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Vacuum pyrolysis of woody biomass to bio-oil production

Odunsu biyokütleden vakum piroliz ile biyoyakıt üretimi

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Highlights

- ❖ Bio-oil is an encouraging renewable energy source due to finite fossil resources.
- ❖ Bio-oil, derived from oak wood was examined by a series of chromatographic/spectroscopic methods including elemental composition, FT-IR, and GC/MS analysis to determine the chemical structure.
- ❖ The bio-oil comprises a complex mixture of oxygen-containing aromatic compounds such as phenols, alcohols, ketones, aldehydes, organic acids, and benzenes.

Graphical Abstract

In this study, vacuum pyrolysis of oak wood (*Quercus petraea* (Mattuschka) Lieb.) residue was performed in a fixed-bed reactor at a high-temperature of 500 °C.

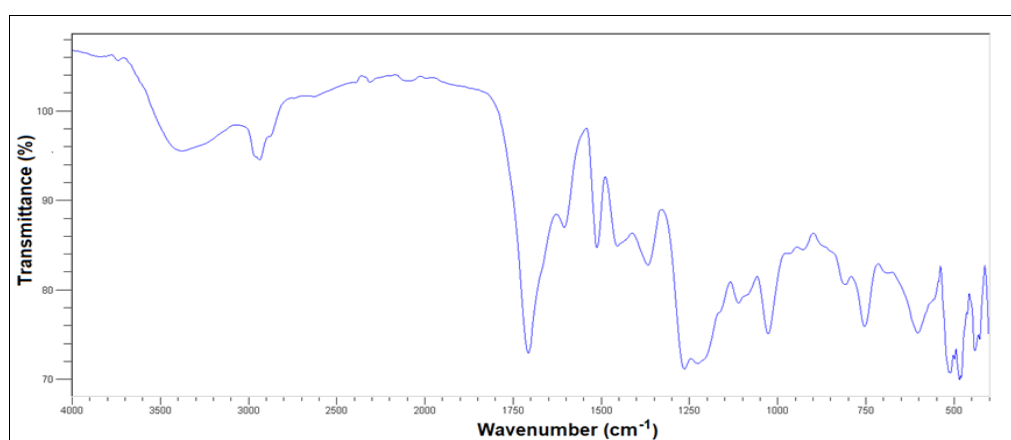


Figure. FT-IR spectrum of the bio-oil

Aim

The purpose of this paper is to the examination of the bio-oil produced from the vacuum pyrolysis of oak (*Quercus petraea* (Mattuschka) Lieb.) woody biomass in Turkey.

Design & Methodology

Pyrolysis experiments were conducted using a fixed-bed reactor at a high-temperature of 500 °C under a vacuum atmosphere. Bio-oil was characterized using some chromatographic/ spectroscopic methods including ultimate, Fourier transforms infrared spectra (FT-IR), and Gas Chromatography/Mass Spectrometry (GC/MS) analysis respectively.

Originality

There are few studies on vacuum pyrolysis of the woody biomass in the related literature.

Findings

Chemical analysis has shown that the bio-oil has valuable chemical compounds, especially phenol and its derivatives.

Conclusion

Wood-derived bio-oil may be used as an alternative fuel or chemical feedstock in different industrial applications.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Vacuum Pyrolysis of Woody Biomass to Bio-oil Production

Araştırma Makalesi / Research Article

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ABSTRACT

Woody biomass is an important resource that can be utilized to obtain liquid fuel. Bio-oil is an encouraging renewable energy source due to finite fossil resources. In this study, vacuum pyrolysis of oak wood (*Quercus petraea* L.) residue was performed in a fixed-bed reactor at a high-temperature of 500 °C. Bio-oil, derived from oak wood was examined by a series of chromatographic/spectroscopic methods including elemental composition, FT-IR, and GC/MS analysis to determine the chemical structure. All the results indicated that the bio-oil comprises a complex mixture of oxygen-containing aromatic compounds such as phenols, alcohols, ketones, aldehydes, organic acids, and benzenes. The major compounds were identified as phenol and phenol derivatives. Bio-oil, produced from woody biomass may be used as an alternative fuel or chemical feedstock in different industrial applications.

Keywords: Bio-oil, GC/MS, oakwood, phenol, vacuum pyrolysis.

