

EXTRACTION OF PHENOLIC ACIDS FROM DISTILLERY STILLAGE

W. Mikucka, M. Zielinska, K. Bulkowska

UNIVERSITY OF WARMIA AND MAZURY IN OLSZTYN, POLAND

INTRODUCTION

The distillery industry is a key contributor to the development of the global economy, but it is also considered to be one of the world's major sources of environmental pollution. For 1 L of the spirit produced, 9 to 14 L of distillery stillage are produced. The distillery stillage is characterized by a high content of organic matter susceptible to biodegradation (COD varies between 15 and 176 g O₂/L). Therefore, it is very important to purify distillery wastes because they can cause serious environmental problems. However, more attention should be paid to the recovery of bioactive compounds from by-products from the distillery industry. Many factors influence the content of bioactive phenolic acids in distillery stillage. It mainly depends on the type and variety of raw materials used during the alcoholic fermentation, as well as the storage conditions and the method of processing. Additionally, an important element is a method of extracting phenolic acids. These bioactive compounds differ in terms of structure. Their chemical structure and interactions with other components are not fully known, and this is a very important aspect when choosing solvents and determining the conditions of the extraction process. For these reasons, the preparation of samples for extraction and the parameters of the process require special attention. The present study aimed at comparing the effect of different concentrations of solvents (ethanol, methanol) on the extraction of phenolic acids from distillery stillage.

MATERIALS AND METHODS

The recovery of phenolic acids from the stillage was carried out using ultrasonically assisted extraction. Distillery stillage from the production of concentrated unpurified ethyl alcohol from cereals (a company in north-east Poland) was used. The characteristics of the stillage is shown in Table 1.

Table 1. Characteristics of distillery stillage

Parameter	Unit	Average concentration (± standard deviation)
COD	mg/L	43600 (±1294)
Total nitrogen	mg/L	4345 (±386)
Ammonium nitrogen	mg/L	8.4 (±2.7)
Total phosphorus	mg/L	280 (±76)
Volatile fatty acids	mg/L	788 (±137)

Two experimental series of extraction were carried out.

METHANOL
(60%, 70%, 80%, 90%, 100%)

ETHANOL
(60%, 70%, 80%, 90%, 100%)

Chromatographic separation was performed by the High-Pressure Liquid Chromatography (HPLC) (Figure 1) fitted with a UV-Vis detector equipped with Supelcosil C18 column (150 mm × 4.6 mm, 5 µm) working at 35°C. A gradient elution program was employed, using 1 mL water/formic acid (99.85/0.15, v/v) (solvent A) and acetonitrile/formic acid (99.85/0.15, v/v) (solvent B) as elution solvents.

