



## Research paper

## Investigation of innovative and conventional pyrolysis of ligneous and herbaceous biomasses for biochar production

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## ABSTRACT

In this work three biomasses, two ligneous (rubberwood and eucalyptus) and one herbaceous (*Phragmites australis*), were fed to three different pyrolysis reactors: the Jiggled Bed Reactor (JBR) and a Mechanically Fluidized Reactor (MFR), working in slow batch pyrolysis mode, and a Bubbling Bed Reactor (BBR) operating as a continuous fast pyrolysis process. The obtained biochars were successively physically activated in the batch JBR. The research had three objectives: 1. Investigate biochar production through two different pyrolysis routes (slow-intermediate batch vs fast-continuous) and three different reactor designs (MFR vs BBR vs JBR); 2. Analyze the efficiency of biochar physical activation processes performed through JBR reactor; 3. Compare activated biochars to evaluate whether an herbaceous feedstock may be effective as ligneous biomasses. The results of the study disclosed a good validation of the performances of the JBR. In detail, the two-step JBR process (bio-char production + activation) resulted in the highest yields. Secondly, it returned analogous values of surface area ( $385 \text{ m}^2 \text{ g}^{-1}$ ) and micro-pores area ( $283 \text{ m}^2 \text{ g}^{-1}$ ) respectively, compared to the BBR and the MFR. Thirdly, micro-pore volume ( $0.13 \text{ cm}^3 \text{ g}^{-1}$ ) and pore size ( $21 \text{ \AA}$ ) were similar to the values obtained with both the MFR and the BBR. Finally, the overall results demonstrated that *Phragmites australis* can be employed for the production of biochar and activated carbon, showing a behavior similar to ligneous biomasses.

## 1. Introduction

Pyrolysis is a thermochemical process involving the thermolysis of carbon-based materials in the absence of an oxidizing agent. Pyrolysis processes are optimized adjusting the operating conditions (temperature, heating rate, residence time, reactor configuration and feedstock type) in order to maximize the desirable gaseous, liquid or solid products [1]. Traditionally, pyrolysis processes are classified as [2–4]: *slow pyrolysis*, characterized by temperatures in the range of 400–800 °C, slow heating rates ( $< 0.2 \text{ }^\circ\text{C s}^{-1}$ ) of the feedstock, and long residence times, with biochar as main product; *fast pyrolysis*, characterized by temperatures between 450 and 550 °C, high heating rates ( $100\text{--}1000 \text{ }^\circ\text{C s}^{-1}$ ) and very short residence times ( $< 2\text{s}$ ), with bio-oil as the main product. The core distinction between pyrolysis reactors depends on the gas-solid contact mode, which divides the reactors into fixed beds, fluidized beds, and entrained beds. From the design point of view, the main types of reactors are: fixed beds, rotary drums, auger reactors, bubbling fluidized beds, circulating fluidized beds, rotative cone pyrolysis, ablative pyrolysis and vacuum pyrolysis.

Biomasses are considered “carbon-neutral” feedstock, meaning that they don't involve any addition to the  $\text{CO}_2$  inventory [3]. Furthermore, the use for biochar production of agricultural waste [5] and non-edible biomasses, particularly herbaceous ones that have short growing periods (measurable in months) [6], leads to undeniable environmental and socio-economic outcomes compared to the use of conventional ligno-cellulosic feedstocks, such as wood and coconut husks. Studies specifically concerning *Phragmites*-derived biochar [7–9], highlighted that throughout the late autumn and winter, *Phragmites* lose their green appearance and dry under natural conditions, making this invasive herbaceous biomass one of the best natural feedstocks for pyrolysis without the need of energy-wasting drying processes. *Phragmites australis* also seems to be a promising energy plant and a chemical feedstock due to its high productivity potential (it can provide up to 28 tons of dry mass per acre per year [9]).

The interest in biochar has been recently increasing, thanks to its possible applications in soil improvement, carbon sequestration and activated carbon production, as well as in more advanced applications for the manufacturing of catalysts, composites and electronic

