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Thermal degradation and compositional changes of wood treated in a semi-industrial scale reactor in vacuum

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ABSTRACT

Heat treatment is an eco-friendly and efficient way to improve the defective properties of woods, such as hygroscopic nature, lack of dimensional stability, and low resistance against biological degradation, and to produce a green and sustainable wood material for construction and buildings. The aim of this study is to investigate the thermal degradation of a hardwood (poplar, *Populus nigra*) and a softwood (fir, *Abies pectinata*) in a semi-industrial scale reactor in which a vacuum environment is adopted to intensify the thermal degradation process. Four different stages of thermal degradation during wood heat treatment are defined, based on the intensity of differential mass loss (*DML*). Meanwhile, a number of analyses on untreated and treated woods are performed to evaluate their thermal degradation characteristics and compositional change during treatment. The FTIR analysis clearly demonstrates the thermal degradation through dehydration, deacetylation, depolymerization, and condensation reactions during the heat treatment. In addition, the XRD analysis indicates an increase in relative crystallinity of cellulose. The correlation of devolatilization index (*DI*) with respect to mass loss of the two wood species is strongly characterized by linear distribution, which is able to provide a simple and useful tool in predicting mass loss of wood treated in wood industry.

1. Introduction

Nowadays, energy shortage and environmental issues have been the biggest challenges facing the world. Accordingly, sustainability, industrial ecology, eco-efficiency, and green chemistry are guiding the development of materials, products, and processes [1]. Forests are the main greenhouse gas sinks in the world, and play an important role in mitigating the climate change. Trees absorb carbon dioxide and utilize water and sunlight to grow and produce oxygen as a byproduct. The resulted materials can be used in construction and paper production, and can provide chemical feedstocks. Furthermore, at the end of a product life cycle, the material constituents can be combusted or composted to return the chemical constituents to the grand cycles [2–4].

Woods are regarded as renewable and sustainable materials. However, the utilization of woods is limited by its poor resistance to fungal attack (low durability) and the lack of dimensional stability [5,6]. Among various techniques for overcoming these problems, Wood heat treatment is an eco-friendly technology because no chemicals are utilized and added into this process to improve the wood's durability and dimensional stability, whereas toxic chemicals may be used in other chemical modification methods [5,6]. The heat treated woods have longer durability and better dimensional stability, stemming from the reduction in water absorption and biological degradation which are caused from the thermal decomposition of hemicellulose, cellulose, and lignin [5,7]. Wood heat treatment (WHT), similar to mild pyrolysis or torrefaction [8], is conducted in an inert atmosphere at temperatures between 180 °C and 240 °C with a low heating rate (0.1–1 $^{\circ}\mathrm{C\,min^{-1}}$). Heat treatment of wood has been widely investigated and carried out since the early 20th century, and the technique has been improved quickly in developing countries and especially in Europe [4].

The vacuum process is a novel and promising technology which is suitable for biomass pyrolysis, carbonization, and wood heat treatment [9–11]. The applications of the aforementioned thermochemical

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Table 1
Literature review of wood pyrolysis, carbonization, and heat treatment in vacuum.

