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Dr hab. Ewa Ostrowska-Ligęza prof. SGGW
Department of Chemistry, Institute of Food Sciences
Warsaw University of Life Sciences

THE REVIEW

Doctoral dissertation mgr Yolandy Victorii Rajagukguk
„Physicochemical, thermal and spectroscopic characterisation of oils from berry seed by-products in terms of authenticity assessment"

Completed under the supervision of:
Prof. dr hab. Jolanta Tomaszewskiej-Gras
Department of Food Technology of Plant Origin
Faculty of Food Science and Nutrition
Poznań University of Life Sciences

Szkoła Główna
Gospodarstwa
Wiejskiego w Warszawie

Instytut Nauk o Żywności

ul. Nowoursynowska 159 C
02–776 Warszawa
+48 22 593 75 10
inoz@sggw.edu.pl
www.sggw.edu.pl

Seeds of berry fruits are a by-product that can be reused to enhance the final product or to obtain high-quality oil for use in the food industry or other branches of industry. Fruit pomace is characterized by the presence of bioactive compounds, fiber and pectins, which currently allows it to be used as a food additive or processed into valuable animal feed. Fruit seeds are a component of pomace. The pomace also contains remnants of fruit pulp, peels, stems, and leaves. As a by-product, fruit seeds can represent a valuable raw material in the food industry. Oil obtained from these seeds is characterized by a high content of polyunsaturated fatty acids. Another advantage of such oil is its high content of bioactive compounds as well as the distinctive and unique composition of the polysaccharide fraction. Demand for vegetable oils extracted from berry seeds is constantly growing. These oils are used not only as a food ingredient (dietary

supplements and nutraceuticals) but also in the cosmetics industry. Unfortunately, detailed regulations regarding the quality of these oils have not yet been developed, which exposes consumers to unfair practices related to their production, storage, and distribution. The doctoral dissertation of Ms. M. Sc. Yolanda Rajagukguk perfectly responds to the market demand for obtaining such oils of strictly defined quality and verified authenticity.

The doctoral dissertation submitted for evaluation consists of a series of six publications, also accompanied by an abstract, research objectives and hypotheses, a detailed scope of research and research methods, as well as an overview and discussion of the results. Based on extensive research conducted, the PhD Student formulated 9 conclusions, grouping them according to the established objectives. The dissertation also includes a list of all scientific publications with UPP affiliation (13) and scientific communications (5) authored by the doctoral candidate.

In the reviewed doctoral dissertation, Ms. M.Sc. Yolanda Rajagukguk presented the main aim.

The main aim of her research was to develop rapid and reliable methods for assessing the authenticity of berry seed oils (raspberry, strawberry, blackcurrant) based on their physicochemical, thermal and spectroscopic profiles.

- The main aim was verified based on specific objectives and working hypotheses:
- to optimise thermal and spectroscopic methods for authenticity assessment of berry seed oils
- to characteristic the physicochemical properties of authentic berry seed oils
- to assess the authenticity of berry seed oils differing in storage time, botanical origin from commercial samples and extraction methods.
- Following the aims, the research hypotheses of the study were divided into the following points:
- H1: Seed freshness, clarification process and analytical conditions affected thermal and spectroscopic properties of berry seed oils.
- H2: Authentic berry seed oils possess distinctive physicochemical properties that can be used as reference markers for authenticity.
- H3: Berry seed oils differing in storage times can be distinguished through E-nose volatile profiling.
- H4: Differential scanning calorimetry (DSC) and Fourier transform infrared spectroscopy (FTIR) are feasible for rapid authenticity screening of commercial berry seed oils.

- H5: Berry seed oils from different extractions (i.e. cold-pressing, n-hexane, supercritical CO_2) exhibited distinguishable thermal and spectroscopic profiles.

The PhD Student presented the results of the research in a series of six articles published in scientific journals (all of them were published in journals with a citation index in the JRC – IF database):

P1 – Rajagukguk, Y. V., Islam, M., Tomaszewska-Gras, J. (2023). Influence of Seeds' Age and Clarification of Cold-Pressed Raspberry (*Rubus Idaeus L.*) Oil on the DSC Oxidative Stability and Phase Transition Profiles. Foods, 12(2), 358.

