



Production of bio-oil with low contents of copper and chlorine by fast pyrolysis of alkaline copper quaternary-treated wood in a fluidized bed reactor



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ABSTRACT

Fast pyrolysis of ACQ (alkaline copper quaternary)-treated wood was carried out in a bench-scale pyrolysis plant equipped with a fluidized bed reactor and char separation system. This study focused on the production of a bio-oil with low copper and chlorine contents, especially by adopting the fractional condensation of bio-oil using water condensers, an impact separator and an electrostatic precipitator. In addition, various analytical tools were applied to investigate the physicochemical properties of the pyrolysis products and the behavior of the preservative during pyrolysis. The bio-oil yield was maximized at 63.7 wt% at a pyrolysis temperature of 411 °C. Highly water-soluble holocellulose-derived components such as acetic acid and hydroxyacetone were mainly collected by the condensers, while lignin-derived components and levoglucosan were mainly observed in the oils collected by the impact separator and electrostatic precipitator. All the bio-oils produced in the experiments were almost free of copper and chlorine. Most copper in ACQ was transferred into the char.

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1. Introduction

Since the late 1990s, ACQ (alkaline copper quaternary) has become the predominant choice in wood preserving industries worldwide. Its large production has resulted in a huge amount of ACQ-treated wood waste [1]. ACQ-treated wood contains hazardous components, such as copper and DDAC (didecylidimethylammonium chloride). For its disposal, therefore, a great caution must be exercised. Typical proposal methods for ACQ-treated wood waste have been incineration and landfill. Landfill of ACQ-treated wood waste, however, could lead to serious soil contamination. Incineration of ACQ-treated wood waste would also pose serious environmental problems due to the emissions of highly toxic smoke and fumes, because municipal waste incinerators generally are not equipped with treatment facilities for these types of toxic elements [2].

The thermochemical conversion of biomass has been investigated for many years as a supply source of renewable solid, gaseous and liquid fuels. Among thermochemical conversion technologies, pyrolysis can be applied as an environmentally friendly tool for

energy and material recovery or as a disposal method for various wastes. In particular, fast pyrolysis, which rapidly heats the biomass at moderate temperatures in the absence of oxygen, is a strong tool for producing bio-oil, which is a complex mixture of organic compounds that can be used as transport oils or chemical feedstocks [3]. Biomass pyrolysis has usually been performed using bubbling and circulating fluidized bed reactors [4,5] or different reactors, such as a microwave-heated reactor [6–8]. The pyrolysis temperature is the key reaction parameter in the fast pyrolysis for a high bio-oil yield. Other important parameters are feed size, feed rate and the kind of fluidizing medium [9,10].

Regarding the research on the pyrolysis of preservative-treated wood, the pyrolysis of copper, CCA (chromium and arsenic)-treated wood in the temperature range of 300–450 °C has been dominant until now. Only a few studies examined the fate of contaminants of CCA-treated wood during pyrolysis [11,12], which concluded that most copper present in the feed material was retained in the char. Fu et al. also pyrolyzed CCA-treated wood and reported that copper favored the formation of levoglucosan [13]. Meanwhile, research on the pyrolysis of ACQ-treated wood is rare. In a study carried out with ACQ-treated wood at a reaction temperature of 500 °C, the obtained bio-oil yield was 46.6 wt%, and most copper was transferred into the char during pyrolysis [14].

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In this study, the fast pyrolysis of ACQ-treated wood was conducted in a bench-scale pyrolysis plant with a fluidized bed reactor and char separation system. Main goals of the study were to produce a bio-oil containing low chlorine and copper contents with a high yield and to investigate the possibility of obtaining bio-oil with very high concentrations of value-added compounds by adopting the fractional condensation of bio-oil. For the purpose, the reaction conditions (reaction temperature, feed size and feed rate) were optimized. In the characterization of the bio-oil, various analytical tools were applied: at first, the RRF (relative response factor) for each component was applied in the GC (gas chromatography) analysis and furthermore, HPLC-MS (high-performance liquid chromatography-mass spectrometry), IC (ion chromatograph), UPLC-IT-TOF (ultra-speed liquid chromatography-ion trap-time of flight), and ICP-AES (inductively coupled plasma-atomic emission spectrometry) were applied for the quantification of DDAC, chlorine and levoglucosan in bio-oils.

2. Experimental design

2.1. Feed material

ACQ-treated wood (*hemlock* sp.) generally used as a construction material such as deck material was obtained from the BS Wood Inc. in Korea. It was milled to powder to obtain a fraction with a particle size of 0.425–1 mm. Fine particles are essential for the fast pyrolysis by ensuring rapid reaction of the particles. After milling, the feed material was dried in an oven at 110 °C for 24 h to remove moisture of the feed material. The dried feed material was used in the experiments. The main characteristics of the feed material are shown in Table 1.

