



Effect of cellulose, hemicellulose and lignin contents on pyrolysis and combustion of natural fibers



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ABSTRACT

This paper investigates the effects of cellulose, hemicellulose and lignin on the pyrolysis and combustion of several natural fibers (cotton linter, flax, hemp, sugar cane, bamboo and coir). Different parameters have been selected to study the relations between chemical composition, pyrolysis and combustion: char yield (Res), effective heat of combustion (EHC), activation energy of combustion (Ea) and CO/CO₂ ratio during cone calorimeter test. A correlation was found between these parameters and the lignin content in a large range of composition. The natural fibers with high content of lignin exhibit high char yield, high EHC, high Ea and low CO/CO₂ ratio. However, a particular behavior was observed at low lignin/cellulose ratio. The presence of a low content of lignin with a high content of cellulose affects the degradation pathway of the latter and leads to charring and to incomplete combustion of these fibers, limiting their contribution to the heat evolved during burning.

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1. Introduction

Natural fibers/polymer biocomposites are emerging as a viable alternative to traditional glass reinforced composites. Many natural fibers are now available as reinforcement for polymers (flax, hemp, jute, coir, kenaf, sisal, abaca, . . .) [1,2]. It is generally recognized that these fibers exhibit some benefits compared to glass fibers: low density, good specific mechanical properties, non-abrasive properties, low acoustic and thermal conductivity, biosourcing and biodegradable. Conversely, their wide use in industrial applications remains limited by some drawbacks that must be overcome: variability of the dimensional and structural properties (e.g. the density, the diameter, the length, the cellulose rate, . . .), water sensitivity, poor compatibility with polymer matrix, low thermal sensitivity and high flammability [2–4]. In some sectors such as building, public transportation, electrical equipments, reaction to fire is a key specification. Therefore, the use of biocomposites in those sectors requires that their flame retardancy is improved. As organic compound and significant component of biocomposites, natural fibers are an important source of fuels during a fire. Moreover, natural fibers can be used as char, promoting a barrier effect [4–6]. Hence, the pyrolysis and the combustion of natural fibers must be

accurately investigated to better understand their contribution to a fire scenario.

The pyrolysis of lignocellulosic biomass has been the subject of many papers in the literature [7–10]. The general topic of these papers is the thermochemical conversion of biomass to generate heat. This subject differs to some extent from the thermal behavior of the biomass during an accidental fire in so far as decomposition occurs generally in well controlled conditions (temperature and atmosphere). However some useful information can be extracted from this literature to apprehend the burning behavior of natural fibers. Thermogravimetric analysis (classical or modulated) is the main technique used in these studies. In some cases, TGA is coupled with FTIR analysis. Several papers investigated the pyrolysis mechanisms of the three main components of biomass (cellulose, hemicellulose and lignin) [11–13]. The pyrolysis of hemicellulose and cellulose occurs quickly with a weight loss from 220 to 315 °C for hemicellulose and from 315 to 400 °C for cellulose. Lignin decomposes over a wider range of temperature (from 160 to 900 °C) and generates a high char yield [13]. Some of these studies concluded that the pyrolysis of any biomass is the superposition of the three main components decomposition [13–16]. However, contradictory results were also mentioned. Pasangulapati et al. studied the pyrolysis and gas evolution profiles of four biomass materials with different composition in cellulose, hemicellulose and lignin [11]. They observed that the three components have weight loss profiles and activation energies very different. However, no significant difference was observed on the same parameters for the

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Table 1
Chemical composition of the different natural fibers used in this work.

Fibers	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Soluble (%)	Ash (%)	Morphology (length ^a)
Cellulose	100	0	0	0	0	Microfiber (μm)
Lignin	0	0	100	0	0	Powder (μm)
Xylan	0	"100"	0	0	0	Powder (μm)
Cotton linter	89.7 (0.4)	1.0 (0.3)	2.7 (0.2)	6.3 (0)	0.3 (0.1)	Microfiber (cm)
Flax	80 (1)	13 (1)	2 (1)	4 (1)	1 (1)	Fiber (mm)
Hemp	74.1 (0.5)	7.6 (0.3)	2.2 (0.2)	15.8 (0.4)	0.3 (0.1)	Long fiber (cm)
Sugar cane	51.8 (0.4)	27.6 (0.8)	10.7 (0.4)	9.1 (0.9)	0.8 (0.1)	Fiber (mm)
Bamboo	54.6 (0.6)	11.4 (0.6)	21.7 (0.3)	10.6 (0.1)	1.6 (0.1)	Fiber (mm)
Coconut	51.3 (1.1)	11.7 (0.7)	30.7 (0.4)	5.7 (0.4)	0.6 (0.1)	Long fiber (cm)

^a The length is an order of magnitude.

four biomass materials (switchgrass, wheat straw, redcedar, dried distilled grains with solubles). However, some changes could be noticed about the released gases. The lignin-rich biomass released a larger amount of methane.

