

Concentration of elements in woody and herbaceous biomass as a function of the dry ashing temperature

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Abstract

The main purposes of the study presented in this paper are to select the optimal temperature of dry ashing and to carry out posterior analyses of elements in the ash formed. The concentration of elements from biomass was compared with the concentration of elements in ash obtained at 400, 500, 550, 600 and 800 °C, utilising four woody and herbaceous biomasses: pine chips, poplar chips, thistle and wheat straw.

A dry ashing temperature in the 500–550 °C range was found to produce ashes low in organic carbon (less than 1 wt%) and to recover the greatest amount of the element contents present in the original biomass. A slight loss of potassium and sodium is determined in the ashes produced at temperatures as low as 400 °C for herbaceous biomass compared with the potassium and sodium analysed in biomass.

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1. Introduction

Lignocellulosic biomass is considered a renewable and CO₂ neutral energy resource, with a high potential for utilization in the future for purposes of heat and electricity generation. Inorganic matter is mainly found in plants as ions, salts and inorganic species bound to organic species. During the combustion, they are volatilised, oxidised, sulphated, etc. Consequently, the contents of elements and compounds change according to the dry ashing temperature. At incineration temperatures above 600 °C, the liberation of CO₂ will take place at the same time as the liberation of other volatile inorganic compounds such as sodium and potassium compounds. Many authors have reported the release of inorganic elements and compounds at high temperatures both in laboratories [1,2] and in combustion plants [3–5], where the importance of the volatilisation and reactions of elements such as potassium, sodium, sulphur and chlorine for ash melting behaviour, slagging, fouling and corrosion is well known.

The optimal temperature and the best process for dry ashing of lignocellulosic biomass are currently matters of debate and

investigation. Generally, the lignocellulosic biomass dry ashing is carried out in the laboratory at temperatures of up to 600 °C, as is shown in the norm for determining the ash content in wood [6], where 580–600 °C is the temperature range selected. The International Energy Agency (IEA) has recommended an incineration temperature of 550 °C for straw [7]. Researchers have tried to produce ash with the least variation in their elements and even in compounds by means of oxidation at low temperature in an oxygen plasma. However, Olanders [8] concluded that oxidation of the biomass at a low temperature (60–70 °C) by oxygen plasma produces ashes with high organic carbon content. Ash is defined as the mineral residue that remains when something is burned and, consequently, the oxidation at very low temperatures should be discarded as a mean to determine the ash content. Moreover, low temperature ashing using plasma produces compounds that are not characteristic of heating or combustion [3].

The results of the analysis of the chemical composition are strongly influenced by the method used, not only in the determination step, but also during the sample preparation and particularly the digestion step [9]. The analyses of elements in the biomass and/or in the ash formed in the dry ashing process are important parameters in biomass investigation nowadays [10]. The content in elements determined in straw ash such as potassium, sodium, calcium, magnesium, silicon, aluminium, phosphorus, etc. can be converted to a fuel basis, while the content in sulphur, chlorine and carbon, which results from

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carbonate, cannot [7]. The ash obtained in the dry ashing process is applied to other tests and analyses such as ash melting behaviour [10–12], characterisation of crystalline compounds in the biomass by X-ray diffraction [1,3,8,13], etc.

The main objectives of this work are to select the optimal dry ashing temperature for woody and herbaceous biomasses by obtaining the most representative ash of the original biomass and subsequently to carry out analyses of elements. This paper also expects to contribute to a better understanding of the chemical characterisation of lignocellulosic biomass and its ash.

2. Materials and methods

2.1. Materials

Four types of lignocellulosic biomasses were considered in this study. Two of them corresponded to herbaceous biomasses: wheat straw and thistle (*Cynara cardunculus*). The whole thistle plant was harvested. This plant has an important future use as solid fuel collected from energy crops due to its adaptation to semiarid climatic conditions, e.g. Mediterranean areas. The other two biomasses were woody biomasses: pine chips and poplar chips. They consisted of branches and leaves from tree pruning.