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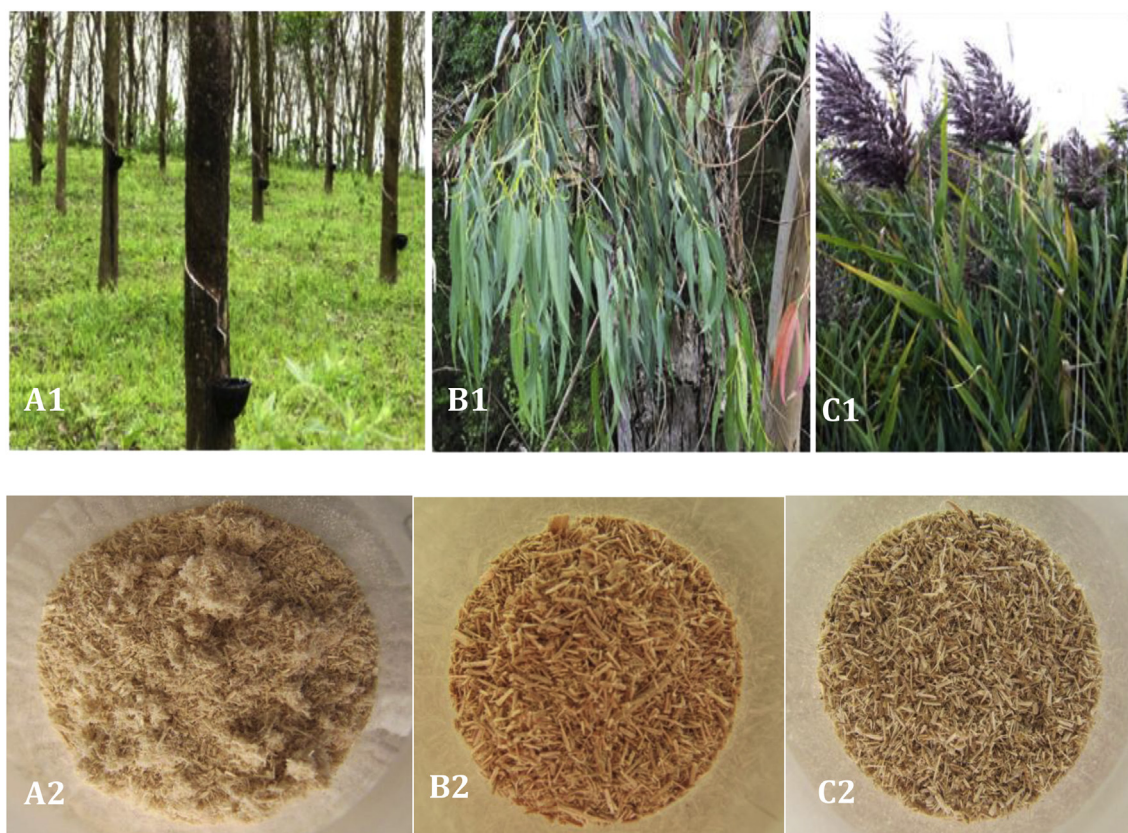


Fig. 1. Biomasses considered in the study (1. unaltered, 2. ground and dried): A. Rubberwood; B. Eucalyptus; C. *Phragmites australis*.

components, or as food and feed additive [4]. Biochar mostly contains aromatic forms of carbon than cannot be readily returned to the atmosphere as  $\text{CO}_2$  even under favorable environmental conditions [10]. Consequently biochar is commonly considered a “carbon-negative” material, since carbon is sequestered for hundreds to thousands of years [11].

From the physical-chemical viewpoint, biochar is a porous material made of carbon and ashes (e.g. the inorganic components of the biomass employed as feedstock), having a mesoporous or a microporous structure, depending on the operating conditions employed for its production and the feedstock type. Biomass composition, especially its H to C ratio and mineral content, have an important bearing on pyrolysis yields [11]. Each of the three major constituents of a ligno-cellulosic biomass (hemicellulose, cellulose, lignin) has its preferred temperature range of decomposition. The individual constituents undergo pyrolysis differently, making varying contributions to yields: cellulose and hemicellulose are the main sources of volatiles, while lignin degrades more slowly, making a major contribution to the bio-oil and char yields thanks to its aromatic content. The size, shape and physical structure of the biomass also have some influence on the heating rate and, therefore, on pyrolysis products. A recent study [12] quantitatively related biochar proximate analysis (e.g. volatile matter, VM; fixed carbon, FC; ash) to its elemental composition in terms of H to C and O to C ratio values, i.e. to its quality. Another study [4] pointed out that biochars produced at higher temperatures are effective in adsorption of organic contaminants due to the increase in surface area, micropores area and hydrophobicity. On the contrary, biochars obtained at lower temperatures are able to develop stronger electrostatic interactions towards cationic nutrients in the soil. More precisely, high temperature leads to the increase in FC content, ash, inorganic elements, pH, surface area and porosity while lower temperature results in an increase in biochar yield, VM, electrical conductivity and cation exchange capacity (CEC).

The main challenges related to the development of engineered commercial biochars are due to three crucial issues at the moment not yet fully defined: a systematic and consistent characterization methodology for biochar; standard requirements for specific biochar applications; the understanding of the correlations among feedstock features, pyrolysis and activation conditions, and biochar characteristics. This work aims to contribute to fill the above-mentioned knowledge gaps, investigating the performances of three pyrolysis reactors on the grounds of the quantity and quality of the biochar obtained from different biomasses. Three types of biomass were fed to each reactor: two ligneous (rubberwood and eucalyptus) and one herbaceous (*Phragmites australis*). Three lab-scale reactors were employed and each was fed with the three biomasses: two conventional devices, a Mechanically Fluidized Reactor (MFR) and a Bubbling Bed Reactor (BBR), which respectively run slow and fast pyrolysis; and a novel reactor developed at ICFAR (Institute for Chemicals and Fuels from Alternative Resources), the Jiggled Bed Reactor (JBR), operating in slow pyrolysis mode. The nine sets of biochar samples were afterwards physically activated in the JBR. Biomasses, chars and activated carbons were characterized considering pyrolysis yield, moisture content, proximate and elemental analyses, BET surface area, pore volume and size, and SEM-EDS analyses.

This research had three main objectives, which investigated the three key elements of activated biochar production: feedstock, pyrolysis and activation. The first objective was to investigate biochar production utilizing two different pyrolysis processes (slow batch vs fast continuous) and three different reactor designs (MFR vs BBR vs JBR). Secondly, the efficiency of biochar physical activation processes (performed batchwise via the JBR) of the samples derived from the three reactors was studied. In summary, an evaluation of the JBR was carried out to determine whether it could offer a valid experimental simulator to more conventional MFR and BBR, combined with its peculiar ability of also activating the char. Finally, a comparison among biochars

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