In the thesis, a valorization concept consisting of the multidirectional use of waste biomass from distilleries was proposed, which is commensurate with the circular economy principles in which waste is used as a raw material. This strategy allows for contributing to the sustainable development of the distillery industry and diminishing the environmental pollution resulting from distillery operations.

In this study, the recovery of bioactive compounds was proposed as an option for the valorization of distillery stillage coming from cereal processing to alcohol. Because the content of extracted polyphenols and the resulting antioxidant activity of the product depend on the substrate used for alcohol production and the method of recovery, there is a necessity to optimize extracting conditions for each waste material. Therefore, the objective of this study was to determine the potential of distillery stillage as a source of bioactive polyphenols, the possibilities of their recovery, and the effect of phenolic acids on methane production from distillery stillage. The studies included: i) extraction of polyphenols from the stillage with commercial organic solvents by using conventional solid-liquid extraction (CSLE), ultrasound-assisted extraction (UAE) and microwave-assisted extraction (MAE), and with water by using subcritical water extraction (SWE), ii) the use of tetrahydrofurfuryl alcohol (THFA) and biochar produced from the solid fraction of cereal processing waste (including distillery stillage) to recover phenolic acids from the liquid fraction of the stillage, and iii) investigating the effect of phenolic acids on the methane potential of distillery stillage.

It was shown that distillery stillage was a source of bioactive polyphenols. All the solvents used (ethanol, methanol, acetone, ethyl acetate, water) could be applied in the extraction of polyphenols from the stillage, but the yield and composition of polyphenols, extraction rate, and antioxidant activity as a measure of the extract quality depended on the type and concentration of the solvent and the method and conditions of extraction (solid-to-solvent ratio, time, temperature). Because of the more efficient recovery of polyphenols, higher antioxidant activity of the extracts and a significantly shorter time of extraction, MAE and UAE were found to be "green" alternatives to conventional extraction. Independently of the extraction technique, ethanol was the most effective solvent in recovering polyphenols. The use of SWE resulted in a high content of polyphenols in the extracts, with simultaneous elimination of organic solvents from the extraction process. The biomass remaining after SWE of polyphenols can be considered a valuable product – hydrochar. However, the antioxidant activity of the extracts after SWE was a few times lower than those obtained with ethanol. The extracts that were characterized by the highest antioxidant activities resulted from MAE with

80% ethanol, 80% methanol and 70% ethyl acetate. The addition of water into all organic solvents increased the content of polyphenols and antioxidant activity of the extracts.

Synergistic processing of different streams of cereal waste may be a promising direction in the waste-to-profit management of agro-food waste. It was investigated that not only commercial solvents but also waste-derived THFA and biochar were suitable for the recovery of phenolic acids from distillery stillage. The recovery efficiency depended on THFA concentration, biochar dose, and desorption temperature. The content of phenolic acids in the THFA extracts was higher than those obtained with acetone, ethyl acetate and water, similar to that obtained with methanol, and lower by about 7% than that obtained with ethanol. Adsorption/desorption of phenolic acids from the extracts allowed to maintain ~95% of the antioxidant activity.

In the extracts obtained with all extraction conditions and solvent types, polyphenols that were efficient at scavenging radicals predominated over those with a reducing power. Independently of the extraction conditions, phenolic acids were present in the extracts mainly in the free form, which allows to avoid hydrolysis of the stillage before the extractions. Among these acids, ferulic and *p*-coumaric acids (both belonging to hydroxycinnamic acids) predominated. Such characteristics of the extracts open various possibilities for the industrial applications of recovered phenolic compounds.

The negative effect of phenolic acids on the methane potential of distillery stillage strongly depended on the type of acid and its concentration. The presence of phenolic acids decreased the abundance of acetoclastic and hydrogenotrophic methanogens during the stillage fermentation. The obtained results concerning the inhibition constants of methane production with particular phenolic acids may be valuable for improving biogas production from wastes with different compositions and concentrations of bioactive compounds.