

Dissertation abstract

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Title: „Multiscale analysis of the influence of technological parameters of the natural deep eutectic solvents extraction process on the properties of the fruit pomace pectin”

The increasing importance of technologies aligned with the principles of sustainable development and green chemistry supports the exploration of efficient methods for recovering valuable compounds from waste biomass. Fruit pomace, generated as the by-product of the food industry, is recognized as a good source of bioactive compounds, including pectins with a wide range of applications in food, pharmaceutical and cosmetic industries. The functional properties of pectins are determined by their chemical structure, including galacturonic acid (GalA) content, the degree of esterification, and domain structure. Conventional pectin extraction methods, based on acid hydrolysis at elevated temperatures, are associated with significant environmental and technological limitations. An alternative approach involves Natural Deep Eutectic Solvents (NADES), which can serve as effective and environmentally friendly extraction media.

The main objective was to optimize the pectin extraction process from fruit pomace with the use of NADES and to evaluate the impact of technological parameters on the properties of the obtained products. The research was conducted using fruit pomace of blackcurrant (model raw material), raspberry, strawberry and sea buckthorn.

The series of preliminary extractions was performed in water, in selected media with varying pH and ionic strength, and in NADES systems, which enabled the selection of choline chloride (ChCl) and citric acid (CA) mixture as the most advantageous for this type of biomass. The crude extracts were purified via methanol precipitation and dialysis, then analyzed spectroscopically to determine the content of polysaccharides, including uronic acids, as well as polyphenols and proteins.

Process parameters were optimized using Design of Experiments (DoE) and Response Surface Methodology (RSM). The independent variables included the NADES mixture composition (ChCl, CA) and the extraction time. Based on statistical analysis (ANOVA) and RSM-based predictions within an I-optimal experimental design, the optimal parameters were identified and subsequently applied to the remaining fruit pomaces. The products obtained

under optimal conditions were purified by ion-exchange chromatography (IEC), and the dominant pectin fractions were characterized using chromatographic (LH-20, HPLC-SEC, GC-MS), spectroscopic (FT-IR, ^1H NMR, ^1H - ^{13}C HSQC NMR), and microscopic (SEM) techniques.

Pectin fractions, regardless of the biomass type, exhibited similar chemical characteristics, differing in the proportions of their sugar components. Products were characterized by a high content of GalA and the presence of neutral sugars (Gal, Rha, Glc, Xyl), which influenced their structural organization, particularly the type of side chains. LH-20 analysis indicated the presence of polyphenol-polysaccharide complexes forming conjugates with pectin, while HPLC-SEC confirmed the medium- and high-molecular-weight nature of these products. FT-IR spectra revealed the low-methoxyl character of the dominant fractions, and NMR analyses confirmed the presence of HG and RG-I predominant domains. SEM observations demonstrated differences in surface morphology between the crude and purified fractions.

The use of optimal parameters (ChCl/CA 1.5/2; 4 h) for blackcurrant pomace resulted in a yield of 4.3% (w/w) and a GalA content in polysaccharide chains of approximately 55%. For raspberry and sea buckthorn pomace (ChCl/CA 1.5/2; 4 h) and strawberry (ChCl/CA 2/1.5; 4 h), the yields obtained were 5.6%, 7.75%, and 4.33%, respectively, with GalA content of 64.1%, 77.75%, and 54.5%. Purification by IEC was associated with an increase in the GalA content.

The obtained results confirm that NADES provide an effective and environmentally friendly alternative to conventional pectin extraction methods, enabling control of the structural properties of products through the appropriate selection of process parameters.