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ADVANCED CHARACTERIZATION OF PLUM FRUITS (*PRUNUS DOMESTICA* L.) BY FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) AND SCANNING ELECTRON MICROSCOPY (SEM): IMPACT OF APPLIED TREATMENTS ON QUALITY AT DIFFERENT PHENOLOGICAL STAGES

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Abstract

This study investigated plum cultivar 'Centenar' (Prunus domestica L.) from an orchard located in north-eastern Romania, using high-precision techniques such as Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The research aimed to characterize the structural and chemical dynamics of the fruits during three phenological stages: early growth phase (BBCH 71), ripening phase (BBCH 74), and maturity (BBCH 89), in relation to the use of conventional methods and treatments. FTIR analysis allowed the identification of chemical composition variations, particularly in the content of polysaccharides, pectins, and phenolic compounds, illustrating the impact of treatments and growth stage on the biochemical profile. In addition, SEM allowed a detailed observation of fruit microstructure, revealing differences in cell integrity, a factor correlated with mechanical durability and post-harvest keeping capacity. This study's results provide particularly valuable insights into the interactions between the applied treatments and fruit quality, contributing to the development of optimized agricultural strategies for plum cultivation under specific pedoclimatic conditions.

***Key words**: Prunus domestica L., biochemical profile, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), phenological stages, pedoclimatic conditions.*

INTRODUCTION

Prunus domestica L. (European plum) is a significant species valued for its nutritional, medicinal properties, and economic importance. *Prunus domestica* L. has a rich content of bioactive compounds, including phenolics, flavonoids, and essential minerals. Plums are the most numerous and diverse fruittree species, making them important for current and future development due to their variety, widespread distribution, and adaptability (Arion et al., 2014)

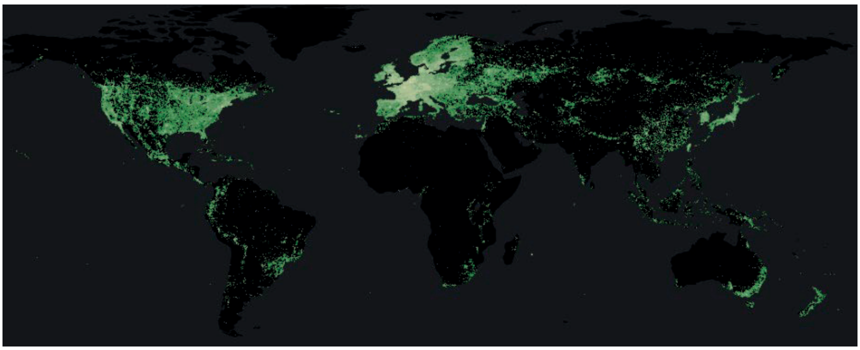

Figure 1. Distribution of *Prunus domestica* L. throughout the world (https://www.gbif.org/species/7931731)

In addition to being enjoyed as fresh fruit globally, it is also cultivated commercially consumed in dried forms and as various processed additives, finding multiple uses in the food industry (Xu et al., 2024).

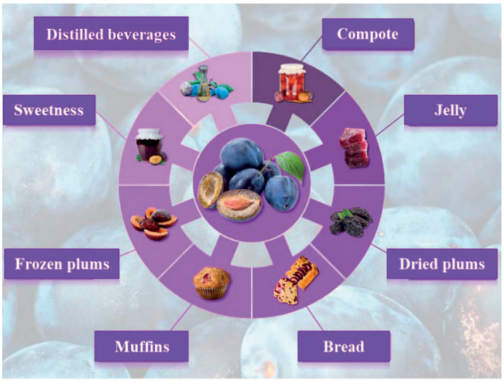

Figure 2. The versatility of plums in the food industry

Plum quality and composition are influenced by a complex interplay of factors, including soil composition, environmental conditions, and agricultural practices (Calistru et al., 2024). During phenological development, the fruit undergoes dynamic biochemical and structural changes that impact its quality and sustainability. Understanding its biochemical profile across key phenological stages - early growth phase, ripening phase, and maturity - is critical for optimizing cultivation practices. Conventional agricultural methods often employ generalized approaches that may fail to account for the dynamic biochemical and structural transformations occurring during these stages, potentially compromising fruit quality. These attributes are shaped by inherent developmental processes and specific conventional agricultural treatments, such as synthetic fertilizers, pesticides, and region-specific pedoclimatic conditions (Rakonjac et al., 2024).
In north-eastern Romania, plum cultivation is a cornerstone of local agriculture, optimizing yield requires a detailed understanding of how soil composition, environmental conditions, and agricultural practices interact across phenological stages (Cara et al., 2025). Specifically, it is critical to elucidate the biochemical and structural changes that occurduring each stage of fruit development.