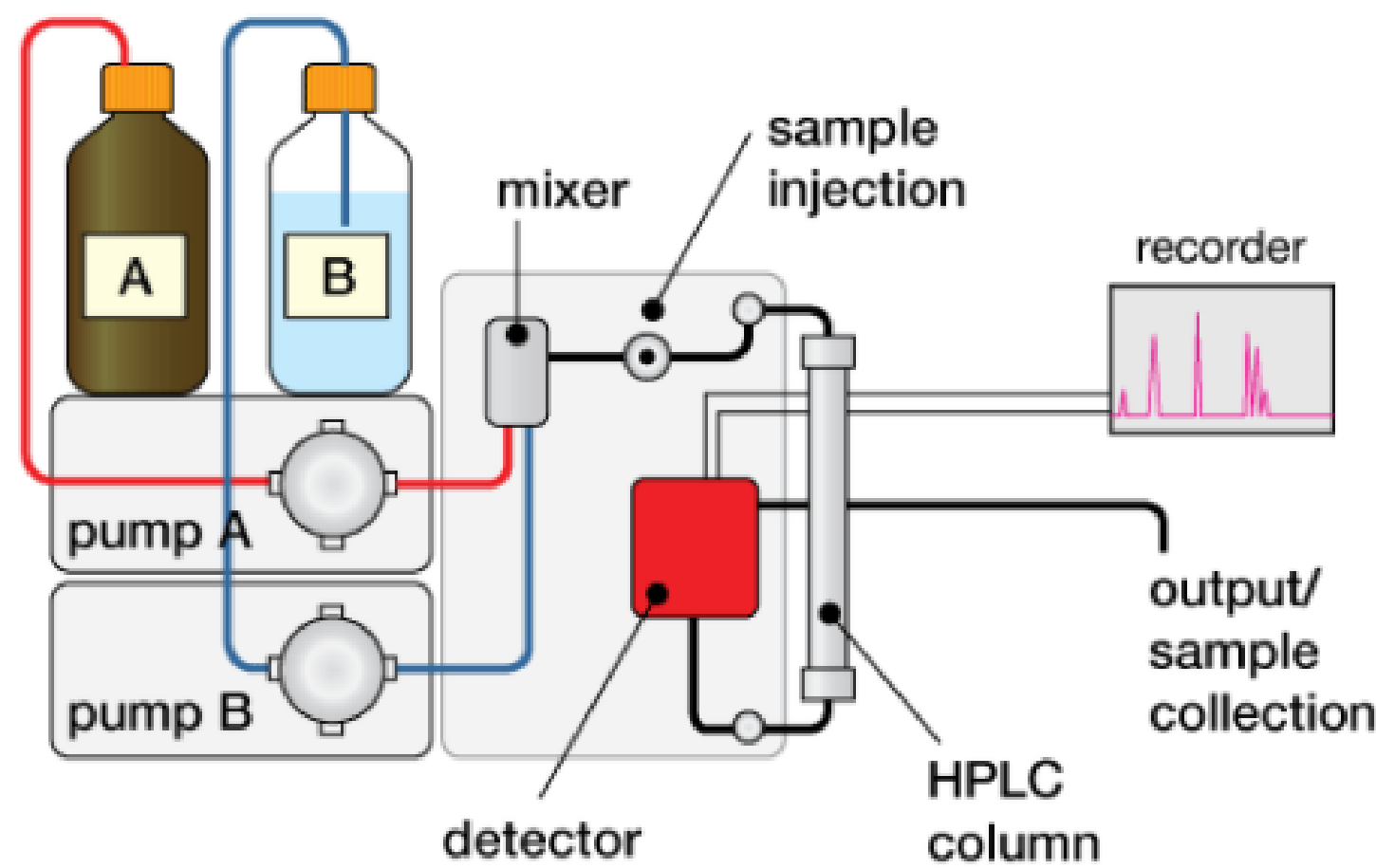


Figure 1. HPLC setup is diagrammed

The detection was performed at the wavelength of 260 nm (p-OH-benzoic, vanillic, syringic acid) and 320 nm (p-coumaric, ferulic, sinapic acid).

RESULTS

The phenolic acids present in the extracts were quantitatively characterized by HPLC. Figure 2 shows the total concentration of phenolic acids that were recovered from the distillery stillage using 60%, 70%, 80%, 90%, and 100% ethanol and methanol solutions.

