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Performance of a conical spouted bed pilot plant for bio-oil production by poplar flash pyrolysis

Jon Makibar [a], A. Ruth Fernandez-Akarregi [a], Maider Amutio [b], Gartzen Lopez [b], Martin Olazar [b,*]

[a] IK4-Ikerlan, Juan de la Cierva 1, Arabako Parke Teknologikoa, E01510 Miñao, Araba Spain
[b] University of the Basque Country UPV/EHU, Department of Chemical Engineering, P.O. Box 644, E48080 Bilbao, Spain

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ABSTRACT

Poplar (*Populus nigra*) flash pyrolysis has been performed at the IK4-Ikerlan 25 kg h^{-1} pilot plant equipped with a conical spouted bed reactor. Gas, bio-oil and char yields and properties have been studied in the 425–525 °C range. This reactor has been proven to be especially suitable for this process as high bio-oil yields have been obtained, with the maximum being 69 wt.% at 455 °C. The bio-oil has been collected in two fractions: the lighter one, which accounts for 85 wt.% of the bio-oil, has a high water content and is composed mainly of acids and ketones, whereas the heavier fraction has a lower water content and is rich in phenols. These fractions are miscible, obtaining a bio-oil with a water content lower than 25 wt.% and a higher heating value in the 16–18 MJ kg^{-1} range.

1. Introduction

The bio-refinery concept, which involves chemical, biochemical and thermochemical processes to obtain fuels and raw materials from biomass as alternative to fossil fuels, has been widely developed during last decades [1,2]. A sustainable bio-based energy system should consider the incorporation of these routes into conventional oil refineries [3,4] and should also guarantee the availability of raw biomass materials without competing with food crops [5,6]. Accordingly, the implementation of short rotating wood crops for the production of biomass has increased over the last decades [7,8]. Short rotating species such as poplar or willow have a great potential in Europe because of their sustainability and high production per hectare [9,10]. A suitable management of these wood crops, i.e., appropriate infrastructure and transportation, environmental sustainability, financial feasibility of the plantation and so on, will allow a viable bio-based energy production in a local or regional scenario [7,9].

Bio-oil production by flash pyrolysis is a promising biomass valorization route for energy, fuels and chemicals production at industrial scale [11,12]. In this sense, bio-oil can be produced in the areas where biomass is highly available, such as short rotating wood crops, and be upgraded in large power plants or biorefineries to produce heat and power [13,14] or fuels and chemicals [15,16]. Biomass flash pyrolysis technology has undergone considerable development during last decades, especially regarding reactor configuration, and has been implemented worldwide by several firms such as Ensyn Technologies, Dynamotive, Metso & VTT, KIT or BTG, using different reactor configurations that include fluidized bed, transport and circulating fluidized bed, rotating cone, ablative, auger and vacuum moving bed reactors [17–19].

In this study, a conical spouted bed reactor (CSBR) is proposed as an alternative to fluidized beds, as its special characteristics make it suitable for the flash pyrolysis of several biomass types [20–22]. Indeed, the low gas residence times avoid the secondary cracking reactions that reduce bio-oil yield [23]. The particle cyclic movement allows continuously removing the char from the bed as a result of the density difference between pyrolyzed biomass and sand [24,25] and leads to high mass and heat transfer rates, which permits handling biomass particles with irregular texture and higher particle size than in fluidized beds, thus reducing the associated costs [26]. Furthermore, the technology has been scaled up to a 25 kg h^{-1} pilot plant as a result of the collaboration between IK4-Ikerlan Research Centre and the University of the Basque Country (UPV/EHU) [27]. Thus, the scale-up problems related to bed stability and high gas flow rates have been adequately solved by means of internal devices [25]. Additionally, two strategies have been proposed to progress towards industrial scale of the conical spouted bed technology for biomass flash pyrolysis: vacuum operation to reduce gas flow rate and enhance bio-oil collection [24]; and oxidative pyrolysis as a way to attain autothermal regime [28].

In order to assess the suitability of this technology for the pyrolysis of short rotating crops as a way to progress in the achievement of a bio-based sustainable energy scenario, the pyrolysis of poplar sawdust

* Corresponding author. Tel.: +34 946012527; fax: +34 946013500.
E-mail address: martin.olazar@ehu.es (M. Olazar).

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has been carried out in the IK4-Ikerlan conical spouted bed 25 kg h^{-1} pilot plant. Accordingly, product yields and properties have been determined, paying special attention to bio-oil composition and fuel properties.

2. Materials and methods

2.1. Raw material characterization

The raw material used in this study is *populus nigra* wood sawdust, which has a wide size distribution, with a diameter in the 50 μm–3 mm range and a length between 5 and 15 mm. The characterization of the poplar sawdust and the methods followed are summarized in Table 1.

