ANNOTATION

of the dissertation submitted for the degree of Doctor of Philosophy (PhD)
in the educational program
8D07101 – "Technological Machines and Equipment"
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Improving the efficiency of extraction equipment based on the development of modes for extracting valuable components from grape waste

General characteristics of the work.

The dissertation is devoted to the development of batch extraction equipment employing molecular hydrogen and to the substantiation of rational operating parameters for recovering bioactive components from grape processing waste. The effects of molecular hydrogen pressure, temperature, and process duration on the total content of phenolic and flavonoid components, monomeric anthocyanins, and the antioxidant activity of grape skin extracts were investigated. The study includes a mathematical description of mass transfer, engineering substantiation of equipment parameters, and a techno-economic assessment of the proposed solution.

Relevance of the research topic.

In accordance with the Concept for the Development of the Agro-Industrial Complex of the Republic of Kazakhstan for 2021–2030, improving the efficiency of agricultural raw-material processing and the rational use of secondary resources are among the priority areas. Grape pomace generated during wine and juice production is a promising source of bioactive components, including phenolic and flavonoid components as well as monomeric anthocyanins. However, conventional extraction methods involve lengthy processing times and offer limited opportunities for intensifying mass transfer, necessitating the development of efficient extraction equipment and the substantiation of rational operating conditions.

One promising approach to improving extraction efficiency is the use of molecular hydrogen. Nevertheless, the effect of molecular hydrogen pressure on the recovery of bioactive components from grape skins and the characteristics of the process in sealed extraction equipment have not previously been investigated. Therefore, developing extraction equipment with adjustable molecular hydrogen pressure and substantiating rational process parameters constitute a relevant scientific and technical challenge aimed at improving the efficiency of bioactive-component recovery from grape processing waste.

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Aim of the dissertation: To develop extraction equipment operating in a molecular hydrogen atmosphere to improve the efficiency of recovering valuable bioactive components from grape pomace, and to substantiate effective extraction operating conditions.

Research objectives.

1. To analyze existing methods and equipment for extracting bioactive components from grape processing waste and to substantiate the feasibility of using molecular hydrogen as a means of improving process efficiency.

2. To investigate the effects of extraction parameters on the recovery of bioactive components from grape skins, including a preliminary experimental evaluation of the effectiveness of molecular hydrogen in maceration, hydrodistillation, and supercritical CO_2 extraction, and to develop the design of extraction equipment equipped with a molecular hydrogen supply system.

3. To develop a mathematical description of the extraction of bioactive components from grape skins and to investigate the effects of technological parameters (pressure, temperature, and treatment duration) on polyphenol recovery using experimental extraction equipment.

4. To assess the techno-economic efficiency of recovering bioactive components from grape skins using the experimental extraction equipment.

Object of research. Extraction equipment for recovering bioactive components from grape pomace.

Research methods. The work employed theoretical and experimental methods to investigate the extraction of bioactive components from grape skins. The theoretical studies included mathematical modeling of extraction kinetics, analysis of mass-transfer patterns, and engineering calculations for the experimental extraction equipment. Experimental studies were conducted using the developed extraction equipment by varying molecular hydrogen pressure, temperature, and process duration. Extraction efficiency was evaluated using physicochemical and spectrophotometric methods for determining the contents of phenolic components, flavonoid components, and monomeric anthocyanins, as well as the antioxidant activity of the extracts, together with high-performance liquid chromatography.

Scientific novelty of the work. A design solution was developed for extraction equipment operating under adjustable molecular hydrogen pressure. The equipment incorporates a sealed working chamber and is intended for recovering bioactive components from grape skins (Appendix A). Based on experimental studies, rational operating parameters for the extraction equipment were determined. Specifically, at a molecular hydrogen gauge pressure of 5×10^5 Pa, a temperature of 35 °C, and an extraction duration of 1800 s, the contents of phenolic compounds, flavonoids, and monomeric anthocyanins in the extracts increased, as did the antioxidant activity indicators.

Main propositions submitted for defense:

–The use of a modified ethanol-based extractant saturated with molecular hydrogen increases the recovery of phenolic compounds (TPC), flavonoids (TFC), and monomeric anthocyanins (TMA) from grape skins, as well as the antioxidant activity of the resulting extracts as assessed by DPPH and ABTS.

–The sealed extraction equipment enables the recovery of bioactive compounds from grape skins through the use of an extractant saturated with molecular hydrogen and the establishment of a hydrogen atmosphere under excess pressure.

Practical significance of the work.

