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Evaluation of the combustion characteristics of raw and torrefied grape pomace in a thermogravimetric analyzer and in a drop tube furnace

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ABSTRACT

Grape pomace is a by-product of the wine making industry with great availability and energetic potential. Torrefaction is a pre-treatment that may enhance the biomass quality as a fuel, and consists in exposing the biomass to an inert atmosphere at a temperature between 200 °C and 300 °C. This study evaluates the combustion characteristics of raw and torrefied grape pomace in a thermogravimetric analyzer and in a drop tube furnace. Initially, the raw grape pomace was torrefied in an argon inert atmosphere at 260 °C. Subsequently, the combustion of the raw and torrefied grape pomace was examined in the thermogravimetric analyzer through non-isothermal runs at a heating rate of 10 °C/min from room temperature to 800 °C. Finally, the combustion of both biomass residues was evaluated in the drop tube furnace at 1100 °C. The data reported includes gas temperature, major gas species concentration and particle burnout measured along the axis of the drop tube furnace. The main conclusions of this study are (i) for the devolatilization stage, the thermogravimetric data yielded apparent activation energy values of 84.9 and 85.2 kJ mol⁻¹, and for the char oxidation stage of 137.5 and $109.2 \text{ kJ} \text{ mol}^{-1}$ for the raw and torrefied grape pomace, respectively; (ii) the NO_x concentrations along the drop tube furnace were always higher for the combustion of the torrefied grape pomace than for the combustion of the raw grape pomace because the former residue has a higher nitrogen content; and (iii) the burnout values along the drop tube furnace were always lower for the combustion of the torrefied grape pomace than for the combustion of the raw grape pomace because the former residue has a lower volatile content and a higher fixed carbon content.

1. Introduction

Grape pomace is a by-product of the wine making industry with great availability and energetic potential. According to the International Organization of Vine and Wine the world wine production in 2016 was 259 MhL [1]. During wine production approximately 25% of the grape weight results in by-product/waste (termed 'pomace', which is comprised of skins and seeds) [2]. This means that in 2016 the world grape pomace production was approximately 9 Mt. Traditionally, grape pomace has been used to obtain wine alcohol, food colorings, and grape seed oil; more recently, research has focused in the production of other value-added products, such as extracts of bioactive compounds, recovery of tartaric acid, and the making of flours [3].

In the context of the valorization of grape pomace for energy purposes, Encinar et al. [4] studied its pyrolysis, Pala et al. [5] examined its hydrothermal carbonization and torrefaction, Basso et al. [6] and Petrović et al. [7] investigated its hydrothermal carbonisation, Lapuerta et al. [8] investigated its gasification, and Miranda et al. [9,10] evaluated its densification and combustion. While demonstrating the good potential for the use of grape pomace as a biofuel, these studies also exposed important drawbacks, including the grape pomace hydrophilic character, its low heating value and the high specific energy required for its grinding. The torrefaction of the grape pomace may help to minimize these shortcomings. This thermo-chemical pre-treatment consists in exposing the biomass to an inert atmosphere at temperatures between 200 °C and 300 °C. Chen and Kuo [11] studied the impact of the torrefaction on the lignocellulosic structure of biomass and concluded that the hemicellulose and part of the cellulose are decomposed during torrefaction, while lignin is not considerably affected. Yan et al. [12] reached the same conclusions and reported an increase in the higher heating value and fixed carbon content and a decrease in the volatile matter and moisture contents for the torrefied biomass. These authors also verified a decrease in the oxygen and hydrogen contents, approximating the composition of the torrefied biomass to that of the coal. Arias et al. [13] and Phanphanich et al. [14] observed improvements in the torrefied biomass grindability due to the breakdown of