The HHV (higher heating value) of the feed material was 19.9 MJ/kg. The ultimate analysis showed that the feed material contained about 0.01 wt% chlorine, which appeared to originate from the DDAC in the feed material. The metal analysis showed that the copper content was 0.2 wt%. The chemical composition analysis showed lignin, cellulose and hemicelluloses contents of the feed material were 29.2, 41.1 and 27.1 wt%, respectively. The lignin content (>25 wt%) indicated that the feed material was softwood [15].

2.2. Pyrolysis plant and reaction conditions

Pyrolysis experiments were performed in a bench-scale plant consisting of a feeding system, fluidized bed reactor, char-separation system, quenching system and gas circulating system, with a throughput of up to 1 kg/h. The feeding system was equipped with two screw feeders to feed the material at a variable rate. The feed material entered directly into the fluidized bed reactor filled with quartz sand with a mean diameter of 0.4 mm. The fluidized bed reactor was made of a 316 stainless steel tube with an inner diameter of 110 mm and a height of 390 mm and indirectly heated by electricity. The reaction temperature was defined as the average value calculated from values measured by three thermocouples located in the reactor. The char separation system consisted of a cyclone and a ceramic hot filter designed to capture particles bigger than 10 and 2 μm, respectively. The quenching system, where pyrolysis oil was condensed, consisted of a steel condenser, and a glass condenser, an impact separator and an electrostatic precipitator. The steel condenser and glass condenser were cooled to 20 and –20 °C using water and ethanol, respectively. The impact separator was specially designed to capture slow-moving, high molecular weight components in the pyrolysis vapor by inducing the collision of those components with a series of plates. A compressor (N0150ATE, KNF) was used to circulate non-

Table 1
Main characteristics of ACQ-treated wood.

Proximate analysis ^a		Ultimate analysis ^c	
	wt%		wt%
Moisture	6.4 ± 0.21	Carbon	47.9 ± 0.05
Volatile matter	86.4 ± 0.14	Hydrogen	6.3 ± 0.02
Fixed carbon ^b	6.8 ± 0.45	Nitrogen	0.6 ± 0.03
Ash	0.4 ± 0.02	Sulfur	–
		Oxygen ^c	45.2 ± 0.23
		Chlorine	0.013 ± 0.001
Metal analysis		Chemical composition ^c	
	wt%		wt%
Na	0.03 ± 0.005	Cellulose	41.8 ± 0.26
K	0.04 ± 0.002	Hemicelluloses	28.1 ± 0.55
Mg	0.01 ± 0.008	Lignin	30.1 ± 0.28
Ca	0.05 ± 0.002		
Si	0.01 ± 0.001		
Cu	0.2 ± 0.002		

(–): Not detected.

^a As a received basis.

^b Calculated by difference.

^c Dry and ash free.

condensable gas leaving the quenching system through a pre-heater and into the fluidized bed reactor. During the pyrolysis, the gas was sampled using Tedlar gas bags (2 L) at intervals of 10 min to analyze their composition. To adjust the pressure in the plant, excess vapor was burned in a flare stack. A diagram of the pyrolysis plant is shown in Fig. 1.

In the experiments, a series of experiments were conducted in the temperature range of 350–520 °C to investigate the influence of reaction temperature on the product yield. To examine the effects of other operational parameters (feed size and feed rate), two experiments were also carried out. In each experiment, the amount of feed material was 200 g, and 2.6 kg of quartz sand was used as the fluidized bed material. In all experiments, product gas was used as the fluidizing medium. The reaction conditions are shown in Table 2.

Experiments R1–R4 were performed to determine the influence of the reaction temperature. R5 and R6, the results of which can be compared with those of R2, were conducted to determine the influence of feed size and feed rate, respectively. The flow rate of fluidizing medium (29–35 Nl/min) was maintained at three times the minimum fluidizing velocity (3 μ_{mf}) and slightly adjusted at different reaction temperatures to maintain a constant vapor residence time in the reactor. The residence time of the pyrolysis gases in the reactor in all of the experiments, which was 0.5–1 s, was calculated according to a reference [16]. Mass balance was established for all the experiments. In the mass balance, the yield of product gas was calculated by multiplying the gas volume and the gas density calculated from the gas composition using GCs. The bio-oil yield presented in the mass balance was the sum of the oils collected by the condensers, impact separator and electrostatic precipitator. The char yield was calculated by the difference between the amount of feed material used and the sum of the other products (gas and bio-oil).

2.3. Analysis of feed material and products

The moisture and ash contents of the feed material were determined according to the European Standards for solid biofuels (EN 14774 and EN 14775) [17]. The volatile matter was determined by the method of ISO standard (ISO 17246) [18], and the fixed carbon was calculated by the difference. The chemical composition of the feed material was analyzed according to the van Soest procedure [19], and the chlorine content was measured by bomb decomposition followed by determination of chloride with an ion

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