



# Effect of torrefaction on the pyrolysis characteristics of high moisture herbaceous residues



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## ABSTRACT

High moisture herbaceous residues (HR) cause a series of environment problems. Torrefaction has proven to be a promising technology for upgrading the fuel properties of biomass. In this study, a typical HR, *Licorice* residue (LR), was torrefied in a tubular reactor under nitrogen at 210, 240 and 280 °C. The effect of temperature on the fuel properties and the decomposition characteristic of the torrefied samples, as well as the pyrolysis products, were investigated. The mass and energy yield were altered in the range of 91.8–52.3% and 99.3–72.9%, respectively. The higher heating value (HHV) value of the torrefied sample increased by 8.1–39.5% from light to severe torrefaction, compared to untorrefied LR, and reached up to 23.3 MJ/kg at 280 °C. Torrefaction decreased the content of oxygenated organic compounds and the acidity of bio-oil from pyrolysis. The increase in alkanes from torrefied sample pyrolysis implies that torrefied LR has the potential for use in liquid fuel production. The present findings revealed that torrefaction turns industrial LR with poor energy content into a more easily exploitable solid fuel.

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## 1. Introduction

Traditional Chinese herbal medicine has long been used to treat illnesses, in new drug development, and as flavoring additives for food in China and worldwide [1]. Herbaceous residues (HR) are the main byproducts of the medicine industry. It is estimated that approximately 12 million tons of HR are produced annually in China [2]. *Licorice* is a popular food additive and an important component in medicine, and can be found in approximately 60% of all traditional Chinese prescriptions [3]. With the rapid development of the herbal medicine industry, the amount of *Licorice* residue (LR) continues to increase. However, LR is usually discarded randomly or treated by landfill [4,5]. Currently, no effective ways to utilize LR have been developed.

LR is a type of biomass resource and is characterized by a high moisture content. This makes it an attractive feedstock to convert into renewable energy by biological means [4,6]. Zhou et al. found that herbal residue enhanced the humification and rate of food waste composting maturity [4]. Other researchers investigated biogas production from HR by acid and alkaline pretreatment [7].

However, the bioconversion efficiency is limited because of the low biodegradability of HR, and this process is time consuming.

Thermochemical processing is regarded as one of the most effective methods for biomass utilization. LR can be converted into gaseous, solid and liquid fuels, as well as other chemicals, through pyrolysis or gasification [8]. For example, LR can be gasified or combusted to produce gas fuel and energy for production activities. This process enhances the recycling energy of the factory and reduces the demand for fossil fuels [2,9]. Mi et al. investigated the influence of the heating rate and particle size on the pyrolysis characteristics of medicine waste and evaluated the pyrolysis kinetics [10]. Wang et al. obtained a maximum bio-oil yield of 34.26% under the catalyst pyrolysis of HR [11]. However, the properties of the products obtained from herbal residue pyrolysis are not yet fully understood.

However, the main drawback of LR is that the moisture content is as high as 70%, especially in the medicine industry. As a result, LR decays easily and produces unpleasant odors. Moreover, LR is not gasified or combusted easily due to such a high moisture content. Therefore, thermochemical technologies for biomass cannot be directly used for LR, and the wet feedstock must be dewatered before being converted. Torrefaction, also called mild pyrolysis, is the thermochemical pretreatment of biomass under temperatures ranging from 200 to 300 °C [12,13]. Generally, torrefaction can be divided into three stages, light (200–235 °C), mild (235–275 °C),

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and severe torrefaction (275–300 °C) [14]. Torrefaction improves the fuel quality and enables it to be used as a fuel in widespread applications.

Currently, many studies have been conducted focusing on the torrefaction of biomasses with high moisture content, such as microalgae and macroalga [15], tomato peel or food waste [16,17], sewage sludge [18], pomaces and nut shells [19], and olive mill waste [20]. It was found that a higher heating value (HHV) of torrefied samples increases with the increase in torrefaction temperature. The fuel properties of these wet biomasses are improved greatly after torrefaction, and they show similar fuel properties to biomass or even coal. However, to date, no torrefaction study focusing on LR or the pyrolysis characteristics of torrefied LR was found in the literature. In this study, the influence of torrefaction on the characteristics of LR and the properties of the products produced were investigated. Our findings may help better understand the properties of LR pyrolysis and develop an effective utilization process.

## 2. Materials and experimental methods

### 2.1. Materials and torrefaction experiments

LR samples were obtained from a pharmaceutical factory in Wuhan, Hubei province. The moisture content (MC), ash, volatile matter (VM) and fixed carbon (FC) of the raw and LR samples were analyzed according to ASTM standards E871-82, D1102-84 and E872-82, respectively. Each test was repeated at least thrice and all measured data presented were the average value. It can be found that the water content of the LR was as high as 73.18%, and thus LR has a poor energy content.