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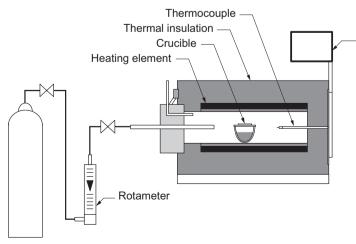
chemical bounds in the hemicellulose. Both studies concluded that the energy required to grind the torrefied biomass is lower than that to grind the raw biomass. Bach and Skreiberg [15] reported changes in the biomass hydroscopic behaviour due to the destruction of the hydrophilic hydroxyl (-OH) groups during torrefaction - the biomass becomes unable to form hydrogen bonds with water, adopting a hydrophobic behaviour. Wilk et al. [16] confirmed the advantages of the torrefied biomass over the raw biomass; specifically, the torrefied biomass presents a higher heating value, a higher energy density, a lower oxygen/ carbon ratio, and a lower moisture content, being easier to grind and having a hydrophobic character. Shoulaifar et al. [17] evaluated the change in the chemical association of ash-forming elements in birch wood as a function of the extent of torrefaction, and observed significant changes in the distribution of calcium, magnesium and manganese. Finally, Yani et al. [18] observed that torrefaction at temperatures between 220 °C and 280 °C removes from the raw biomass (mallee leaf) quantities of chlorine between 54% and 77% and changes the occurrence forms of the alkali and alkaline earth metallic species in the torrefied biomass. As a result, the ash-forming species in the raw and torrefied biomass have different abilities to produce inorganic particulate matter during combustion. It is important to note that the torrefaction process also presents disadvantages, such as the additional cost of pre- and post-processing. The former includes the grinding and drying processes, while the latter includes the cooling and densification by pelletizing and briquetting to improve the handling and transportation of the torrefied biomass.

Although a number of studies are available on the effects of the torrefaction on the structure and composition of the biomass, investigations centred on the combustion of torrefied biomass are rare and, when reported, these studies employed mostly thermogravimetric analysis (TGA) only [19]. It is widely accepted that combustion in drop tube furnaces (DTFs) emulates in a more realistic way the combustion behaviour in industrial equipment than combustion in TGAs [20], which motivated us to conduct here experiments both in a TGA and in a DTF. Under these circumstances, this study aims to evaluate the combustion characteristics of raw and torrefied grape pomace using these two experimental methods.

2. Materials and methods

2.1. Biomass residues

The biomass residues studied here were raw and torrefied grape pomace. The raw grape pomace was supplied by a local Portuguese wine producer (Agrovil), located in Azambuja, and it was sun-dried to around 7% moisture content before being used in this study. The raw



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biomass was crushed and sieved below 1 mm, and then it was torrefied in the apparatus sketched in Fig. 1. It consists of a muffle furnace equipped with a temperature control system. The furnace has internal dimensions of $100 \times 90 \times 180$ mm and a ceramic fibre insulation with a thickness of 60 mm. For each torrefaction test, 30 g of raw grape pomace was loaded into a corundum crucible (50 mL) and placed inside the furnace chamber. Subsequently, 20 L/h of argon at 260 °C (± 2 °C) was supplied to the furnace during 1 h, and afterwards the crucible was removed from the furnace and cooled in a desiccator. Before the TGAs and DTFs experiments being performed, the raw and torrefied samples were analyzed to characterize their physical and chemical properties. The particle size distribution of the raw and torrefied grape pomace was measured using the Malvern 2600 Particle Size Analyzer.

2.2. Thermogravimetric experiments

The TGA tests were made in a Netzsch STA 449 F3 Jupiter. The samples (~15 mg) were heated in alumina crucibles from ambient temperature to 800 °C at a constant heating rate of 10 °C/min in 40 mL/min of air. The instrument accuracy is 10^{-6} g. The thermogravimetric (TG) curves show the sample weight loss as a function of the temperature and the derivative (DTG) curves show the rate of weight loss with the temperature. These curves permitted to determine the combustion characteristics of the raw and of the torrefied biomass residues, as presented and discussed in section 3.2. In addition, the gases generated during the TGA experiments were analyzed using a quadruple mass spectrometer (QMS Netzsch). The mass spectrometer results showed *m*/*z* signals of 18 and 44, which represent H₂O (*m*/*z* = 18) and CO₂ (*m*/*z* = 44).

2.3. Drop tube furnace experiments

Fig. 2 shows a schematic of the drop tube furnace (DTF) used in his study, which is described in detail elsewhere [21,22]. In brief, the DTF is electrically heated and its combustion chamber is a cylindrical ceramic tube with an inner diameter of 0.038 m and a length of 1.3 m. A water-cooled injector pointing downwards feeds the solid residues and the air into the DTF. The solid particles are air-transported to the injector with the aid of an ejector system that receives the biomass residues from a twin-screw volumetric feeder.

The measuring techniques and experimental uncertainties are fully described elsewhere [21,22]. In brief, a 76 μ m diameter platinum/ platinum: 13% rhodium fine wire thermocouple was used for the measurements of temperature along the axis of the DTF combustion chamber, with experimental uncertainties due to radiation losses lower than about 5%. In addition, a 1.5 m long, water-cooled stainless steel

Fig. 1. Schematic of the torrefaction apparatus.

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