Reaction	Material	Experimental condition	Objective	Reference
Pyrolysis	Palm oil decanter cake	Temperature: 400–600 °C Heating rate: 15 °C min $^{-1}$ Pressure: below 300 hPa	Potential of bio-oil production from palm oil decanter cake (PDC) at various temperatures.	[9]
	Paper waste sludge	Temperature: 300, 425, 550 °C Heating rate: 30 °C min −1 Duration: ~ 54 s Pressure: 80 hPa	Comparison of vacuum, slow, and fast pyrolysis processes to transfer energy from paper waste sludge (PWS) to bio-oil and biochar.	[12]
	Chinese fir sawdust	Temperature: 500 °C Duration: 30 min Pressure: 100 hPa	Characteristics of co-pyrolysis from Chinese fir sawdust (CFS) and waste printed circuit boards (WPCBs) at different mass ratios.	[13]
	Rape straw	Temperature: 400–600 °C $^{-1}$ Heating rate: 4–20 °C min Duration: 15–75 min Pressure: 50–650 hPa	Optimization of bio-oil yield from vacuum pyrolysis of rape straw by using orthogonal design method.	[14]
Carbonization	Teak sawdust	Temperature: 600 °C Heating rate: 20 °C min $^{-1}$ Duration: 60 min Pressure: 50–650 hPa	Preparation of activated carbon from carbonization of teak sawdust, and the investigation of pore structure.	[10]
	Rice husk and sewage sludge	Temperature: 900 °C Heating rate: 10 °C min $^{-1}$ Duration: 120 min Pressure: 50 hPa	Synergetic effect on gas production during co-pyrolysis of rice husk and sewage sludge.	[15]
	Sugar cane bagasse	Temperature: 460 °C Heating rate: 17 °C min $^{-1}$ Duration: 60 min Pressure: 80 hPa	Production of biochar/activated carbon from carbonization of sugar cane bagasse.	[16]
Heat treatment of wood	Norway spruce, silver fir, European larch, European beech, oak, European ash, wild cherry, and black locust	Temperature: 160–230 °C Pressure: 250 hPa	Potential application of near infrared spectroscopy for quality control of heat treated wood from softwood and hard wood	[17]
	Spruce and fir	Temperature: 160–220 °C Duration: 240–900 min Pressure: 210 hPa	Investigation of properties (EMC, color, anti- swelling efficiency and durability) changes after heat treatment.	[18]
	Bracitinga, feroba mica, and cumaru	Temperature: 180–220 °C Duration: 60 min Pressure: 600 hPa	Examination of physical and technical properties of heat treated wood from common South America wood species.	[19]
	Beech	Temperature: 230 °C Heating rate: 0.2 °C min $^{-1}$ Pressure: 200 hPa	Comparison of chemical composition and durability of beech under vacuum and nitrogen.	[20]
	Turkey oak	Temperature: 160 °C Duration: 180 min Pressure: 200–230 hPa	Evaluation of wood thermal modification through combined steam and vacuum process.	[21]

Table 2
Basic properties of poplar and fir.

Wood material	**Poplar (*Populus nigra*)**	**Fir (*Abies pectinata*)**
Density (kg cm^{-3}) [*a]	324	349
Proximate analysis (wt%) [*a]		
Volatile matter (VM)	84.74	85.53
Fixed carbon (FC)	14.70	14.28
Ash	0.56	0.19
Fiber analysis (wt%)		
Hemicellulose	22.45	19.09
Cellulose	49.91	46.32
Lignin	24.61	29.85
Ash and others	3.02	4.75

[a] dry-basis.

processes with the vacuum technique are summarized in Table 1. In a vacuum process, heat is mainly transferred to the sample through conduction, and a vacuum pump is employed to continuously remove volatile compounds released from biomass, thereby accelerating the thermal degradation of polysaccharides in biomass [3]. Vacuum pyrolysis of biomass has been studied which offers a good performance for pyrolysis product yields. The liquid yield from vacuum pyrolysis is typically about 20–40 wt%, which is comparable with that of slow pyrolysis. Meanwhile, vacuum pyrolysis has the advantages of simple reactor design and no requirement of inert gas compared to fast pyrolysis [9,12]. Some studies of biomass carbonization in vacuum have also been carried out, and the obtained results pointed out that the porosity in biochars was intensified, resulting in micro-macroporous structured carbonaceous particles [22]. Moreover, it was reported that biochars obtained from vacuum carbonization have more “open” pores and higher reactivity than atmospheric pyrolysis biochars. Hence, vacuum carbonization is a potential process to promote the performance of char gasification [23].

As far as wood heat treatment in vacuum is concerned, it is an alternative and novel technology for thermal modification of wood where the oxygen content in a reactor is reduced to avoid wood combustion [17]. Wood thermal modification with vacuum has demonstrated the following advantages: (1) efficient drying and thermal modification with reduced energy consumption and duration [21,24]; (2) easier and cheaper management of volatile wastes produced during heat treatment [21,24]; (3) decreasing odor that normally characterizes many thermal treated woods [21,24]; (4) higher reactivity of thermal degradation and more effective to reduce hygroscopicity of wood under vacuum than nitrogen [11,19]; and (5) providing greater color homogeneity of wood products [21,24]. Accordingly, thermal modification of wood in vacuum is an attractive and promising technology to improve wood properties and to be applied in wood industry. The comparison of wood