authentication, since many food producers and manufacturers aggressively adulterate pure edible oils to gain profit in the easiest ways. Thus, oil authentication is very important, especially for the quality control of edible oil products as well as to prevent the violation of human rights for consumers to use genuine products. With the establishment of several analytical methods such as DSC, authentication studies could be done as DSC is suitable for fats and oils analysis. As the research progressed, the researchers attempted to combine the DSC with several chemometric analyses such as PCA, PLSR, SMLR and LDA. Interestingly, this coupled technique was really promising as the interpretation of a large dataset of DSC would be easier. Despite these advantages, with every method developed, there will also be some disadvantages encountered. Therefore, choosing and determining the appropriate methods before conducting the studies were essential to obtain useful and meaningful results.

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## References

Sudhakar, A., Chakraborty, S. K., Mahanti, N. K., *et al.* (2023). Advanced techniques in edible oil authentication: A systematic review and critical analysis. *Critical Reviews in Food Science and Nutrition*, *63*(7), 873-901.

Bunaciu, A. A., Vu, D. H., & Aboul-Enein, H. Y. (2024). Edible oil discrimination by fourier transform infrared (FTIR) spectroscopy and chemometrics. *Analytical Letters, 57*(3), 445-455.

Chatziantoniou, S. E., Triantafillou, D. J., Karayannakidis, P. D., *et al.* (2014). Traceability monitoring of Greek extra virgin olive oil by Differential Scanning Calorimetry. *Thermochimica Acta, 576,* 9–17.

Chiavaro, E., Cerretani, L., di Matteo, A., *et al.* (2011). Application of a multidisciplinary approach for the evaluation of traceability of extra virgin olive oil. *European Journal of Lipid Science and Technology,113*(12), 1509–1519.

Chiavaro, E., Rodriguez-Estrada, M. T., Bendini, A., *et al.* (2010). Correlation between thermal properties and chemical composition of Italian virgin olive oils. *European Journal of Lipid Science and Technology,112*(5), 580–592.

Didham, M., Truong, V. K., Chapman, J., *et al.* (2020). Sensing the addition of vegetable oils to olive oil: The ability of UV–VIS and MIR spectroscopy coupled with chemometric analysis. *Food Analytical Methods, 13*(3), 601–607.

Dogruer, I., Uya, H. H., Uncu, O., *et al.* (2021). Prediction of chemical parameters and authentication of various cold pressed oils with fluorescence and mid-infrared spectroscopic methods. *Food Chemistry, 345*, 128815

Embaby, H. E., Miyakawa, T., Hachimura, S., *et al.* (2022). Crystallization and melting properties studied by DSC and FTIR spectroscopy of goldenberry (*Physalis peruviana*) oil. *Food Chemistry, 366*, 130645.

Fahimdanesh, M., Bahrami, M., & Zargani, M. (2014). 66 check fraud sesame (*Sesamus Indicum*) oil using differential scanning calorimetry (DSC) analysis. *International Journal of Plant Animal and Environmental Sciences, 4*(2), 554-560.

Giménez, M. J., Pistón, F., Martín, A., *et al.* (2010). Application of real-time PCR on the development of molecular markers and to evaluate critical aspects for olive oil authentication. *Food Chemistry, 118*(2), 482–487.

Godoy, A. C., dos Santos, P. D. S., Nakano, A. Y., *et al.* (2020). Analysis of vegetable oil from different suppliers by chemometric techniques to ensure correct classification of oil sources to deal with counterfeiting. *Food Analytical Methods, 13*, 1138–1147.

Goggin, K. A. & Murphy, D. J. (2018). Monitoring the traceability, safety and authenticity of imported palm oils in Europe; Monitoring the traceability, safety and authenticity of imported palm oils in Europe. *Oilseeds Fats, Crops and Lipids, 25*(6), 603.

Gonzalez-Ortega, R., Rajagukguk, Y. V., Ferrentino, G., *et al.* (2024). Detection of butter adulteration with palm stearin and coconut oil by differential scanning calorimetry coupled with chemometric data analysis. *Food Control, 157*, 110165.

Huang, K., Zhong, P. & Xu, B. (2023). Discrimination on potential adulteration of extra virgin olive oils consumed in China by differential scanning calorimeter combined with dimensionality reduction classification techniques. *Food Chemistry, 405*, 134996.

Indriyani, L., Rohman, A. & Riyanto, S. (2016a). Authentication of avocado oil (Persea americana Mill.) Using differential scanning calorimetry and multivariate regression. *Asian Research Journal of Agriculture, 10*(2), 78–86.

Indriyani, L., Rohman, A. & Riyanto, S. (2016b). Physico-chemical characterization of avocado (Persea americana mill.) oil from three Indonesian avocado cultivars. *Journal of Medicinal Plants Research, 10*(1), 67–78.

