# Elemental Characterization of Brewery Waste Using Deep Eutectic Solvent and Determination by Spectrometry Techniques

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## Highlights

- A Comparison of different brewery waste pre-processing methods was carried out.
- Deep eutectic solvent was applied as extraction solvent of chemical elements prior to elemental determination by FAAS.

## Abstract

Several methodologies for sample preparation and elemental analysis are reported in the literature, with the aim of quantifying the most different chemical elements of nutritional and environmental relevance from the industries such as them of grains processing. However, these methodologies use toxic and problematic reagents (e.g. organic solvents or inorganic and concentrated acids). In this context, there is a need to propose new environmentally friendly methods, including the use of deep eutectic solvents (DES), which have mutable characteristics depending on the reagents used. Then, the propose is to evaluate different sample pre-processing (drying, grinding, in natura, freezing) combined with the use of DES to extract the analytes. In addition, the aim is also reducing the cost and energy consumption of the method and replacing problematic and environmentally aggressive reagents. The processed malt pomace samples were digested in a standard reference method using nitric acid and hydrogen peroxide under heating at 100 °C with stirring. Subsequently, the analytes were determined by inductively coupled plasma optical emission spectrometry (ICP-OES), for the determination of Cu, Fe, Mn, and Zn. The DES used in the extraction was prepared using choline chloride (ChCl) and oxalic acid (Ox) in a 1:1 molar ratio, heated in a microwave system in 30-second cycles with vortex stirring until a homogeneous and stable liquid formation. For the preprocessing of the bagasse, the following methods were evaluated: (i) freeze-drying followed by grinding in a knife mill; (ii) oven drying followed by grinding in a knife mill; (iii) oven drying followed by freezing in liquid nitrogen ($N_2$) and grinding in a mortar. These procedures were compared with the results obtained from the no processing sample. The extraction used 3 mL of DES for 450 mg of dry sample, heating for 30 s in a microwave and stirring in a Vortex, followed by dilution with ultrapure water (3 mL). After centrifugation, the supernatant was separated from the precipitate and diluted 10-fold prior to FAAS analysis. The standard calibration curves in aqueous medium and with matrix matching were prepared to evaluate matrix effects. It was found that there was no significant matrix interference in the results under the applied conditions. High recoveries rate were obtained when using the freeze-dried and crushed sample compared to the crude sample. The recoveries for the standard additions to the dried samples ranged from 70 to 96% and were considered adequate. The results show that the DES has high potential for extracting the analytes investigated. Moreover, the results show that the sample preprocessing is relevant and has a significant influence on the availability of the elements, which impacts the extraction efficiency.

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