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Review of the doctoral dissertation
'Physicochemical, thermal, and spectroscopic characterization of oils from berry seed by-products in terms of authenticity assessment"
by Yolanda Victoria Rajagukguk, M.Sc.
written under the supervision of prof. dr. hab. Jolanta Tomaszewska-Gras

Formal comments

The basis for this review is the sesolution of the Scientific Council of the Food and Nutrition Technology Faculty of Poznań University of Life Sciences adopted on October 25, 2025, pursuant to which I was appointed as a reviewer of the above-mentioned doctoral dissertation. According to the letter by prof. UPP dr hab. Dorota Cais-Sokolińska, Chairwoman of the Scientific Council of the Food and Nutrition Technology Faculty, the dissertation submitted for review falls within the discipline of food and nutrition technology. The dissertation meets the formal requirements for this type of work presented in the procedure for the obtaining a doctoral degree.

Doctoral Candidate Profile

The Doctoral Candidate completed her Master's degree in 2017 at Surya University. In the years 2017–2023, she undertook several research internships at universities such as Prince of Songkla University, the Czech University of Life Sciences Prague, the University of Bologna, and the Free University of Bozen–Bolzano. In 2021, she commenced doctoral studies at the Borderland University in Poznań. The Doctoral Candidate has obtained three research grants, two of which were awarded under the Young Academic Staff research funding scheme of the Poznań University of Life Sciences, and the third being a PRELUDIUM 22 grant funded by the National Science Centre (NCN). The research conducted as part of the evaluated doctoral project was financed by an NCN OPUS grant (2018/31/B/NZ9/02762) as well as by a PRELUDIUM 22 grant (2023/49/N/NZ9/00861) awarded to the Doctoral Candidate.

Structure and organization of the dissertation

The structure of the dissertation submitted for evaluation is typical of modern doctoral theses based on previously published research results. The doctoral dissertation consists of a description of the scientific achievement written in English and a collection of six English-

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language publications that constitute this achievement. The first part of the dissertation comprises 40 pages and contains a list of publications included in the doctoral dissertation, information on research funding, a table of contents, a list of abbreviations, abstracts in English and Polish, and a description of the achievement. This description consists, in particular, of: an introduction addressing issues related to the management of fruit industry-waste, problems associated with the adulteration of edible oils, and methods for their detection; the rationale for selecting the topic, a scheme of the research conducted; the objectives and five detailed hypotheses; materials and methods; a discussion of the results based on six papers constituting the identified scientific achievement; aspects of novelty and the limitations of the conducted research; conclusions; bibliography; a list of tables; a list of figures.

The second part of the doctoral dissertation consists of a series of six original publications, thematically coherent, published in English-language scientific journals. All of these papers were published in 2023–2025 in reputable journals such as: *Foods*, *Journal of Food Composition and Analysis*, *NFS Journal*, *Journal of Food Engineering*, and *LWT*. The final paper was included in the doctoral dissertation in manuscript form; although it had already received positive reviews, it had not yet been formally published at the time of submission. Nevertheless, it was published in December 2025 in the *Journal of Food Composition and Analysis*. At the end of the second part of the dissertation, statements by all co-authors of the publications included in the dissertation, and from the Doctoral Candidate regarding her individual contribution to the research and to the preparation of the publications, were added.

The literature listed in the Bibliography section contains 33 references. The cumulative Impact Factor for the publication cycle was 29.0, and the total score according to MEiN amounted to 720 points (including the last publication after the final allocation of points). All publications forming the basis of the dissertation were multi-authored, and the Doctoral Candidate was the first author in each. The publications included in the cycle are very carefully prepared, and their graphical presentations are appropriately selected and well constructed.

The research conducted as part of the evaluated doctoral dissertation was funded by an NCN OPUS grant (2018/31/B/NZ9/02762) and a PRELUDIUM 22 grant (2023/49/N/NZ9/00861) awarded to the Doctoral Candidate.