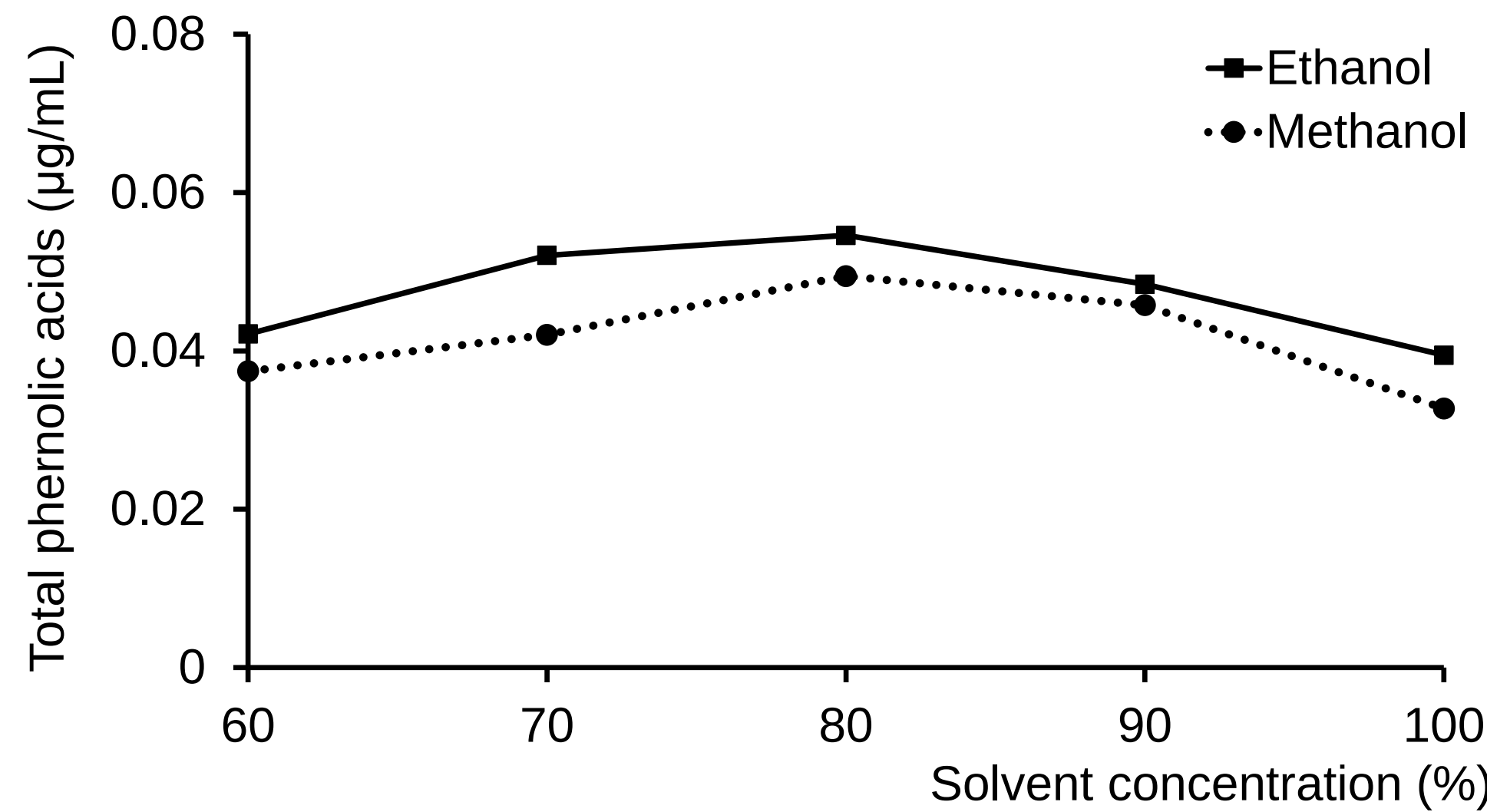


Figure 2. Total concentration of phenolic acids after extraction from the distillery stillage depending on the solvent concentration

The most effective solvent turned out to be an 80% ethanol solution; extraction efficiency reached 75%. After extraction, the concentration of total phenolic acids was 0.0546 µg/mL.

100% ethanol and methanol turned out to be the least effective solvents. Phenolic acid recovery from distillery stillage using 100% ethanol and methanol was 0.0394 µg/mL and 0.0327 µg/mL, respectively.

Both in ethanol and methanol extracts, p-coumaric acid was the phenolic compound of the highest concentration. Table 2 shows the concentrations of p-coumaric acid that were recovered from the distillery stillage using ethanol and methanol solutions.

Table 2. Concentrations of p-coumaric acid in distillery stillage

Concentration of p-coumaric acid (µg/mL)	Concentration of ethanol (%)				
	60	70	80	90	100
	0.0155	0.0175	0.0182	0.0169	0.0161
Concentration of p-coumaric acid (µg/mL)	Concentration of methanol (%)				
	60	70	80	90	100
	0.0150	0.0164	0.0172	0.0162	0.0124

The phenolic acid that was recovered from the distillery stillage in the smallest amount was sinapic acid. When using 100% ethanol, the concentration of sinapic acid was 0.00189 µg/mL, while when using 100% methanol it was 0.00138 µg/mL.

CONCLUSIONS

Distillery stillage is a source of polyphenolic compounds.

Optimizing the extraction method is important before reusing or managing the distillery stillage in the environment.

The extract produced may be labeled as a "natural product".

Optimization of the extraction process from distillery stillage performed in this work allows the production of high-value extracts without pretreatment, only using solvents available in any distillery.

From the ecological point of view, the direction of further research on the valorization of stillage should be the search for new techniques for recovering valuable polyphenolic compounds that meet the requirements of "green extraction".

REFERENCES

- Bustamante M.A., Pérez-Murcia M.D., Paredes C. et al. Bioresource Technology, 98, 3269-3277, 2009.
- Chandrasekar V., Martín-González M., Hirst P. et al. Journal of Food Process Engineering, 38, 571-582, 2015.
- Farcas A.C., Socaci S.A., Dulf F.V. et al. Journal of Cereal Science, 64, 34-42, 2015.
- Fito J., Tefera N., Kloos H. et al. Sugar Technology, 21, 265-277, 2019.
- Sun B., Leandro M.C., de Freitas V. et al. Journal of Chromatography A, 1128, 27-38, 2006.

CONTACT



WIOLETA MIKUCKA
Department of Environmental Biotechnology, University of Warmia and Mazury in Olsztyn
wioleta.mikucka@uwm.edu.pl