In addition, the pyrolysis behavior of the raw material has been assessed by means of a thermogravimetric analysis carried out in a TGA Q500IR thermobalance, with a heating ramp of 15 °C min^{-1}, Fig. 1. Poplar decomposition starts with the evaporation of the moisture at 100°C, followed by a wide band that peaks at 345°C, in which the devolatilization of the three biomass main components (cellulose, hemicellulose and lignin) overlaps [29]. The highest peak corresponds to cellulose degradation, which takes place between 280 and 370 °C, and the main shoulder that precedes cellulose degradation in the 200–350 °C range is assigned to hemicellulose. However, lignin devolatilization contributes to both peaks, because it occurs in a wide temperature range (between 180 and 600°C), and also to the long tail at higher temperatures [30]. Furthermore, this analysis allows for a qualitative determination of the hemicellulose, cellulose and lignin content of the biomass, which will affect the product distribution during pyrolysis [31]. Accordingly, based on the thermogravimetric curve obtained, a similar product yield and composition to other woody biomass materials is expected [32].

2.2. Pyrolysis pilot plant

The 25 kg h^{-1} IK4-Ikerlan pyrolysis pilot plant is based on the spouted bed technology, which has been developed at laboratory scale at the University of the Basque Country UPV/EHU for several types of biomass [20–22,33], as well as waste materials such as scrap tires or plastics [34,35]. The development of this technology, in addition to the design and operation of the plant has been thoroughly described elsewhere [27]. Fig. 2 shows the P&ID diagram of the plant, which is made up of a conical spouted bed reactor, biomass feeding system, gas preheater, two cyclones for cleaning the gaseous product stream, the bio-oil condensation and retention system composed of a scrubber, demister and coalescence filters, a blower and a flare to burn the purge gas.

Table 1
Poplar characterization.

Properties		Method
Ultimate analysis (wt.%)		
Carbon	44.2	ASTM 5373
Hydrogen	6.3	ASTM 5373
Nitrogen	0.7	ASTM 5373
Oxygen	48.8	ASTM 5373
Proximate analysis (wt.%)		
Moisture	9.3	ASTM 3302
Volatile matter	75.4	UNE 32019
Fixed carbon	14.8	
Ash	0.5	UNE 32004
HHV (MJ kg^{-1})	17.4	UNE 32006

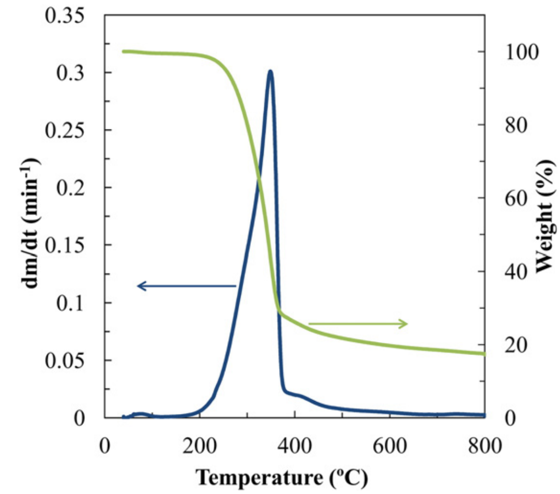

Fig. 1. Evolution of the TG and DTG curves of poplar sawdust with temperature.

2.3. Experimental procedure

Pyrolysis runs have been conducted in continuous mode for operation periods of 4–6 h. In order to assess the effect of temperature on product yields and properties, the runs have been carried out in the 425–525 °C range, which has been established to maximize the bio-oil yield [36,37]. The bed was made up of 6 kg of sand (with a Sauter particle diameter of 1.4 mm), guaranteeing a good heat transfer and isothermicity [23]. The fluidizing gas flow rate was 2 times that corresponding to minimum spouting conditions to ensure stable spouting and low residence time of the volatiles in the reactor, and has been set in the 350–360 Nl min^{-1} range, depending on operation temperature [25]. The gases were previously preheated to a temperature 10–30 °C higher than the reaction temperature. Non-condensable gases fedback from the outlet of the condensing system are used as fluidizing agent. However, nitrogen is used for the starting-up of the process until temperature is set to the desired value and biomass feed is started. The excess gas formed in the pyrolysis process is purged from the system, by adjusting the reactor internal pressure to atmospheric, and burnt in a flare.

Mass balance closure has been performed by weighing the biomass in the hopper before and after each run, the char fraction collected in the reactor and cyclones and the bio-oil collected in the scrubber, demister and filters. Accordingly, gas yield is calculated by difference. Char can be continuously elutriated from the reactor as a result of the cyclic movement of the particles in the CSBR, which has been optimized in previous hydrodynamic studies [25]. Thus, char particles describe higher trajectories at the fountain region of the reactor due to their lower density compared to both sand and biomass, and are removed by the out-going volatile stream [24].

The bio-oil collected in the condensation system has been subjected to several characterization methods in order to determine its properties. Water content has been measured by Karl Fischer tritation (KF DL31 Mettler Toledo), and the viscosity was determined by the cone and plate Rheometer Model Rheo-Stress 1 (HAAKE). The identification of the main products that compose the bio-oil has been carried out by GC/MS, in an Agilent HP6890 chromatograph (equipped with a FID detector) connected to a MS 5973 mass spectrometer. This technique has also been used to quantify the concentration of each compound by adding an internal standard (cyclohexane, not formed in the process) to the samples to be analyzed and considering the chromatographic response factors for the individual compounds. The higher heating value of the bio-oil has been measured by means of a Parr 1356 isoperibolic bomb calorimeter, and its ultimate analysis has been