Substantive evaluation of the doctoral dissertation

The topic undertaken by the Doctoral Candidate is highly engaging both from a cognitive and an application-oriented perspective. Ensuring product authenticity is essential for both consumers and producers, as it supports fair market competition. The identification of threats arising from food adulteration relies on advanced chemical-composition analysis, physicochemical monitoring of quality parameters, and correct interpretation of analytical data. Effective enforcement of food law, especially in the area of product adulteration, requires analytical methods suitable for use by official food-control authorities.

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In the Abstract section, the Doctoral Candidate explains the main objective of the doctoral dissertation very clearly and accessibly—to evaluate the suitability of DSC (Differential Scanning Calorimetry), FTIR (Fourier Transform Infrared), and UV-Vis methods, combined with chemometric techniques (including data fusion and low-level profile analysis) for authenticity assessment, classification, and botanical-origin differentiation of berry-seed-pressed oils, considering extraction method and raw-material quality variability.

The Introduction section provides a well-constructed and informative entry point into the dissertation topic. The Doctoral Candidate outlines the research scope, emphasizing the importance of food-industry waste management. Berry seeds, treated as an industrial by-product, are presented as a valuable raw material for edible oil production, rich in functional bioactive components. The candidate also includes a concise literature overview of the use of DSC and FTIR in seed-pressed oil authentication. The innovative aspect of the research lies in the use of full thermal and spectroscopic profiles (non-targeted analysis), rather than only isolated marker features. This section is complemented by a research-process scheme, enabling the reader to follow the research stages:

1. Method optimization;
2. Physicochemical characterization;
3. Authenticity confirmation studies for berry-seed-pressed oils.

The research scheme is clear and logically structured.

The following section, **Research and Hypotheses**, defines the main objective: to develop a fast and reliable authenticity-assessment method for oils pressed from raspberry, strawberry, and blackcurrant seeds, based on physicochemical, thermal, and spectroscopic profiles. The candidate specifies the following objectives:

1. Thermal and spectroscopic method optimization (Article 1, Article 2);
2. Authentic-oil physicochemical characterization (Article 3);
3. Authenticity assessment across variability factors: 1) Storage time (*Article 4*), 2) Botanical origin (*Article 5*), 3) Extraction method (*Article 6*).

Yolanda Rajagukguk, M. Sc., formulates five hypotheses:

H1) Seed freshness (*Article 1*) and the clarification process (*Article 2*) affect thermal and spectroscopic oil properties;
H2) Authentic berry-seed-pressed oils exhibit differentiating physicochemical properties (*Article 3*) that can be used as reference indicators for authenticity assessment;
H3) Electronic-nose volatile-compound profiling enables storage-time differentiation (Article 4);
H4) Differential scanning calorimetry (DSC) and Fourier-transform infrared spectroscopy (FTIR) are suitable for the rapid commercial oil-authenticity screening (Article 5);
H5) Different extraction methods (e.g., cold pressing, n-hexane extraction, and supercritical CO_2 extraction) result in differentiating thermal and spectral oil profiles (*Article 6*).
The objectives and hypotheses are correctly formulated.

In the **Materials and Methods** section, the Doctoral Candidate describes sample origin and preparation. The study used oils extracted from 9 batches of seeds (3 per fruit species:

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raspberry, strawberry, blackcurrant) and 38 commercial oils (10 blackcurrant, 18 raspberry, 10 strawberry). Seeds were ground into powder prior to extraction, using three methods: cold pressing with or without filtration (yielding oils with or without seed particles), n-hexane extraction, and supercritical CO_2 extraction.

During the storage experiment, oils were kept for 0, 3, 6, 9, and 12 months at room temperature, in daylight, simulating consumer use conditions. Among physicochemical parameters, acid value, peroxide value, anisidine value, content of conjugated dienes and trienes, antioxidant potential (DPPH method), and color were determined. The dissertation also used differential scanning calorimetry, gas chromatography (GC-FID for fatty acid composition, GC E-Nose for volatile compounds), liquid chromatography (for tocopherol content), as well as spectroscopic methods: UV-Vis and FTIR. For data analysis, the candidate employed numerous statistical methods such as analysis of variance with Tukey's post-hoc test, principal component analysis (PCA), multiple linear regression (MLR), partial least squares regression (PLS-R), partial least squares discriminant analysis (PLS-DA), and soft independent modelling of class analogy (SIMCA). Additionally, in one of the publications included in the cycle (*Article 6*), low-level data fusion of data from DSC and FTIR methods was applied. The statistical methods were well chosen and highly diverse and this aspect of the dissertation is evaluated very highly.

