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Sequential extraction of oak wood sawdust and oxidative degradation of the extraction residue



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ABSTRACT

Oak wood sawdust (OWSD) was extracted under supercritical CO_2 sequentially with petroleum, carbon disulfide (CDS), methanol, acetone, and isometric CDS/acetone mixture to afford extracts 1–5 (E_1 – E_5), respectively. Then the extraction residue (R_E) was subjected to sequential oxidation with aqueous hydrogen peroxide (AHPO)/ acetic anhydride (AAH) at 65 °C to afford soluble portions 1–4 (SP_1 – SP_4). In total, *ca.* 95.8% of organic matter in OWSD became soluble by the sequential extraction and oxidation. No significant difference in the distribution of functional groups was observed in Fourier transform infrared spectra of OWSD and R_E , while the distribution of functional groups in the oxidation residue is significantly different from that in either OWSD or R_E . In total, 117 and 168 compounds were detected in E_1 – E_5 and SP_1 – SP_4 , respectively, with a gas chromatograph/mass spectrometer. E_1 – E_5 are rich in esters, while carboxylic acids are abundant in SP_1 – SP_4 in addition to esters. HO released from AHPO/AAH played a crucial role in oxidatively degrading R_E .

1. Introduction

Biomass can be converted into liquid fuels and value-added chemicals by solubilization, degradation, and subsequent processing [1]. Supercritical fluid extraction (SFE) is a new material separation and refining technology. The most used solvent in supercritical state is CO_2 due to its great versatility, non-explosive, non-flammable, non-toxic and cost-efficient properties along with its easy removability from the solutes. Supercritical CO_2 has a good gas diffusion coefficient, liquid solubility, and zero surface tension and can quickly penetrate into the solid material to extract soluble portions (SPs). It can be mixed with any organic solvent, resulting in a more efficient extraction [2–8]. However, most of organic matter (OM) in many biomass cannot be extracted by the process. Subsequently degrading the insoluble portion is necessary for understanding the macro-molecular structures of the biomass to provide important information on the efficient utilization of the biomass.

Many oxidants, such as H_2O_2 [9], O_2 /alkali [10, 11], RuCl₃ [12–16], NaOCl [17, 18], and aqueous hydrogen peroxide (AHPO)/

acetic anhydride (AAH) [19, 20], have be used for coal conversion under mild conditions. Among them AHPO/AAH is relatively cheap, ecofriendly, and strong oxidant.

In the present investigation, we tried to understand molecular features of OM in oak wood sawdust (OWSD) by sequential extraction with different extrainers under supercritical CO_2 , subsequent sequential oxidation of the extraction residue (R_E) with AHPO/AAH, and multiple analyses of OWSD, extracts, R_E , SPs from the sequential oxidation, and the oxidation residue (R_O).

2. Experimental

2.1. OWSD and reagents

OWSD was collected from a farm in the vicinity of Dunhua City, Jilin Province, China. It was washed and then air-dried for a week, chopped into small pieces, and pulverized to pass through an 80-mesh sieve ($< 180 \,\mu$ m) followed by desiccation in a vacuum at 80 °C for 24 h before use. Table 1 shows data of proximate, ultimate, and group

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	MAs	methyl alkanoates						

Table 1

Proximate, ultimate, and group composition analyses (wt%) of OWSD, $R_{E},$ and $R_{O}.$

Sample	Proximate analysis			Ultimate analysis (daf)				Group composition analysis (db)			
	M _{ad}	$A_{\rm d}$	VM _{daf.}	С	Н	Ν	S	O _{diff}	Cellulose	HC	Lignin
OWSD R _E R _O	5.84	2.54	87.03	46.76 47.72 42.61	5.77 6.02 6.07	0.68 0.63 0.00	0.34 0.31 0.00	46.45 45.32 51.33	44.72 58.74 76.27	31.96 33.49 21.7	17.35 7.24 1.07

daf. : dry and ash-free base; M_{ad} : moisture (air dried base); A_{ad} : ash (dry base, i.e., moisture-free base); $VM_{daf.}$: volatile matter (dry and ash-free base); diff : by difference; HC: hemicellulose.

composition analyses. Petroleum (PE), carbon disulfide (CDS), methanol, acetone, AHPO (30%), and AAH used in the experiment are commercially purchased analytical reagents. All the organic solvents were purified by distillation prior to use. CO_2 used in the experiment is a purchased food reagents from Liyang industry gas Limited company of Jilin city, Jilin Province, China.

2.2. Sequential extraction of OWSD and sequential oxidation of R_E

As Fig. 1 shows, OWSD was extracted under supercritical CO₂ sequentially with PE, CDS, methanol, acetone, and isometric CDS/acetone mixture (IMCDSAMS) to afford extracts 1–5 (E_1 – E_5), respectively. Then R_E was subjected to sequential oxidation with AHPO/AAH at 65 °C to

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