P2 - Rajagukguk, Y. V., Islam, M., Grygier, A., Tomaszewska-Gras, J. (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.

P3 - Rajagukguk, Y. V., Islam, M., Grygier, A., Siger, A., Rudzińska, M., Tomaszewska-Gras, J. (2024). Physicochemical Characteristic of Cold-Pressed Blackcurrant-, Strawberry- and Raspberry Seed Oils During Storage and Its Influence on Thermo-Oxidative Stability. NFS Journal, 37, 100195.

P4 - Rajagukguk, Y. V., Cevoli, C., Grigoletto, I., Tomaszewska-Gras, J. (2024). Rapid Determination of the Storage Time of Cold-Pressed Berry Seed Oil Using Flash Gas Chromatography E-Nose Coupled with Chemometrics. Journal of Food Engineering, 364, 111795.

P5 - Rajagukguk, Y. V., Tomaszewska-Gras, J. (2025). Advanced Authenticity Screening of Commercial Berry Seed Oils Using Full FTIR Spectra and DSC Curves Coupled with Chemometrics. LWT, 117680.

P6 - Rajagukguk, Y. V., Ryszczyńska, S., Grygier, A., Siger, A., Waśkiewicz, A., Tomaszewska-Gras, J. (2025). Data Fusion of DSC Melting Curves and FTIR Spectra in Relation to Physicochemical Properties for Distinguishing Berry Seed Oils by Various Extraction Methods. Journal of Food Composition and Analysis, 148(3), 108475.

The PhD Student was the first author of six articles. The content of the doctoral dissertation (six publications) constitutes extensive documentation of the candidate's work. Ms. M.Sc. Yolanda Rajagukguk, has mastered a very broad research methodologies, what deserves special recognition and has contributed to ensuring the high quality and reliability of the scientific research. This allows for a high assessment of the PhD Student's knowledge, experience, ability to solve research problems, use of the

scientific literature, and interpretation of results.

The first stage of the study examined seed freshness, the clarification process, and the conditions for thermal and spectroscopic analysis. It was investigated whether these parameters affected the thermal and spectroscopic properties of berry seed oils. Cold-pressed oil from three batches of raspberry seeds was examined. Analyses were performed on freshly pressed oils and after 10 and 20 months of storage. Lower seed quality and the oil clarification process negatively impacted antioxidant properties and oxidative stability. DSC phase transition profiles did not show differences depending on the clarification process. A large number of thermal analyses were performed with varying heating and cooling rates (1; 2 and 5 °C/min).

In connection with the large number of analyses conducted, I have the following question:
What considerations justified performing such a large number of thermal analyses?

Ms. M. Sc. Yolanda Rajagukguk stated that the optimal heating rate was 5 °C/min, while the cooling rate was 2 °C/min. Please provide a detailed and comprehensive justification for such choice. UV-VIS spectroscopic analyses were also performed, followed by FTIR. The conclusion that UV-VIS is a less useful method is already known, as this analysis is unreliable in the study of complex matrices such as oils.

Based on the results of FTIR analyses, it is reasonable to use NMR (nuclear magnetic resonance) spectroscopic techniques to analyse oils.

Is the use of the NMR method justified for expanding analyses related to the study of oil authenticity?

In the next stage of the study, the physicochemical properties of raspberry, strawberry, and blackcurrant seed oils were assessed. The highest levels of carotenoids, chlorophylls, and α-linolenic acid were observed in blackcurrant seed oil. Raspberry seed oils were characterized by the highest antioxidant activity (DPPH), tocopherol content, and thermo-oxidative stability (OIT). The highest content of α-linolenic acid was observed in strawberry seed oil. During the first year of storage, all oils obtained from berry fruit seeds exhibited a high OIT (Oxidative Induction Time) value, despite a decrease in antioxidant content. The PhD Student used a Flash GC E-Nose system combined with PLSR models to assess changes in volatile compound profiles, which allowed for the determination of oil storage time. The use of this technique greatly enriched the dissertation.