Torrefaction was performed on a horizontal bench-scale tubular reactor (internal diameter 46 mm, length 400 mm), as described in our previous work [21]. The torrefaction temperature was set at 210 °C, 240 °C and 280 °C, which represents light, mild and severe torrefaction, respectively. Prior to each trial, the reactor was heated up to the preset temperature with nitrogen at 200 mL/min and was kept constant for 10 min. Afterwards, ~5 g LR samples were loaded in the porcelain ark, and the quickly pushed into the center of the reactor. After 60 min, the torrefaction was complete, and the ark was moved to the inlet and cooled with nitrogen (1 L/min) to ambient temperature. Finally, the torrefied samples were collected and weighted.

### 2.2. Characterization of torrefied samples

Proximate analysis of the torrefied LR was carried out according to ASTM standards [22]. The elemental compositions of the different samples were determined by a CHNS/O elementary analyzer (Vario Micro cube, Germany). The contents of carbon, hydrogen and nitrogen were determined directly, while the oxygen content was calculated by difference. The surface functionality of the samples was analyzed by FTIR spectroscopy (VERTEX 70 Bruker, Germany). The sample was first ground into fine particles and then oven-dried at 105 °C for 24 h. Then, a 0.7 mg sample was mixed with 70 mg of KBr (spectroscopy grade), and the mixtures were pressed at 10 tons/cm<sup>2</sup> pressure for 5 min to prepare the testing pellet. Prior to the test, the pellet was dried to remove absorbed water.

The mass yield ( $M_y$ ), energy yield ( $E_y$ ) and energy density ratio ( $E_d$ ) were calculated by using the following equations according to previous studies [23]:

$$M_y = \frac{M_t}{M_r} \times 100\% \quad (1)$$

$$E_y = M_y \times \frac{HHV_t}{HHV_r} \times 100\% \quad (2)$$

$$E_d = \frac{HHV_t}{HHV_r} \quad (3)$$

where  $M_t$  and  $M_r$  are defined as the mass of the torrefied LR and the raw sample, respectively. The  $HHV_t$  and  $HHV_r$  are the high heating values of the torrefied LR and the raw sample when dry, respectively.

### 2.3. Thermogravimetric and TG-FTIR analysis

The pyrolysis behavior of the torrefied and untorrefied LR was investigated using a thermogravimetric analyzer (Q600, TA) under nitrogen and air.  $10 \pm 0.2$  mg of the sample was evenly spread in an alumina crucible and heated from room temperature to 800 °C at different heating rates (10, 20, 30, 50 °C/min) under a gas flow rate of 50 mL/min and then held for 3 min at the final temperature. Each experimental result was tested at least twice to ensure repeatability. Meanwhile, a TG analyzer coupled with a FTIR spectrophotometer (Nicolet iS10, Thermo Scientific) was applied to investigate the on-line evolution of the gaseous products. The mass of the sample was approximately  $10 \pm 0.2$  mg for each test to avoid the temperature gradient in the sample and to ensure the kinetic control of the process. Then, the samples in the crucible were heated from 40 to 800 °C under a heating rate of 20 °C/min. All the experiments were conducted under a nitrogen atmosphere with a flow rate of 100 mL/min. The FTIR was connected to the TGA by a stainless-steel transfer pipe and a gas cell, which was heated to 180 °C to prevent condensation of the produced gas. The IR spectra were collected at a resolution of 4 cm<sup>-1</sup> and with a wavelength range from 400 to 4000 cm<sup>-1</sup>.

### 2.4. Py-GC/MS experiments

Pyrolysis experiments were carried out using a Pyroprobe pyrolyzer (CDS 5200) coupled with GC/MS (Agilent Technologies 7890A/5975C). Prior to the experiments, the torrefied and untorrefied LR were dried at 105 °C for 24 h, and then ground and sieved. Particles that passed through a 100-mesh sieve were collected for the experiments. In each experiment,  $0.45 \pm 0.01$  mg of the sample was loaded into a quartz tube and placed into the pyroprobe. The pyrolysis temperature was set at 450, 600, and 800 °C, with a heating rate of 20 °C/ms and pyrolysis time of 15s. Meanwhile, the temperature of the gas transmission pipeline and injection valve was set at 290 °C to prevent the condensation of volatiles.

The pyrolytic volatiles were directly swept to the GC/MS by high-pure helium as the carrier gas. The GC was equipped with a HP-5 capillary column (length, 30 m; internal diameter, 250 μm; film thickness, 0.25 μm). The GC oven was programmed at 40 °C for 3 min and then to 200 °C at 5 °C/min. After that, the temperature was increased to 280 °C at 10 °C/min and maintained for 2 min. The mass spectrometer was operated in electron-impact ionization mode at 70 eV and scanned from 40 to 450  $m/z$ . The NIST library was used to identify each compound based on the retention time and the matching mass spectrum of the standards in the spectral library.

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