This study, conducted in a north-eastern Romanian orchard, employs advanced analytical tools - Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM) - to evaluate plum fruits. FTIR spectroscopy provides insights into the molecular composition of the fruit, enabling the identification of functional groups such as hydroxyl (-OH), carbonyl (C=O), and carboxyl (-COOH) associated with carbohydrates, proteins, lipids, and phenolic compounds. For instance, absorption bands around 3300 cm^{-1} and 1600 cm^{-1} correspond to O-H stretching and C=O stretching vibrations, characteristic of phenolic acids and flavonoids. Complementing FTIR, SEM provides high-resolution imaging of the fruit's microstructure, particularly the mesocarp, which determines texture, juiciness, and overall quality (Kaur et al., 2023**)**. SEM reveals details about cell arrangement, cell wall thickness, and intercellular spaces, enabling the identification of variations in parenchyma cell morphology. For example, the presence of large, thin-walled cells with abundant intercellular spaces is associated with a soft and juicy texture. Additionally, SEM detects microstructural changes caused by ripening, storage, or mechanical damage, such as cell wall breakdown or the formation of microcracks (Basiak et al., 2019).
Traditional methods for assessing fruit quality are often time-consuming and destructive. In contrast, FTIR and SEM offer non-destructive, rapid, and reliable alternatives that provide detailed information about the elemental composition and microstructure of plum fruits (Geng et al., 2020). By integrating these techniques, this study aims to enhance our understanding of the biochemical and structural changes in plums across phenological stages, ultimately contributing to improved cultivation practices and postharvest management in north-eastern Romania.

MATERIALS AND METHODS

Research area

The research was conducted at Adamachi Farm - Iaşi University of Life Sciences (47°15`N, 27°30`E), located on the Moldavian Plateau in northwest Romania. The study area is characterized by a strongly anthropogenic landscape, with an average altitude of 80-95 m. According to the Koppen climate classification, the region is characterized by a humid subtropical climate C_{fa}, with an annual average temperature of approximately 10°C and an annual rainfall of about 518 mm (https://climateknowledgeportal.worldbank.org/country/romania, accessed on 20 December 2024)

Orchard Management and Agronomic Practices

The orchard was established in 2014 using a randomized block design with three replicates. Each replicate consisted of 30 plum trees. Post-emergent, non-selective herbicides were applied to manage weed competition. Specifically, fluazifop-P-butyl (150 g/L) and isopropylamine salt (360 g a.i. L^{-1}) were administered in the spring at a rate of 2,880 mL/ha (1.39 kg a.i. ha^{-1}) to reduce competition for water and nutrients during the active growth phase of the plum trees.

Fertilization was carried out using NPK 16-16-16 fertilizer from Ameropa Company® (Targu Mures, Romania). The fertilizer was applied at a rate of 425 kg/ha, following the manufacturer's recommended doses for plum cultivation. Applications were conducted during four phenophases: BBCH 01, BBCH 60, BBCH 69, and BBCH 73, ensuring compliance with industry standards.

Collection and preparation of samples

Fruit samples of the 'Centenar' variety were collected at different growth stages - BBCH 71 and BBCH 74 - and at full maturity in August 2023 (BBCH 89). The collection process was conducted with utmost precision to ensure accuracy and reliability in the analysis. The composite samples were stored in polyethylene bags and cleaned with ultrapure water to eliminate any residual chemicals and debris. After cleaning, the samples were dried under mild conditions (25-30 degrees, protected from sunlight), and the skin and pulp were manually separated. The pulp was freeze-dried (BIOBASE BK-FD10T equipment, Jinan, China) at −42°C and 0.10 mbar for 48 hours until reaching a moisture content of 7% (MF-50 moisture analyser, A & D Company, Tokyo, Japan). The freeze-dried pulp was then ground using MC 12 equipment (Stephan, Germany), and stored in zip-lock bags covered with aluminum foil at room temperature for future use.

Research Methodology

The identification and analysis of aliphatic, phenolic, aromatic, and carbonyl functional groups through infrared spectroscopy facilitate the distinction of the chemical composition phenological stages of the plum fruit powder. For these analyses, an Interspec 200-X FTIR spectrometer was employed.