Odunsu Biyokütleden Vakum Piroliz ile Biyoyakıt Üretimi

ÖZ

Odunsu biyokütle, sıvı yakıt üretiminde kullanılabilecek önemli bir kaynaktır. Biyoyağ, sınırlı fosil kaynakları nedeniyle umut verici bir alternatif enerji kaynağıdır. Bu çalışmada, meşe ağacı (*Quercus petraea* L.) odun talaşı, 500 °C sıcaklıkta vakum atmosferi altında sabit yataklı reaktörde piroliz edilmiştir. Biyoyağın kimyasal yapısı Elementel, FT-IR ve GC/MS analizi gibi bazı kromatografik ve spektroskopik teknikler kullanılarak analiz edilmiştir. Tüm sonuçlar gösterdi ki, biyoyağın fenoller, alkoller, ketonlar, aldehytler, organik asitler ve benzenler gibi çok çeşitli aromatik bileşikler içerdiği belirlenmiştir. Baskın bileşikler fenol ve fenol türevleri olarak tanımlanmıştır. Odunsu biyokütleden üretilen biyoyağ, alternative yakıt olarak veya farklı endüstriyel uygulamalarda kimyasalların üretiminde kullanılabilir.

Anahtar Kelimeler: Biyoyağ, fenol, GC/MS, meşe odunu, vakum piroliz.

1. INTRODUCTION

Over the last decade, comprehensive development has occurred in the field of renewable and sustainable energy system consisting of solar, wind, geothermal, and biomass due to concern in energy security and limited fossil resources. Biomass energy has increasingly been considered as a fundamental opportunity to substitute conventional fossil fuels, due to their local abundance and easily collection. Biomass can be transformed into fuels and chemicals by thermo-chemical processing such as gasification, torrefaction and pyrolysis. Pyrolysis can be defined as the thermal conversion of biomass into various types forms of energy fuels consisting of liquid, char, and gas in the lack of oxygen/air at the temperature range from 300°C to 900 °C [1-7].

The liquid produced at about 500 °C is referred to as bio-oil. Storage and transportation stability are the important property of bio-oil. Physically, it is a free-flowing liquid with a distinctive smoky smell of dark brown colour. Generally, the chemical composition of the bio-oil highly

depends on the biomass type. In particular, the bio-oil produced from woody biomass mainly composed of oxygen-containing aromatic compounds including phenols, alcohols, esters, organic acids, aldehydes, furans, ketones, and their derivatives [8-13].

The bio-oil obtained from woody biomass pyrolysis has been widely studied in the literature. The influence of pyrolysis conditions such as reactor types, carrier gas type, reaction temperature, pyrolysis atmosphere, etc. was examined in many previous studies. They reported that these conditions changed the content and compositions of the bio-oil obtained [14-22]. However, there are few studies on vacuum pyrolysis of the woody biomass in the related literature [23-25].

The purpose of this paper is to the examination of the bio-oil produced from the vacuum pyrolysis of oak (*Quercus petraea* L.) woody biomass in Turkey. Pyrolysis experiments were conducted using a fixed-bed reactor at a high-temperature of 500 °C under a vacuum atmosphere. Moreover, produced bio-oil was characterized using some chromatographic/spectroscopic methods including ultimate, Fourier transforms infrared spectra (FT-IR), and Gas

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Chromatography/Mass Spectrometry (GC/MS) analysis respectively.

2. EXPERIMENTAL

2.1. Materials

The wood residues from Oak (*Quercus petraea* L.) was supplied by the province of Düzce, Turkey. Before the vacuum pyrolysis, feedstock samples were chipped off and sieved to give a particle size of 0.600–1.000 mm. Air-dried samples were bagged in plastic. Some important properties of the oak wood residue are given in Table 1.

Table 1. Some important properties of oak wood samples

Proximate analysis (%)	Method	Value
Moisture content (%)	[26]	9.47
Volatiles	[27]	77.86
Ash	[28]	0.69
Fixed carbon	calculated from difference	11.98
Component analysis (%)	Method	Value
α -cellulose	[29]	47.93
Holocellulose	[30]	81.21
Lignin	[31]	25.17
Extractive	[32]	4.36
Elemental analysis (%)	Value	
Carbon	51.25	
Hydrogen	6.44	
Nitrogen	0.54	
Oxygen	41.77	
HHV (MJ/kg)	21.64	

The thermal decomposition of the wood sample was analysed by a thermogravimetric analyser (TGA) (Perkin Elmer Pyris 1). TGA experiment consisted of a 10 °C/min heating ramp and N₂ was used as carrier gas. The experiment was conducted at temperatures ranging from 20 °C to 700 °C.

2.2. Pyrolysis Experiments

The pyrolysis equipment was formed of a fixed-bed reactor, an electrical heating oven, a condenser, liquid collectors, a vacuum pump, an electronic control device (PID), K-type thermocouple and other components. The reactor was formed of SS316 (stainless steel) with an inside diameter of 24 cm and a length of 36 cm., which was surrounded by a high-temperature furnace. During the tests, the pyrolysis temperature was measured with the PID. The air inside the reactor was removed by the vacuum pump. The schematic of the equipment is displayed in Fig. 1. A typical vacuum pyrolysis test was carried out at 500 °C. 200 g of the sample was put into the reactor and was it was heated up to 500 °C at a heating

rate of 10 °C/min, and sustained for 60 min under a total vacuum pressure of 10 kPa. The liquid was accumulated the glass-made tanks was weighed at the finish of the test. The bio-char product was collected from the reactor and weighed. The gas yield was determined based on the mass balance equation.