Many of the previously mentioned papers studied also combustion of biomass by switching the purge gas from nitrogen to air in TGA experiments [7–10,12]. The thermal degradation studied in these conditions corresponds actually to oxidative pyrolysis. Cheng et al. compared the oxidative and non-oxidative pyrolysis of a woody biomass with those of microcrystalline cellulose, xylan and lignin, extracted by organosolv process, using a modulated-TGA [12]. The presence of oxygen leads to a second decomposition step at high temperature which corresponds to char oxidation. For xylan, no significant difference is observable. Lignin under air starts to decompose at higher temperature and the mass residue at 600 °C is negligible. Many data are available on decomposition temperature and activation energy for oxidative and non-oxidative pyrolysis of various biomasses [17].

If several papers are now available on the fire behavior of composites containing natural fibers [5,18,19], the literature on the combustion characteristics (time to ignition, heat release,...) of natural fibers themselves in a fire scenario is almost non-existent. One of the only papers on the subject was written by Kozłowski et al. [4]. They investigated the flammability of natural fibers using cone calorimeter. They observed that the lignin rich bast fibers (flax, hemp) exhibit a lower heat release rate than leaf fibers (cabuya and abaca). So a high content of cellulose could increase the flammability of the fibers.

The objective of the present work is to determine some of the main parameters of pyrolysis and combustion of natural fibers (cotton linter, flax, hemp, sugar cane, bamboo, coir) involved in the development of a fire. It will be attempted to relate these characteristics to the fiber composition. For this purpose, cellulose, xylan (main component representative of hemicellulose) and lignin will be used as model for the main components of biomass. The contribution to pyrolysis and combustion will be evaluated using the four following parameters: the char yield (Res), the effective heat of combustion (EHC), the activation energy of combustion and the evolution of CO/CO₂ ratio during a cone calorimeter test.

2. Experimental

2.1. Materials

Cellulose (Arbocel BC100, from Rettenmeier & Söhne GMBH), xylan extracted from beechwood (Sigma Aldrich), which is the main component of hemicellulose [20] and an organosolv lignin kindly supplied by the Centre de Mise en Forme des Matériaux (CEMEF-Mines ParisTech at Sophia Antipolis, France) were used as model components of lignocellulosic fibers. Cotton linter (from Maeda Society), flax (Fibras-S6B, from FRD), hemp (from Chanvrière de l'Aube), sugar cane (from Centro Universitario Fundação Santo

André, Brazil), bamboo (grade 3, from Bamboo Fiber Technology) and coir (from Centro Universitario Fundação Santo André, Brazil) were used as natural fibers. The chemical composition of the fibers was determined by FRD[®] by solvent extraction (shown in Table 1). The standard deviation is given into brackets.

2.2. Characterizations

2.2.1. Thermogravimetric analysis (TGA)

TGA was performed under nitrogen atmosphere with a Perkin Elmer Pyris-1 Thermogravimetric Analyzer instrument. A 10 mg-sample was dried at 95 °C during 10 min, to evaporate the moisture, then the sample was heated from 95 to 900 °C at 10 °C/min. The char yield at 600 °C (Res), the peak of mass loss rate (pMLR) and the temperature at pMLR (T_{deg}) were determined. The drying step is not taken into account in the plotted curves. So the TGA curves start at 95 °C corresponding to a 100% of weight. All experiments were repeated three times. The temperature precision is ±2 °C. The dispersion of weight measurements on the same sample was found to be approximately 5%.

2.2.2. Pyrolysis combustion flow calorimeter (PCFC)

PCFC tests were carried out using a Fire Testing Technology (FTT) microcalorimeter. 2 (±1) mg-sample was heated under nitrogen flow up to 750 °C at a heating rate equal to 1 °C/s. Pyrolysis gases were extracted and sent to a combustion chamber in the presence of N₂/O₂ 80/20 flow. For each experiment, sample weight was chosen carefully to ensure that O₂ is always in excess. Heat release rate is calculated from the oxygen consumption according to Huggett's relation [21] (1 kg of consumed oxygen corresponds to 13.1 MJ of heat release). Each test was performed twice.

The peak of heat release rate (pHRR), the total heat release (THR), and the effective heat of combustion (EHC) at 900 °C were determined (combustion is believed to be complete at 900 °C). EHC is calculated as the ratio between THR and mass loss at 600 °C measured using TGA.

In supplementary experiments, temperature of combustion was monitored between 600 and 900 °C. Therefore, combustion efficiency at a temperature T is calculated as follows [22]

$$\chi(T) = \frac{THR(T)}{THR(900\text{ }^{\circ}\text{C})} \quad (1)$$

It should be reminded that pyrolysis conditions were never changed (nitrogen flow). Only the combustion efficiency was affected when the combustion temperature was modified.

All experiments were performed in triplicate. According to the constructor, the accuracy of HRR values in PCFC is around 5%. The dispersion of the results on the same sample was within this range of variation.

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