The morphology, structure, and elemental composition of the plum fruit powder were investigated using a scanning electron microscope (Quanta 450, FEI, Thermo Fisher Scientific, Hillsboro, USA) in conjunction with an energy dispersive X-ray detector (EDX) (EDAX, AMETEK Inc., Berwyn, PA, USA). Each sample was applied on a thin layer of approximately 2 mm onto aluminium stubs with the help of double-sided carbon tape, the stubs being subsequently placed within the analysis chamber. The EDX spectral analysis was conducted using EDAX Inc.'s TEAM version V4.1 system. The samples were analysed under low vacuum conditions, with a magnification of 500× (10 μm), an electron acceleration voltage of 15 kV, and a pressure of approximately 6.1×10^{-4} Pa.

RESULTS AND DISCUSSIONS

The IR spectra of plum fruits, selected at three different stages of maturation (BBCH 71, BBCH 74, BBCH 89), were obtained to analyse variations in the nature of functional groups, illustrating changes in chemical composition throughout fruit development.

The spectra obtained in different stages of maturation are illustrated in Figure 3. The IR spectra of the fruits at all three stages of development were more or less similar.

The characteristic bands observed in the 3600-4000 cm^{-1} region are associated with the vibration of hydroxyl functional groups, and the absorption was caused by the stretching of free or H-bonded OH groups (Thirumavalavan et al., 2011). This functional group (-OH stretching) is present in all stages of fruit maturity and can be attributed to the presence of water (after the lyophilisation process) or polyphenols.

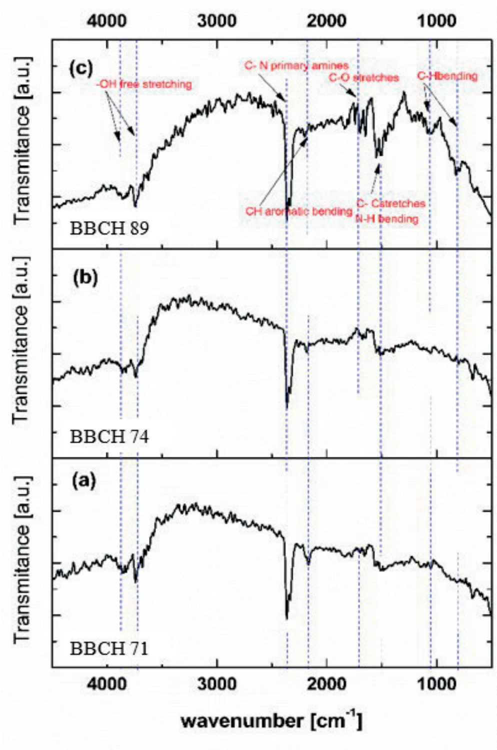

Figure 3. FT-IR spectra of plum fruit at BBCH 71 (a), BBCH 74 (b), BBCH 89 (c)

A decrease in transmittance in this region at the BBCH89 stage indicates a reduction in water content or a modification of polar compounds due to maturation processes. Characteristic C-H vibrational peaks (from aliphatic chains, such as lipids or sugars) are present at all stages, the most intense specific band being observed in BBCH 89. The relatively constant intensity suggests the stability of hydrocarbon structures, but subtle changes reflect the accumulation of sugars or fatty acid degradation.

The absorbance at ~1700 cm^{-1} (C=O vibrations) is significant, indicating the presence of carbonyl groups from organic acids, esters or ketones. An increase in this peak can be observed in BBCH89, corresponding to the accumulation of reducing sugars or volatile compounds in the baking phase.

For wave numbers below 1500 cm^{-1}, one can observe that the so-called fingerprint area of the spectra is a complex one, associated with bending and coupling vibrations and shows marked differences between stages. The modifications may be related to cellulose/pectin degradation (peaks at ~1200 cm^{-1}) or to the synthesis of phenolic compounds (peaks at ~1600 cm^{-1}).

FTIR spectra reflect the biochemical evolution of plum fruit during ripening. Stages BBCH 71 (early growth) and BBCH 74 (intermediate ripening) show compositions richer in water and polar compounds, while BBCH 89 (advanced ripening) indicates accumulation of secondary metabolites (e.g., phenolics, esters) and reduction of structural components. These changes are consistent with natural fruit transformation processes, including cell wall degradation and synthesis of compounds responsible for flavour and texture.

