chemistry proceedings

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Abstract

Optimizing the Enzymatic-Assisted Extraction of Aromatic Compounds from Red Wine Lees †

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† Presented at the 17th International Symposium "Priorities of Chemistry for a Sustainable Development" PRIOCHEM, Bucharest, Romania, 27–29 October 2021.

Keywords: aromatic compounds; red wine lees; enzymatic extraction; optimization

In this research, we aimed to optimize the enzymatic-assisted extraction of volatile oily compounds from red wine lees, which give cognac flavor to the wine. By recovering the wine lees byproducts, the following can be obtained [1]: wastewater, ethanol, antioxidants, tartrate (as calcium tartrate and tartaric acid) and yeast cells. The chemical analysis showed that the volatile composition of wine lees [2,3] consists of esters, acids, alcohols, aldehydes, furanic compounds, terpenes and C13-norisoprenoids, whilst the phenolic composition [4] contains flavonoids and non-flavonoids. In the first stage, an enzymatic treatment was performed before the extraction of light oils using different concentrations of β-glucanase/pectinase from Novozymes (2500 PGNU/g Polygalacturonase and 75 BGXU/g β-glucanase (exo-1,3-), Novozymes, Bagsværd, Denmark), at different reaction temperatures and times. The treatment was performed to a mixture of 1.5/1 *w*/*w* wine lees aqueous sediment (from Ișalnița, Dolj county) and double-distilled water, under agitation. In the second stage, hydrodistillation of the mixture was performed using a Clevenger installation, but with prior removal of the first distillation head from the collecting tube, followed by recirculation of the azeotrope in the system at reflux until the substrate was depleted, at 141 °C in the heated bath for 4 h 20 min as previously reported [5]. The final concentrated distillate, rich in light volatile oils, was collected from the collecting tube, and the concentration of the main esters was determined using Gas Chromatography–Mass Spectrometry from calibration curves of a standard purchased from Sigma Aldrich (Merck Group, Darmstadt, Germany). In order to maximize the concentration of esters in the volatile oils extracted from wine lees, the optimization of the necessary enzymatic-assisted extraction conditions (concentration, temperature and time) was performed using Response Surface Methodology with the Design Expert v11 application (Stat-Ease, Minneapolis, MN, USA). The main esters present in the sample are ethyl octanoate, ethyl decanoate, ethyl dodecanoate and ethyl hexadecanoate. The concentrations of these major esters ranged from 0.158% to 0.481% in hydrodistillate. The final polynomial equation of the estimated coefficients was calculated using Analysis of Variance for the selected factorial model. The experimental values of ester concentrations of the volatile oily compounds are similar to the predicted values obtained using Response Surface Methodology. The extraction temperature and time had higher significance than the β-glucanase/pectinase concentration on the aromatic composition extracted from red wine lees. However, the enzymatic extraction treatment applied to the wine lees before the hydrodistillation process, increasing the concentrations by more than 30% according to the data analysis. We optimized the enzymatic-assisted extraction of volatile oily compounds from red wine lees. Applying an enzymatic pre-treatment with β-glucanase/pectinase before the hydrodistillation process,

Citation: Deșliu-Avram, M.; Radu, E.; Constantinescu-Aruxandei, D.; Oancea, F. Optimizing the Enzymatic-Assisted Extraction of Aromatic Compounds from Red Wine Lees. *Chem. Proc.* **2022**, *7*, 59. https://doi.org/10.3390/chemproc2022007059

Academic Editors: Mihaela Doni, Zina Vuluga and Radu Claudiu Fierăscu

Published: 22 March 2022

the wine lees releases higher ethyl ester concentrations, which are responsible for the aromatic smell and taste, than without the enzymatic pre-treatment, which represents an encouraging process.

Author Contributions: Conceptualization, F.O.; methodology, M.D.-A. and E.R.; investigation, M.D.-A., E.R., D.C.-A.; project administration, M.D.-A.; supervision, F.O., D.C.-A.; validation M.D.-A. and E.R.; visualization, D.C.-A.; writing—original draft preparation, M.D.-A.; writing—review and editing, D.C.-A. and F.O.; funding acquisition, F.O. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by project POC-A1-A1.2.3-G-2015-P_40_352-SECVENT, My_SMIS 105684, "Sequential processes of closing the side streams from bioeconomy and innovative (bio)products resulting from it", subsidiary project 1518/2019. The SECVENT project was co-funded by European Regional Development Fund (ERDF), The Competitiveness Operational Programme (POC), Axis 1.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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