

# Valorization of Agroindustrial Residues: Extraction of Bioactive Compounds for Sustainable Biorefineries

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## 1. Introduction

Since 2015, the United Nations established the Sustainable Development Goals (SDG), which involves ecology, economics and society aspects in order to achieve a more sustainable society (United Nations Development Program 2015). Therefore, industrial processes have been pushed to consider these aspects along their entire value-chain and since the design stage of new processes. Some of the approaches proposed to achieve a sustainable production have focused on energy and resource efficiency, as a shift into a bioeconomy requires the production of both energy and products. Among these, an intensive use of raw materials and the use of renewable feedstocks and biomass highlight due to the high amount of resources available from agroindustry, forestry, etc. These feedstocks may be used to obtain biomolecules and chemicals, biomaterials, biofuels, bioenergy and food/feed.

It has been estimated that the worldwide production of agricultural residues accounts for  $3.7 \times 10^9$  tons, demonstrating their potential as feedstocks to obtain different products (Bentsen, Felby, and Thorsen 2014). Cellulose, hemicellulose, sugars, lignin, extractives, among others, are some of the platforms that can be used from these feedstocks. Among the products that can be obtained, bioactive compounds highlight due to their application in fields as pharmaceuticals, health, nutrition and cosmetics (Oreopoulou and Tzia 2007). These are secondary metabolites that have the ability to interact with living tissues, aimed to have beneficial effects (National Cancer Institute 2019). In addition, the valorization of all possible fractions must be considered for the production process to be sustainable; this means, not only extracting the bioactive compounds, but also evaluating applications for the lignocellulosics and other platforms (Serna-Loaiza et al. 2019).

Based on the aforementioned elements, this work consisted in the extraction of bioactive compounds from three agroindustrial residues: wheat straw (WS), hemp residues (HE) and grape vineshoots (GVS). The initial step consisted in the characterization of the raw materials in order to be able to establish further mass balances. Nano-lignin, cannabidiol and flavonols (quercetin) were the focused bioactive compounds in the raw materials, respectively. Organosolv extraction was performed as the extraction method and the chosen solvent (water-ethanol mixtures and ethanol) and extraction conditions (temperature and time) were adapted to each raw material. Then, in order to evaluate the valorization of the remaining fractions after the extraction of the bioactive compounds, three different procedures were performed: sugar production, lignocellulosic characterization and fiber quality characterization. These procedures aimed to identify possible applications to produce substrates for further fermentations, identification of biogas production potential and application as pulp for paper production.

## 2. Materials and methods

### 2.1. Selected raw materials

The selection criteria were availability of the feedstock (WS), presence of bioactive compounds (HE) and the possibility of further valorization (GVS). For a more detailed description of the raw material selection, please refer to the study case presented in (Serna-Loaiza et al. 2019).

WS is the residue resulting from wheat crops, which is the most grown food crop around the world. This residue can be a source for the extraction and production of nano-lignin, which has been researched for its bactericidal, UV-blocking and antioxidant properties. The composition of wheat straw is approximately 63 %wt cellulose and hemicellulose, and 20 %wt lignin; this shows the potential to obtain a sugar platform that could be further used as substrate for a microorganism fermentation (Beisl et al. 2018; Weinwurm et al. 2017). HE is a source of cannabinoids—especially cannabidiol (CBD)—which have gained interest in pharmaceutical industry (Kitrytė, Bagdonaitė, and Rimantas Venskutonis 2018). CBD is contained mainly in the leaves/blossom, leaving the stem available for fiber and material applications, and the remaining solids after the extraction can be used to obtain sugars. GVS consist of the stem and leaves that are cut after grapes harvesting. They contain compounds of bioactive interest as flavonols (quercetin and myricetin) and inositol, among others. They are mainly contained in the leaves, and the lignocellulose in the stem that can be used for fiber, lignin, and sugar production.

### 2.2. Valorization of the residues: Characterization, extraction and further valorization

An initial separation of the parts of the feedstocks was performed. HE and GVS were manually divided into the leaves/blossoms and the stem. The raw materials were characterized in terms of the structural carbohydrates (glucan, xylan, arabinan, mannan and galactan), lignin (acid soluble and insoluble), ash, extractives and moisture, according to the NREL/TP-510-42618 (Sluiter et al. 2012).

Organosolv was performed in a 1 L stirred autoclave (Zirbus, HAD 9/16, Bad Grund, Germany) with a solid:liquid ratio of 1:10. Then, the solid and liquid fractions were separated with a hydraulic press (Hapa, HPH 2.5, Achern, Germany) at 200 bar and a centrifuge (Thermo Scientific, Sorvall, RC 6+, Waltham, MA, USA) at 30,074 g for 20 min. Table 1 shows the extraction conditions for each of the raw materials. The solid fraction was characterized in terms of the structural carbohydrates. The extracted bioactive compounds and sugars were characterized using HPLC, GC-MS and HPAEC.

**Table 1.** Organosolv conditions for the extraction of bioactive compounds.

Raw Material	Solvent	Temperature [°C]	Time [min]
Wheat Straw	60 %wt aqueous ethanol	180	60
Hemp	100 %wt ethanol	100	100
Grape Vineshoots	70 %wt aqueous ethanol	120	60

## Results and discussion

The characterization of the raw materials indicated the potential of the different fractions. HE-Flowers, GVS-Leaves, GVS-Stem and WS had lignin contents around 22 %wt, while HE-Stem only had 10 %wt. All raw materials have content of structural carbohydrates around 60 %wt, indicating the potential of producing sugars.

At the proposed conditions, it was possible to extract the bioactive compounds from the chosen raw materials. CBD extracts reached concentrations of 1,20 %wt. Nano-lignin concentration achieved values around 30 g/L. The extract from the GVS achieved concentrations of 25 µg/g of quercetin. These results indicate the potential of these agroindustrial residues to obtain bioactive compounds, sugar-based products, material based products and finally, an energy production scheme as biogas under a biorefinery scheme.

## Conclusions

As a main conclusion, it was observed that the chosen raw materials have potential in obtaining bioactive compounds that can become the economic drivers of a biorefinery. In addition, the further valorization of the feedstocks allowed broadening the range of products. Finally, the remaining solids can be used for energy production with potential for biogas production.

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