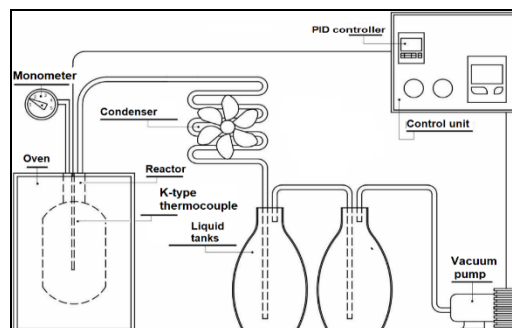


Figure 1. Experimental scheme of the vacuum pyrolysis apparatus

2.3. Methods for the Bio-oil Characterization

The ultimate analyses of the residue and the bio-oil were conducted in the CHNS-932 model analyser. The content of oxygen was calculated by the difference. The high heating value (HHV) was theoretically determined by the formula of the Dulong-Berthelot [33]. The FT-IR analysis of the bio-oil was used as an Alpha FT-IR instrument, in the wavenumber range of 400 to 4000 cm⁻¹. GC/MS characterization of the bio-oil was carried out with an Agilent 6890N GC equipped with an Agilent 5973 mass selective detector using HP 5 column.

3. RESULTS AND DISCUSSION

3.1. TGA Analysis of the Oak Wood Sawdust

Figure 2 displays the TGA curve of the oak wood sawdust. As shown in Fig. 2, the degradation steps of the sample can be divided into three stages: initial degradation starts from 100 °C, it is due to the removal of damp from the wood sample. The second degradation happened at 220–400 °C, due to hemicelluloses and cellulose decomposition. The final decomposition was carried out up to 500 °C was mostly due to the decomposition of lignin; the decomposition was continued to about 700 °C. This result was matched with the TGA curves of wood samples in the literature [34–38].

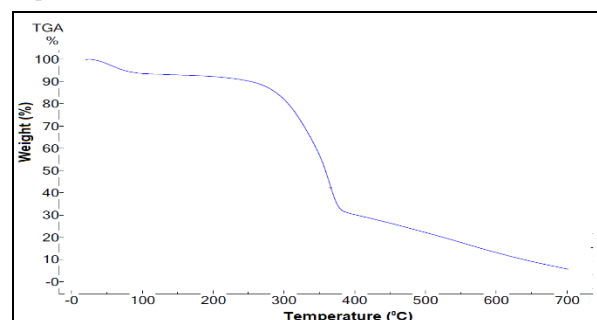


Figure 2. TGA curve of the sample

3.2. Chemical Characterisation

The bio-oil characterisation was examined in this section. The HHV and elemental composition of the bio-oil are summarized in Table 2. A significant reduction in the amount of oxygen was observed, while the carbon content rose from 51.25 % to 61.86 %. The HHV raised from 21.64 MJ/kg to 27.26 MJ/kg., when compared with its feedstock. The HHV of bio-oil is a significant indicator of its classified as a liquid fuel [39]. These results confirmed by previous reports in the literature relating to the elemental determination of the bio-oil produced from woody biomass [40-42].

Table 2. Elemental determination of the bio-oil^a

	C	H	N	O ^b	HHV ^c (MJ/kg)
Feedstock	51.25	6.44	0.54	41.77	21.64
Bio-oil	61.86	6.87	0.21	31.06	27.26

^aash free, ^bBy difference, ^cDulong-Berthelot Correlation.

The bio-oil was analysed by FT-IR, which was conducted to identify the distribution of the functional groups. The spectrum of the bio-oil is shown in Fig. 3. The broad absorption bands in the region between 3600–3200 cm⁻¹ showed that the asset of the O–H groups in the bio-oil. The strong absorbance peak of C–H vibration in the region ~2950 cm⁻¹ is occurred due to the presence of alkenes. The absorbance band at approximately 1750 cm⁻¹ overlapped the C=O stretching band indicating the entity of aromatics. The C–H deformation and stretching vibrations range of 1450–1350 cm⁻¹ is evidence that the presence of alkanes. The spectral range of 1250–800 cm⁻¹ contains various C–H bands that are attributed to the aromatics, in particular phenolic. Ma and co-workers studied bio-oil enriched phenolic compounds obtained from alkali lignin were examined by FT-IR. It was founded that the fingerprint region of aromatic organic compounds such as phenols, alcohols, aldehydes, ketones, and acids was determined between 1000 and 1900 cm⁻¹ [43].

