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Energy Procedia 142 (2017) 636-641



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### 9th International Conference on Applied Energy, ICAE2017, 21-24 August 2017, Cardiff, UK

# Pyrolysis Characteristics of Cellulose Isolated from Selected Biomass Feedstocks using a Thermogravimetric Analyser

## Zhezi Zhang, Mingming Zhu, Dongke Zhang\*

Centre for Energy (M473), The University of Western Australia, 35 Stirling Highway, Crawley, WA 6009, Australia

#### Abstract

Utilisation of biomass by means of pyrolysis has received great interests for the production and use of biofuels. Biomass consists of three major components, namely, cellulose, hemicellulose and lignin and their contents vary from feedstock to feedstock. Understanding the pyrolysis behavior of each component and their interactions holds a key to understanding the complex biomass pyrolysis process. This study was aimed to compare the pyrolysis characteristics of cellulose from various biomass resources. Cellulose samples isolated from selected raw biomass materials, namely, pine wood and wheat straw, in addition to a pure cellulose sample acquired from Sigma Aldrich as a reference, were used in this study. The raw biomass sample was first milled to a particle size fraction of  $< 310 \,\mu\text{m}$  and treated using Soxhlet extraction in a 2:1 (v/v) toluene/ethanol solvent to remove wax. Lignin was then removed by treating the dewaxed sample in a 1M NaClO<sub>2</sub> solution at 343K until the product became white. Finally, hemicellulose was leached out by soaking the de-lignified sample in a 6 wt % KOH solution at room temperature overnight and then at the same concentration at 353K for 2 h. Fourier transform infrared spectroscopy (FTIR) and X-ray diffractometer (XRD) were applied to characterise the surface functional groups and the crystallinity of the cellulose samples. The pyrolysis experiments were performed using a thermogravimetric analyser (TGA) in nitrogen at 10Kmin<sup>-1</sup> heating rate from room temperature to the final temperature of 823K. The FTIR and XRD results indicated that cellulose can be successfully isolated from the raw biomass samples via the chemical treatment used. The cellulose isolated from both pine wood and wheat straw showed similar pyrolysis characteristics but differed significantly from the reference cellulose. The DTG curves of cellulose samples isolated showed characteristic peaks at 600 and 608K, ca 5-13K lower than that of the reference cellulose and exhibited a large flat shoulder in the temperature range of 475 - 575K. It was identified that the cellulose samples isolated from selected biomass had lower crystallinity and smaller crystallite compared to the reference cellulose, which in turn, impacted on their pyrolysis behavior.

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Peer-review under responsibility of the scientific committee of the 9th International Conference on Applied Energy.

1876-6102 ${\ensuremath{\mathbb C}}$  2017 The Authors. Published by Elsevier Ltd.

Peer-review under responsibility of the scientific committee of the 9th International Conference on Applied Energy. 10.1016/j.egypro.2017.12.105

<sup>\*</sup> Corresponding author: Dongke Zhang, Email: dongke.zhang@uwa.edu.au Phone: +61 8 6488 7600, Fax: +61 8 6488 7622

Keywords: Biomass; Cellulose; Chemical treatment; Pyrolysis

#### 1. Introduction

The processing of biomass resources, such as forestry by-products, agricultural residues, and non-food energy crops, for renewable electricity and fuels has been recognised as a most feasible means of reducing CO<sub>2</sub> emissions and combating global climate change. Pyrolysis is considered a promising technology to utilise biomass. Pyrolysis gas and liquid can be used for combined heat and power (CHP) generation, not only providing local energy supply but also reducing liabilities in waste management. The solid residual known as biochar, the by-product of the process, can be used as fuels [1], a soil conditioner [2] and adsorbents [3]. Biomass mainly consists of three major components, namely, cellulose, hemicellulose and lignin and their contents vary from feedstock to feedstock. Due to the complexity and variability of biomass, understanding the pyrolysis behaviour of individual components and their interactions during pyrolysis would be beneficial for the development and optimisation of biomass pyrolysis technology [4-6]. Many researchers have studied pyrolysis behaviour of cellulose [4-6], because it is the most abundant polymer in lignocellulose biomass. And they often used commercial pure cellulose samples isolated from high cellulose content biomass. However, cellulose exists in crystalline and amorphous form and crystallinity of cellulose varies depending on the nature of the raw biomass from which the cellulose is extracted [7]. Antal et al. studied the pyrolysis behaviour of several different pure cellulose samples, including Millipore ash-free filter pulp, Whatman #42 filter paper, CF-11 cellulose, and the Avicel cellulose, in a thermogravimetric analyser (TGA). The four different cellulose samples showed different thermal stability. The most stable sample (Whatman #42 filter paper) decomposed at temperatures 30 °C higher than the least stable sample (Avicel cellulose)[8]. More recently, Lin et al. summarised the kinetic parameters of cellulose pyrolysis from a large collection of literature studies [9] and found that the kinetic parameters obtained from various pure cellulose samples varied significantly. These evidences suggest that the structure of cellulose, such as crystallinity and degree of polymerization, can play a strong influence on the pyrolysis behavior of cellulose, which have not been well understood. Poletto et al. [10] and Wang et al. [11] found that the pyrolysis process was governed by a diffusion controlled mechanism under certain conditions in which the thermal diffusion was affected by the densely packed fibrous structure in the wood, such as the well-packed cellulose chains. Thus, the structural differences in the cellulose chain arrangement can affect the thermal decomposition behaviour of cellulose. However, such information was rare in the literature.

The present work was aimed to examine and compare the pyrolysis characteristics of cellulose samples with different crystallinity. A chemical treatment method commonly used for nanocellulose isolation was adapted and used [12-16] to isolate the cellulose samples from real biomass feedstocks. A commercial cellulose sample widely reported in the literature was acquired and employed as a reference. The research outcomes would shed some light on the biomass pyrolysis mechanisms and ultimately guide the further research and commercial applications.

#### 2. Experimental

Two raw biomass materials, namely, pine wood and wheat straw, were selected for the present study. The as received samples were first milled to a particle size fraction of less than 310  $\mu$ m. The details of the cellulose isolation procedures can be found elsewhere [12, 17]. In brief, wax was first removed from the raw biomass using Soxhlet solvent extraction with a 2:1 (v/v) mixture of toluene/ethanol as the solvent for 6 h. Lignin in the sample was then removed by soaking the dried de-waxed sample in a 1.0M NaClO<sub>2</sub> solution at 343K until the products became white. Finally, the sample was treated in a 6 wt % KOH solution at room temperature overnight and then at the same concentration at 80 °C for 2 h in order to leach hemicellulose. The isolated cellulose was first dried at 323K and 100 kPa and then fed through the cutting mill with a 310  $\mu$ m screen to break the large agglomerates formed during the chemical treatments. The Klason lignin contents of the raw, de-waxed, de-lignified samples were measured following the standard procedure reported in [18]. A reference cellulose sample sourced from Sigma Aldrich was included for comparison. The proximate analysis, wax and Klason lignin content results of the various cellulose samples are shown in Table 1.

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