In the **Research Findings** section, the results of the conducted research are presented in a concise and synthetic form. In the papers published in *Foods (Article 1*) and in the *Journal of Food Composition and Analysis* (*Article 2*), the Doctoral Candidate verifies the first hypothesis (H1). For this purpose, oil was pressed from raspberry seeds that had expired 0, 10, and 20 months earlier, after which the anisidine value and DPPH antioxidant activity were determined, as well as thermal-property profiles obtained using DSC. Storage time had a statistically significant effect on the physicochemical parameters measured. The clarification stage involving the removal of seed particles also proved important for the stability of the oil obtained. It was demonstrated that the complete removal of seed residues contributes to reduced oil stability. A key conclusion from this research is that the stage of full seed-residue removal may be omitted.

In the next publication (*Article 2*), Yolanda Rajagukguk, M. Sc., examined the usefulness of DSC as well as UV-Vis and FTIR spectroscopy for differentiating laboratory-pressed raspberry-seed oils from commercial oils. In several samples, the results clearly diverged from those of the other samples within the same group—for example, the FTIR spectrum of one laboratory-pressed oil differed significantly from the other two. When reading this paper, a question naturally emerges regarding the authenticity of the purchased oils and the purity of the seed material used for pressing. Given the small number of samples, it is difficult to conclude which sample or samples should be considered true outliers. Certain doubts also arise when analyzing the fatty acid composition, as the profiles of two samples differ from those of the other four.

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In *Article 3*, the second hypothesis (H2) was verified. Oils pressed from raspberry, blackcurrant, and strawberry seeds were examined for physicochemical properties. Storage studies were also conducted, and parameters were measured after 0, 3, 6, 9, and 12 months of storage. Oils pressed from berry-fruit seeds proved very stable, and only minor changes in physicochemical parameters were observed during storage. However, distinct differences in physicochemical parameters were observed for oils originating from seeds of different botanical species.

The studies described in *Article* 4 allowed for the verification of the third hypothesis (H3). Analysis of volatile compound profiles using the GC E-nose method enabled differentiation of oil samples stored for 0, 3, 6, 9, and 12 months. Partial least squares regression was applied to estimate storage time. After limiting the variables introduced into the regression model, a strong RPD value of 5.7 was obtained, indicating high predictive ability and the reliability of the model.

The fourth hypothesis (H4) was verified in the studies described in *Article 5*, which were conducted on commercial oils from raspberry, blackcurrant, and strawberry seeds. It was demonstrated that both the DSC and FTIR methods are useful for classifying oils pressed from berry fruit seeds. In future studies, it would be advisable to use a larger and more diverse set of representative samples.

The studies conducted within *Article 6* allowed for the verification of the fifth hypothesis (H5). Oils extracted from the same seed batches using three methods—cold pressing, n-hexane extraction, and supercritical CO_2 extraction—were examined using DSC and FTIR methods. FTIR profiles enabled the differentiation of oils extracted using different methods, while revealing only subtle differences among oils pressed from different species. By contrast, the DSC method enabled the differentiation of oils from different botanical species but not from different extraction methods. The profiles obtained from the different extraction methods therefore contained complementary information. It supported the use of low-level data fusion, which enabled the accurate classification of the oils examined using the SIMCA method.

In the section **Novelty and Limitations of the Research**, the Doctoral Candidate emphasizes the novel aspects of the conducted studies, stating:

1. The use of complete DSC profiles and FTIR spectra for further statistical analysis.
2. The application of low-level data fusion combining DSC and FTIR measurements.
3. A comprehensive approach to the assessment of the authenticity of berry-seed oils.

I concur with the Doctoral Candidate's view that this represents a new research area and an original methodological approach. Oils derived from berry-fruit seeds are a niche product; however, yet they are likely to gain popularity owing to their richness in nutritional and functional components, and due to the possibility of producing them from fruit-processing by-products. The use of full DSC profiles and FTIR spectra is a highly original approach to the statistical use of measurement data. The shift from targeted analyses toward non-targeted

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analyses enables significant time savings and more effective use of measurement data, avoiding the loss of relevant information.

