Assessing The Performance of NIR Spectroscopy in the Quantification of Polyphenols in Grape Seed and Other Plant Extracts

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Grape seed extract (GSE) as a plant-based dietary supplement is a highly valued commodity on the market, due not only to its well-proven health benefits, but also its contribution to waste management and circular economy as a product of agri-food industry byproduct valorization. The absence of rapid, cost-effective and sufficiently selective quality control methods capable of characterizing it on the market lead to misclassification, batch to batch variability and economically motivated adulteration.

This thesis aimed to assess the performance of Near-Infrared Spectroscopy (NIRS) combined with chemometric modeling as a non-destructive, rapid and green analytical alternative for quantifying polyphenols and detecting adulterants in GSE.

Authentic samples of pure GSE, and other proanthocyanidin rich, extracts such as pine bark (PBE), peanut skin (PSE) and green tea (GTE) were prepared along with binary, dual and

ternary adulterated mixtures at ten concentration levels, in line with the industrial practice of adulteration. The pure samples were analyzed using Antioxidant Activity (AA, DPPH), Total Polyphenol Content (TPC, Folin-Ciocalteu) assays and High-Performance Liquid Chromatography (HPLC) for specific phenolic profiling. All samples underwent NIR spectral acquisition with a benchtop NIR XDS instrument, recording spectra within the 400–2500 nm wavelength range. The collected spectral data was subjected to wavelength range selection (1100–2250 nm), pre-treatments and multivariate analysis including Principal Component Analysis (PCA) and Partial Least Squares Regression (PLSR).

The initial chemical characterization revealed distinct and complex chemical profiles between GSE and its adulterants, where GTE showed exceptionally high TPC and AA while PBE was uniquely rich in catechin. The results of univariate statistical comparison highlighted the structural diversity and possible overlapping bioactivity of polyphenolic compounds.

PCA results of NIRS effectively visualized group separations and spectral variance associated with GSE concentration. PLSR models achieved strong prediction performance for extract concentrations, TPC, AA and individual phenolic compounds (namely gallic acid, catechin and epicatechin), with high coefficients of determination (R^2 >0.9) and low prediction errors. The models identified very specific spectral fingerprints for PBE, GTE concentrations (R^2P > 0.98), and AA (R^2P> 0.99, RMSEP=0.03 μmol TE/g).

The findings in this thesis confirm that NIRS, supported by chemometrics, with applicable predictive models can be a viable method for polyphenol quantification and adulteration detection in GSE and other plant extracts. Its rapid, solvent free, and scalable nature makes it an attractive tool for possible future developments to improve quality control strategies in the food industry, especially the dietary supplement sector.