The practical significance lies in the development of a design solution for extraction equipment and the substantiation of its operating conditions. Experimental data were obtained on the effects of molecular hydrogen pressure and operating parameters on the recovery of bioactive components from grape skins. The results can be used to develop grape-pomace processing technologies and to design extraction equipment for the wine and juice industries. The resulting extracts are considered potential ingredients for food products.

The reliability of the results was ensured through the use of physical, chemical, and spectrophotometric analytical methods; investigation of the process under controlled operating parameters; mathematical processing of experimental data; and comparison of the data with model relationships.

Validation of the practical results.

The principal propositions and results of the dissertation were presented and discussed at an international scientific and practical conference and at meetings of the Department of Bioengineering Systems of Shakarim University.

The practical results of the dissertation research were validated at the Research Institute of Food Safety of Almaty Technological University JSC (Republic of Kazakhstan) and at the Center for the Development, Application and Research of Innovative Food Technologies of Iğdır University (Republic of Türkiye), as confirmed by the relevant validation certificates.

Personal contribution of the author.

The author’s personal contribution includes the analysis of scientific and technical literature; planning and conducting experimental research; development of the extraction equipment design; substantiation of operating parameters; processing

and interpretation of the results; preparation of a mathematical description of the extraction process; and a techno-economic assessment.

Publications.

The main results of the dissertation have been published in 6 scientific works, including 1 (one) article in a scientific journal indexed in the Web of Science/Scopus international databases (Q2, CiteScore 2025 percentile: 75); 2 (two) articles in scientific journals recommended by the Committee for Quality Assurance in Science and Higher Education of the Ministry of Science and Higher Education of the Republic of Kazakhstan; and 1 (one) paper in the proceedings of an international scientific and practical conference. In addition, 1 (one) Republic of Kazakhstan utility model patent was granted (No. 11184, registration No. 2025/1115.2), and 1 (one) invention patent application was filed (No. 2026/1111.1).

Structure and scope of the dissertation.

The doctoral dissertation consists of an introduction, 5 chapters, and a conclusion, presented on 174 pages of computer-typed text, including appendices. The work contains 48 figures, 17 tables, a list of references comprising 163 entries, and 6 appendices.

Assessment of the completeness of the research objectives.

The results obtained are consistent with the aim of the dissertation and support the conclusion that all stated objectives were successfully accomplished:

1. Existing methods and equipment for extracting bioactive components from grape processing waste were analyzed, and the technological limitations of conventional extraction methods were identified. The feasibility of using molecular hydrogen to intensify the extraction process and improve the recovery efficiency of bioactive components was substantiated.

2. The effectiveness of molecular hydrogen in maceration, hydrodistillation, and supercritical CO_2 extraction, as well as the effects of pressure, temperature, and treatment duration on the recovery of bioactive components, was experimentally investigated. When 4% molecular hydrogen was used during supercritical CO_2 extraction, the phenolic compound content increased from 21.84 to 33.75 mg GAE/100 mL. Rational parameters for grape skin extraction were substantiated: adjustable molecular hydrogen gauge pressure of 5×10^5 Pa, temperature of 35 °C, process duration of 1800 s, solid-to-liquid ratio of 1:20, and a 70% aqueous ethanol solution as the extractant. Under these conditions, the following values were achieved: TPC, 2.04 mg GAE/g; TFC, 0.68 mg QE/g; TMA, 263.61 mg C3G/g; DPPH, 3.29 mg TE/g; ABTS, 39.01 μmol TE/g; and FRAP, 0.180 mg GAE/g. In addition, a batch extraction equipment design was developed, featuring a molecular hydrogen supply system and allowing the adjustment of process parameters.

3. A mathematical description of the mass-transfer process occurring in the "solid plant matrix–liquid extractant" system under molecular hydrogen was developed. Extraction kinetics were described using the effective volumetric mass-transfer coefficient kf a, and its dependence on pressure, temperature, and treatment

duration was determined. Studies conducted using the experimental equipment showed that these parameters affect the rate of polyphenol recovery from grape skins and the extent of their transfer into the extractant. Based on the results, the main kinetic patterns of the process were identified, and the agreement between the mathematical model and the experimental data was assessed. This made it possible to substantiate rational operating conditions for the equipment.

4. The techno-economic efficiency of using the developed extraction equipment was assessed. At an annual production volume of 837.31 kg of extract, projected revenue amounted to KZT 26.79 million, capital investment to KZT 6.51 million, and the annual financial result, assuming 95% ethanol recovery, to KZT 7.10 million. The calculated payback period of the equipment was 0.92 years, indicating the economic feasibility of the proposed technological solution.