It is noteworthy that the Doctoral Candidate employed non-targeted methods in a well-considered and scientifically mature manner. Where necessary, the raw measurement data were subjected to preliminary statistical processing, which reduced noise and limited excessive dimensionality. This contributed, among other results, to a reduction in model-prediction errors. One example is the VIP algorithm (Variable Importance in Projection) used in in *Article 5* for variable selection, which improved the model's predictive performance. Data fusion combining DSC and FTIR strengthened their advantages while mitigating their limitations, confirming that these techniques provide complementary information.

The strong scientific awareness demonstrated by the author is evidenced by her recommendation to include a larger and more diversified sample set in future models, including intentionally adulterated oils (e.g., with rapeseed or soybean oil), and to explore a broader spectrum of chemometric techniques. The final remarks of the dissertation, presented in the **Conclusions** section, are accurate, concise, and clearly highlight the main scientific contributions. They align with the results discussed across the six-paper cycle. The **Research Findings** section presents the outcomes for each publication individually.

The most important achievements of the discussed doctoral dissertation include:

1. Demonstrating that it is possible to use complete DSC profiles and full FTIR spectra—without the need to analyze their characteristic parameters—for drawing conclusions regarding the quality of oils obtained from berry fruit seeds.
2. Applying low-level data fusion for DSC and FTIR measurement data and demonstrating the complementarity of these methods.
3. Providing evidence that that it is not necessary to implement a clarification step to remove seed particles from pressed oils, as non-clarified oils exhibit slower aging processes.
4. Demonstrating that berry-seed oils are highly durable and thermally stable products, with a high content of antioxidant compounds. The analysis of volatile compounds was identified as a method supporting the estimation of approximate storage time.
5. Highlighting the suitability of DSC and spectroscopic methods for confirming the authenticity of commercial berry-seed pressed oils.

In summarizing the research conducted by the Doctoral Candidate, I wish to emphasize its significant cognitive and practical value. The publications and the dissertation narrative are written in clear, correct, and highly accessible language. The dissertation demonstrates a wide spectrum of analytical and statistical methods. Together with the importance of the topic, the reliability of execution, and the clarity of presentation, this results in my very high overall evaluation of the work submitted for review.

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Questions Concerning the Doctoral Dissertation

While reading the doctoral dissertation, several issues drew my attention. I would like to ask the Doctoral Candidate the following questions:

1. What was the main reason for using a relatively small number of laboratory-pressed seed-oil samples, given that the candidate herself notes that a larger and more diversified sample set would improve model universality?
2. What premises supported the selection of low-level rather than, for example, mid-level data fusion?
3. Were commercial oils preselected for model inclusion, or was their authenticity assumed a priori without empirical screening?

These questions do not in any way affect my highly positive evaluation of the doctoral dissertation and are posed only to stimulate academic discussion.

Final conclusion

In conclusion, I confirm that the doctoral dissertation submitted for review by **Yolanda Rajagukguk, M. Sc.**, entitled ***"Physicochemical, thermal, and spectroscopic characterization of oils from fruit seed by-products in the context of authenticity assessment"***, provides an original and well-substantiated solution to an important scientific problem and meets the statutory requirements for doctoral dissertations under the Act on Academic Degrees and the Academic Title and Degrees and Titles in the Field of Art. Furthermore, it demonstrates the Doctoral Candidate's very broad disciplinary knowledge in food technology and nutrition and proves her ability to conduct independent scientific research and to interpret results, synthesize, and critically discuss results.

The dissertation fulfills all requirements stipulated by the applicable legislation for doctoral theses Therefore, I respectfully submit to the High Council of the Scientific Discipline of Food Technology and Nutrition at the Poznań University of Life Sciences a motion to admit Yolanda Rajagukguk, M. Sc., to the subsequent stages of the doctoral procedure.

The high scientific standard of the research also motivates me to recommend that the dissertation be distinguished within the Faculty of Food Science and Nutrition of Poznań University of Life Sciences, in accordance with the faculty's doctoral-distinction procedures.

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