In addition, the intense band below 1000 cm^{-1} shows higher cellulose content during growth, observed better in BCCH 89 (Sanchez-Rivera et al., 2010).

The changes in the microstructure of the plum fruit with respect to growth and development were imaged using SEM and are illustrated in Figure 4. With the advancement of the maturity stage, the microstructure drastically changed with irregular shapes and larger intercellular areas. The appearance of the sample corresponding to the early development stage (Figure 4a) looked hazy with no particular prominent structure. With the progress of maturity (BBCH 74) some microstructures (Figure 4b) were observed. The difference in the micro-appearance is also related to the variation in chemical compositions. SEM images at stages BBCH 71 and BBCH 74 clearly revealed the microstructure of starch and carbohydrates.

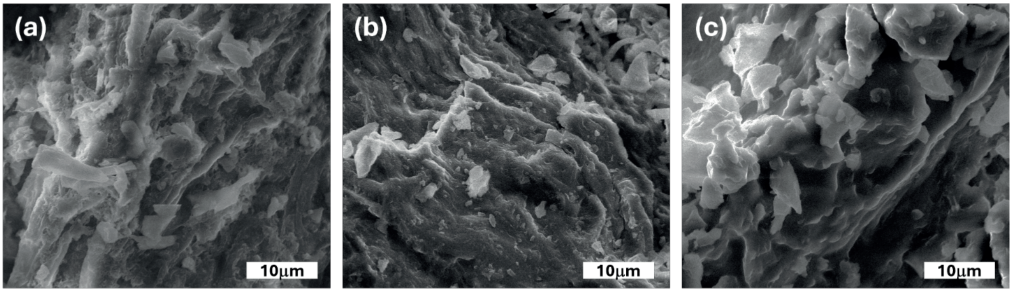

Figure 4. SEM images of plum fruit growth stages at BBCH 71 (a), BBCH 74 (b), BBCH 89 (c)

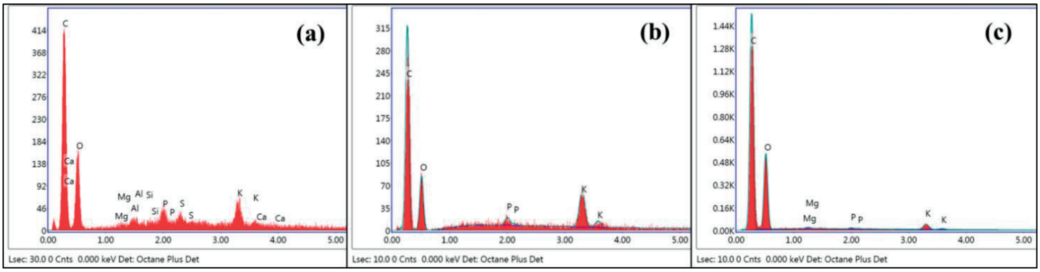

Figure 5. EDX elemental composition of plum fruit at BBCH stages 71 (a), 74 (b), and 89 (c)

EDX analysis (Figure 5) revealed that in the analysed samples, the main elements are C, O, P, and K. K was the most abundant mineral present in all stages, followed by P and Mg. EDX shows the presence of the K element in all three samples (a, b, d). In the case of sample (c), the analysis showed a decrease in the intensity of the peak corresponding to potassium. Also, for sample (a) an individualisation of the intensity of the peak corresponding to calcium was observed, with the rest of the elements identified in samples (b) and (c) being detected in negligible percentages. The composition not only varies during phenophases, but also between different varieties (Motyleva et al., 2017).

CONCLUSIONS

The present study was focused on evaluating the effect of fruit maturation on biochemical and functional compounds in the plum fruits at various stages. The results revealed that this is a potential source of many functionally important nutritional and bioactive compounds. K was the most abundant mineral present in all stages, followed by phosphorous and Mg. FT-IR analysis confirmed the presence of various functional groups and indicated the complex nature of the plum fruits. SEM clearly evinced that the microstructure of the fruits changes drastically, and the degradation of starch and other compounds occurred at the fully matured stage. The SEM images highlight the complex structure, which reflects the quality of the lyophilization process and the potential applications of the powder in the food or pharmaceutical industry (BBCH 89).

Hence, the present study favourably justifies that the plum fruits have enormous potential for commercial application as a source of nutritional and functional compounds which can add a higher value to this locally important crop.

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