2.2. Methods

A scheme of the sample preparation is shown in Fig. 1. Firstly, the biomass sample was ground to pass a 2 mm sieve. Next, the homogenized sample was divided into eight fractions by means of the Jones divider, three fractions were again ground to pass a 0.5 mm sieve and the other five fractions were ashed at 400, 500, 550, 600 and 800 °C. The ash produced at each temperature was ground to pass a 0.25 mm sieve.

In contrast to the norms of determination of the ash content, which use 1–2 g per crucible, a dry ashing procedure was developed in a conventional laboratory furnace to produce a large amount of ash and to carry out posterior analyses of elements and other tests such as sintering tests, X-ray diffraction, etc. The amount of solid biomass placed in a porcelain capsule was about 40 g under controlled air atmosphere (10–20 l/min for six capsules). In these conditions, heating rates were set to try to avoid volatilisation or release of inorganic elements. These were:

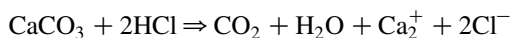
- *Period 1.* Heating rate of 10 °C/min from ambient temperature to 200 °C.
- *Period 2.* Heating rate of 1 °C/min from 200 to 300 °C.
- *Period 3.* Heating rate of 3 °C/min from 300 °C to selected temperature.

It is noted that between 220 and 270 °C, the furnace temperature increases without external heating of the furnace, due to of the fast exo-thermic reactions of lignocellulosic biomass combustion. Thus, a minimum heating rate of 1 °C/min from 200 to 300 °C was selected for two reasons, to reach a state of auto-combustion of the biomass where a strong release of volatile matter is observed (approximately, at 220 °C) and to reach Period 3. The heating rate in Period 3 (3 °C/min) is mainly a function of the scientific and normative recommendations of operating with heating rate low. Finally, the selected temperature was maintained until 24 h had passed for the purpose of reducing the organic carbon.

Chemical analyses of the following elements: Al, Ba, Ca, Fe, K, Mg, Mn, Na, P, S, Si, Sr, Ti and Zn contained in the biomass ash samples and biomass samples were carried out by atomic emission spectrometry (ICP) using a Thermo Jarrell Ash (IRIS AP) simultaneous spectrometer, after digestion of the samples in closed vessel. Samples of biomass and ash were prepared by acid digestion using HCl (only for ash samples), H₂O₂, HNO₃ and HF in a first step, and H₃BO₃ in a second step. These element determinations were carried out in the same day under repeatability conditions to minimise the uncertainty of measurement.

Chlorine was determined in the biomass ash following ASTM D-2361-66 [14], based on the combustion of the sample with the Eschka mixture and quantification of the chloride by indirect Volhard titration. Chlorine contained in the biomass was determined by ionic chromatography, using Metrohm equipment after the combustion of the sample in a calorimeter and lixiviation of the gases and ashes with a NaOH–H₂O₂ solution.

Total carbon was determined in the biomass ash by elemental analysis using a Fisons CHNS-1108 (Carlo Erba) equipment. The elemental analyser contains a tube furnace where the sample is burned, a chromatographic column and a thermal conductivity detector where the gases are separated and measured. The samples used to determine organic carbon were previously treated in a silver capsule with a HCl acid solution to liberate the CO₂ from the carbonates according the following reaction applied to, for example limestone, which is considered an abundant and natural compound found in the lignocellulosic biomass ashes:



Next, the residue was dried and introduced into the Fisons CHNS-1108 elemental analyser to measure the residual carbon residual or organic carbon, following ISO 10694:1995 [15]. The difference between total carbon and organic carbon is considered the inorganic carbon.

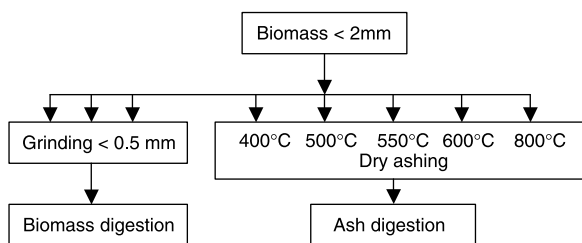


Fig. 1. Scheme of sample preparation.

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