Islam, M., Belkowska, L., Konieczny, P., *et al.* (2022). Differential scanning calorimetry for authentication of edible fats and oilse - What can we learn from the past to face the current challenges?. *Journal of Food and Drug Analysis, 30*, 185 – 201.

Islam, M., Kaczmarek, A., Grygier, A., *et al.* (2023). DSC phase transition profiles analyzed by control charts to determine markers for the authenticity and deterioration of flaxseed oil during storage. *Foods, 12*, 2954.

Jafari, M. Kadivar, M. & Keramat, J. (2009). Detection of adulteration in Iranian olive oils using instrumental (GC, NMR, DSC) methods. *Journal of the American Oil Chemists' Society, 86*(2), 103–110.

Jimenez, A., Rufo, M., Paniagua, J. M., *et al.* (2024). Temperature dependence of acoustic parameters in pure and blended edible oils: Implications for characterization and authentication. *Ultrasonics, 138*, 107216.

Jing, Q., Liu, S., Song, Y. & Toa, X. (2024). TaqMan real-time quantitative PCR for the detection of beef tallow to assess the authenticity of edible oils. *Food Control. 156*: 110139.

Kotti, K., Chiavaro, E., Cerretani, L., *et al.* (2009). Chemical and thermal characterization of Tunisian extra virgin olive oil from Chetoui and Chemlali cultivars and different geographical origins. *European Food Research and Technology, 228*, 735–742.

Li, R., Huang, J., Huang, L., *et al.* (2016). Comparison of GC and DSC monitoring the adulteration of camellia oil with selected vegetable oils. *Journal of Thermal Analysis and Calorimetry, 126*(3), 1735–1746.

Mallamace, D., Vasi, S., Corsaro, C., *et al.* (2017). Calorimetric analysis points out the physical-chemistry of organic olive oils and reveals the geographical origin. *Physica A: Statistical Mechanics and its Applications, 486*, 925-932.

Malvis, A., Simon, P., Dubaj, T., *et al.* (2019). Determination of the thermal oxidation stability and the kinetic parameters of commercial extra virgin olive oils from different varieties. *Journal of Chemistry,* Article ID 4567973.

Mansor, T. S. T., Che Man, Y. B. & Shuhaimi, M. (2012). Employment of differential scanning calorimetry in detecting lard adulteration in virgin coconut oil. *Journal of the American Oil Chemists' Society, 89*(3), 485–496.

Marikkar, J. M. N. & Rana, S. (2014). Differential scanning calorimetry to detect canola oil (Brassica napus L.) adulterated with lard stearin. *Journal of Oleo Science, 63*(9), 867–873.

Marikkar, J. M. N., Dzulkifly, M. H., Nadiha, M. Z. N., *et al.* (2012). Detection of animal fat contaminations in sunflower oil by differential scanning calorimetry. *International Journal of Food Properties, 15*(3), 683–690.

Nur Azira, T. & Amin, I. (2016). Advances in differential scanning calorimetry for food authenticity testing. In: Advances in Food Authenticity Testing, Elsevier Inc., pp. 311–335.

Pereira, L.H., Pereira, J., Garcia, J.S., *et al.* (2023). Seed oil detection in extra virgin olive oil by differential scanning calorimetry. *Journal of Thermal Analysis and Calorimetry, 148*, 6833–6843.

Rajagukguk, Y. V., Islam, M., Grygier, A., *et al.* (2023). Thermal and spectroscopic profiles variation of cold-pressed raspberry seed oil studied by DSC, UV/VIS, and FTIR techniques. *Journal of Food Composition and Analysis, 124*: 105723.

Rajagukguk, Y. V., Utcu, M. A., Islam, M., *et al.* (2022). Authenticity assessment from sesame seeds to oil and sesame products of various origin by differential scanning calorimetry. *Molecules, 27*(21): 7496.

Sumara, A., Stachniuk, A., Olech M, *et al.* (2023). Identification of sunflower, rapeseed, flaxseed and sesame seed oil metabolomic markers as a potential tool for oil authentication and detecting adulterations. *PLoS ONE 18*(4): e0284599.

Tan, C. P. & Che Man, Y. B. (2000). Differential scanning calorimetric analysis of edible oils: Comparison of thermal properties and chemical composition. *Journal of the American Oil Chemists' Society*, *77*(2), 143–155.

Tomaszewska-Gras, J. (2016). DSC coupled with PCA as a tool for butter authenticity assessment. *Journal of Thermal Analysis and Calorimetry*, *126*:61–68.

Ye, Q. & Meng, X. (2022). Highly efficient authentication of edible oils by FTIR spectroscopy coupled with chemometrics. *Food Chemistry*, *385*: 132661.