Conventional extraction methods are time-consuming, energy-intensive, and require organic solvents. A PhD Student extracted oil from the seeds of three plant species (raspberries, strawberries, and blackcurrants) using cold pressing, n-hexane extraction, and supercritical CO_2 extraction. Obtaining FTIR

spectra and thermal profiles of the extracted oils allowed to determine the extraction method used. This data will allow for the assessment of the "clean label" of commercial oils. The final stage of the doctoral dissertation involved assessing the authenticity of oils from berry fruit seeds. PLS-DA models were based on DSC and FTIR data, these models were optimized and demonstrated effectiveness in classifying commercial oils according to their botanical origin. A new "DSC-FTIR" dataset was created through a novel approach of combining data obtained using two different instrumental analysis methods. A rapid classification was performed using the SIMCA model using a combination of DSC-FTIR data. The use of the model based on the DSC-FTIR dataset enabled significantly better classification results for oil types and extraction methods compared to approaches using single analytical methods.

Considering the quality of the doctoral dissertation presented by Ms. M. Sc. Yolanda Rajagukguk and the originality of the results obtained, I hereby recommend that the dissertation be awarded distinction.

A significant and innovative achievement of this dissertation is the use of comprehensive data obtained from DSC and FTIR analyses and their combination with chemometrics. Previously, data obtained from melting and crystallization profiles or DSC thermal diagrams were used on a point-by-point basis. The analysis included onset and maximum temperatures, enthalpies, and the intensity of phase transitions (maximum peak heights). The PhD Student used complete datasets, which shortened the time required to analyse the obtained results, expanded the set of data utilized, and reduced the number of interpretational errors. Combining full DSC thermal data with FTIR spectroscopic data enabled the evaluation of the oil extraction method from berry fruit seeds. Based on this, it is possible to assess the solvent used (e.g., n-hexane), allowing for the assessment of the purity of the commercial berry seed oil label. Ms. M. Sc. Yolanda Rajagukguk used thermal, spectroscopic, and physicochemical data analysis using chemometrics to assess the authenticity of the studied berry seed oils. The development of this model can be used for oil authenticity studies, which will enable the establishment of appropriate standards regarding the quality of oils from berry seeds and other oilseed plants. These achievements were published in very good periodicals (Foods, Journal of Food Composition and Analysis, LWT, Journal of Food Engineering and NFS Journal), which all have high impact factors (IF). An additional strength of the doctoral dissertation is the very broad scope of the research and the great care taken in preparing the articles that comprise the dissertation.

This is the first study of such topic and represents a significant contribution to knowledge about assessing the authenticity and quality of berry seed oils. Ms. M. Sc. Yolanda Rajagukguk's doctoral thesis expertly combines instrumental analysis of oils with statistical methods for processing analytical data. The

doctoral thesis contains minor editorial errors, such as the use of the singular instead of the plural but this does not detract from the overall value of the doctoral dissertation.

FINAL CONCLUSION

I declare that the doctoral dissertation submitted for review fully meets the requirements for doctoral theses and I submit to the Scientific Council of the Discipline of Food Technology and Nutrition of the Poznań University of Life Sciences a request to admit Ms. M. Sc. Yolanda Rajagukguk to the further stages of the doctoral procedure.

The research results obtained by Ms. M. Sc. Yolanda Rajagukguk constitute a significant contribution to the field of food science and nutrition. The information gathered is highly valuable and greatly expands knowledge regarding the possibilities for assessing the quality and authenticity of oils obtained from berry seeds. The information obtained, in addition to its cognitive value, also has practical applications. The doctoral dissertation was prepared correctly and with great methodological care, utilizing numerous analytical methods, chemometric tools and statistical analyses. Taking the above into account, I am submitting a request for the award of distinction to doctoral dissertation.

Ewa Ostrowska-Ligęza