



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

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## ARTICLE INFO

### Keywords:

Oak wood sawdust  
Sequential extraction  
Oxidative degradation

## ABSTRACT

Oak wood sawdust (OWSD) was extracted under supercritical CO<sub>2</sub> sequentially with petroleum, carbon disulfide (CDS), methanol, acetone, and isometric CDS/acetone mixture to afford extracts 1–5 (E<sub>1</sub>–E<sub>5</sub>), respectively. Then the extraction residue (R<sub>E</sub>) was subjected to sequential oxidation with aqueous hydrogen peroxide (AHPO)/acetic anhydride (AAH) at 65 °C to afford soluble portions 1–4 (SP<sub>1</sub>–SP<sub>4</sub>). 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<sub>E</sub>, while the distribution of functional groups in the oxidation residue is significantly different from that in either OWSD or R<sub>E</sub>. In total, 117 and 168 compounds were detected in E<sub>1</sub>–E<sub>5</sub> and SP<sub>1</sub>–SP<sub>4</sub>, respectively, with a gas chromatograph/mass spectrometer. E<sub>1</sub>–E<sub>5</sub> are rich in esters, while carboxylic acids are abundant in SP<sub>1</sub>–SP<sub>4</sub> in addition to esters. HO-released from AHPO/AAH played a crucial role in oxidatively degrading R<sub>E</sub>.

## 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<sub>2</sub> 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<sub>2</sub> 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<sub>2</sub>O<sub>2</sub> [9], O<sub>2</sub>/alkali [10, 11], RuCl<sub>3</sub> [12–16], NaOCl [17, 18], and aqueous hydrogen peroxide (AHPO)/

acetic anhydride (AAH) [19, 20], have been 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<sub>2</sub>, subsequent sequential oxidation of the extraction residue (R<sub>E</sub>) with AHPO/AAH, and multiple analyses of OWSD, extracts, R<sub>E</sub>, SPs from the sequential oxidation, and the oxidation residue (R<sub>O</sub>).

## 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 μm) 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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Nomenclature			
AAAs	acetylalkanoic acid	MAs'	methyl alkenoates
AAs	alkanoic acids	MC	molar content
AAs'	alkenoic acids	MHAs	methyl hydroxyalkanoates
AAH	acetic anhydride	MM	mineral matter
ADAs	alkanetricarboxylic acid	MSs	monosaccharides
ADOs	alkanediones	NADAs	normal alkanedioic acids
AHPO	aqueous hydrogen peroxide	NAs	normal alkanes
ANSAs	alkyl non-substituted acetates	NCCAs	nitrogen-containing carboxylic acids
BADAs	branched alkanedioic acids	NSAAs	non-substituted alkenoic acids
BAAs	branched alkanes	NSADAs	non-substituted alkenedioic acids
BCA <sub>s</sub>	benzenecarboxylic acid	OAs	other acetates
BTAs	benzenetricarboxylic acid	OAAAs	oxyalkanoic acids
CDS	carbon disulfide	OADAs	oxyalkanedioic acids
CAs	carboxylic acids	ODCs	other dicarboxylates
DAAAs	diacetoxyalkanes	OEEs	other ethyl esters
DAPs	dialkyl phthalates	OSADAs	other substituted alkanedioic acids
DAs	diacetates	OCs	other compounds
DHFD0 & DHFD0Ds	dihydrofuran-2,5-dione & dihydrofuran-2,5-dione derivatives	OEs	other esters
DMADs	dimethyl alkanedioates	OKs	other ketones
DOAAs	dioxyalkanoic acids	ONCOGs	other nitrogen-containing organic compounds
EAAAs	ethyl acetoxyalkanoates	OM	organic matter
EAs <sup>I</sup>	1,2-epoxyalkanes	OMCAs	other monocarboxylic acids
EAs <sup>II</sup>	ethyl alkenoates	OWSD	oak wood sawdust
EAs <sup>III</sup>	ethyl alkenoates	PAs	phthalic acids
EHAAs	ethyl hydroxyalkanoates	PMAAs	polymethoxyalkanes
E <sub>1</sub> -E <sub>5</sub>	extracts 1–5	PE	petroleum ether
F & FDs	furan-2(5H)-one & furan-2(5H)-one derivatives	RC	relative content
FTIR	Fourier transform infrared	R <sub>E</sub>	extraction residue
GC/MS	Gas chromatograph/mass spectrometer	R <sub>O</sub>	oxidation residue
GSs	glycosides	SCOCs	sulfur-containing organic compounds
HAAAs	hydroxyalkanoic acids	SADAs	substituted alkenedioic acids
2-HMSAs	hydroxy-2-methylsuccinic acid	SBAs	substituted benzoic acids
IMACDSMS	isometric acetone/carbon disulfide mixed solvent	SFE	supercritical fluid extraction
MAs	methyl alkenoates	SP <sub>1</sub> -SP <sub>4</sub>	soluble portions 1–4
		TAs	tartaric acids
		TAs'	triacetates

**Table 1**  
Proximate, ultimate, and group composition analyses (wt%) of OWSD, R<sub>E</sub>, and R<sub>O</sub>.

Sample	Proximate analysis			Ultimate analysis (daf)					Group composition analysis (db)		
	M <sub>ad</sub>	A <sub>d</sub>	VM <sub>daf</sub>	C	H	N	S	O <sub>diff</sub>	Cellulose	HC	Lignin
OWSD	5.84	2.54	87.03	46.76	5.77	0.68	0.34	46.45	44.72	31.96	17.35
R <sub>E</sub>				47.72	6.02	0.63	0.31	45.32	58.74	33.49	7.24
R <sub>O</sub>				42.61	6.07	0.00	0.00	51.33	76.27	21.7	1.07

daf.: dry and ash-free base; M<sub>ad</sub>: moisture (air dried base); A<sub>d</sub>: ash (dry base, i.e., moisture-free base); VM<sub>daf</sub>: 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<sub>2</sub> used in the experiment is a purchased food reagent from Liyang industry gas Limited company of Jilin city, Jilin Province, China.

## 2.2. Sequential extraction of OWSD and sequential oxidation of R<sub>E</sub>

As Fig. 1 shows, OWSD was extracted under supercritical CO<sub>2</sub> sequentially with PE, CDS, methanol, acetone, and isometric CDS/acetone mixture (IMCDSAMS) to afford extracts 1–5 (E<sub>1</sub>–E<sub>5</sub>), respectively. Then R<sub>E</sub> was subjected to sequential oxidation with AHPO/AAH at 65 °C to

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