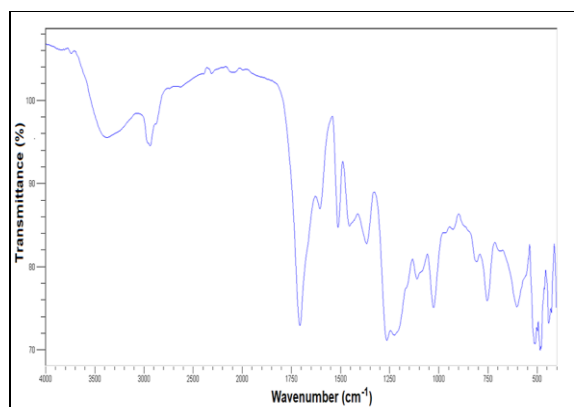


Figure 3. FT-IR spectrum of the bio-oil

GC/MS analysis was conducted for determining the chemical compounds of the bio-oil. The chemical distribution of the bio-oil is listed in Table 3. It was determined that the bio-oil was comprised of a wide species of aromatic oxygenated compounds such as phenols, alcohols, ketones, aldehydes, organic acids, benzenes, and their derivatives. Major phenolic compounds were identified in the bio-oil by GC/MS, including o-cresol, guaiacol, 2,6-xyleneol, creosol, 1,2-benzenediol, 4-ethylguaiacol, syringol, and 4-propenyl-2,6-dimethoxyphenol. The chemical composition of the bio-oil was consistent with the results of the previous GC/MS studies on bio-oil obtained from wood residues [44-47].

Table 3. GC/MS analysis of the oil sample

RT (min)	Category	Name of Compounds	Area (%)
4.88	Ketone	Cyclopentanone	0.31
6.78	Aldehyde	Furan-2-carbaldehyde	3.12
8.58	Alcohol	2-Furylmethanol	2.24
9.84	Furan	Tetrahydro-2,5-dimethoxyfuran	0.51
11.41	Ketone	2-Methyl-2-cyclopentenone	0.81
13.49	Ketone	Furan-2(5H)-one	0.86
14.12	Ketone	Cyclopentane-1,2-dione	1.74
15.89	Aldehyde	5-Methyl furfural	0.71
16.49	Ketone	3-Methyl-2-cyclopentenone	0.43
18.61	Phenol	Phenol	0.94
19.67	Ketone	2,3-Dimethylcyclopent-2-en-1-one	1.76
22.81	Phenol	o-Cresol	2.57
23.57	Phenol	Guaiacol	5.15
25.11	Furan	2-Methyl-1-benzofuran	0.14
25.44	Phenol	2,6-Xyleneol	1.46
28.83	Phenol	p-Ethylphenol	1.15
29.66	Phenol	Creosol	4.29
31.44	Phenol	1,2-Benzenediol	4.11
33.32	Phenol	3-Methoxy-1,2-benzenediol	2.36
33.79	Alcohol	3-Methyl-1,2-benzenediol	1.16
35.01	Phenol	4-Ethylguaiacol	2.67
35.27	Alcohol	1,2-benzenediol- 4-methyl	2.21
35.61	Phenol	p-Vinylguaiacol	1.64
37.58	Phenol	Syringol	8.54
37.64	Phenol	3,4-Bis(methoxy)phenol	1.49
39.24	Phenol	4-Ethylcatechol	0.81
41.75	Acid	3-Hydroxy-p-anisic acid	3.62
41.75	Benzene	Trimethoxybenzene	3.81
45.45	Acid	4-Hydroxy-3-methoxy-benzeneacetic acid	1.15
47.75	Phenol	4-Propenyl-2,6-dimethoxyphenol	6.61

4. CONCLUSION

This study presents an investigation of the bio-oil obtained from the vacuum pyrolysis of oak woody biomass. Produced oil was characterized, using ultimate, FT-IR, and GC/MS analyses respectively. The oxygen content of the produced oil was lower than wood residue and its carbon content was higher. Depending on these results the HHV rising from 21.64 MJ / kg to 27.26 MJ / kg. Chemical analysis has shown that the bio-oil has valuable chemical compounds, especially phenol and its derivatives. Wood-derived bio-oil may be used as an alternative fuel or chemical feedstock in different industrial applications.

DECLARATION OF ETHICAL STANDARDS

The author (s) of this article declare that the materials and methods used in their articles do not require ethics committee permission and/or legal-specific permission.

AUTHORS' CONTRIBUTIONS

Günay ÖZBAY: Performed the experiments and analyse the results. Wrote the manuscript.

Ayhan ÖZÇİFÇİ: Performed the experiments and analyse the results.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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