

The pyrolysis characteristics of wood waste containing different types of varnishes

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Abstract: The wood industry produces large amounts of wood waste. This waste usually contains a number of nonwood materials, such as paints or varnishes. In this study, the pyrolysis characteristics of wood waste containing synthetic, polyurethane, and polyester varnishes were investigated for conversion into renewable liquid fuels. The elemental analysis and higher heating values of the bio-oils were determined. The chemical compounds present in the bio-oils obtained at an optimum temperature were identified by gas chromatography/mass spectroscopy analysis. The product yields and compositions were affected by the types of varnishes. The maximum bio-oil yield of 46.7% was obtained from pyrolysis of waste wood containing polyester varnish at a final pyrolysis temperature of 500 °C. The bio-oil produced from wood waste containing varnishes was composed mainly of phenols, aldehydes, acids, ketones, alcohols, benzenes, and N-containing compounds. The phenols accounted for the largest amount of compounds in the bio-oils. Therefore, the bio-oil produced from varnished wood waste could be a potential substitute for biofuels and green chemicals.

Key words: Bio-oil, gas chromatography/mass spectroscopy, pyrolysis, varnish, waste wood

1. Introduction

Renewable energy has been gaining increasing interest recently since the consumption of fossil energy resources leads to concerns about global climate change, pollution, reduction of availability, and security of the energy supply. Renewable energy includes solar power, hydropower, wind turbines, biomass, and farm methane (Borchers et al., 2007; Zabeti et al., 2012; Mohammed et al., 2014). It is expected to play an important role in the future. Biomass resources include energy crops and agricultural and forestry wastes. Biomass thermochemical conversion routes are usually classified by combustion, gasification, liquefaction, and pyrolysis processes. Pyrolysis is one of the most common thermochemical processes for an efficient way of converting biomass into gas, liquid, and bio-char at temperatures as relatively low as 300–600 °C (Bridgwater, 2003; Balat and Ayar, 2005; Kumar et al., 2009; Murata et al., 2011).

The wooden materials used in various areas have been treated with some wood preservatives to prolong their service life. Therefore, the disposal of wood waste usually contains a number of nonwood materials, such as paints, varnishes, and impregnation materials (Khalfi et al., 2000; Taşçıoğlu and Tufan, 2011). Pyrolysis has been investigated in a number of studies in terms of the

effects of pyrolysis parameters, such as reactor design, temperature, heating rate, residence time, pressure, and biomass type and its characteristics (Ingemarsson et al., 1998; Fagbemi, et al., 2001; Kim et al., 2011; Amutio et al., 2012; Kantarelis et al., 2013; Song et al., 2014). Few studies, however, have reported the effect of wood preservatives on bio-oil properties obtained from pyrolysis of disposed wood (Girods et al., 2008; Ingram et al., 2008; Heo et al., 2010; Kinata et al., 2012; Kim et al., 2014).

The objective of this study was to explain differences in pyrolysis product yields and bio-oil properties produced from wood waste and wood treated with different types of varnishes, and also to determine its possibility of being a potential source of fuel and chemicals.

2. Materials and methods

2.1. Sample preparation

Anatolian black pine [*Pinus nigra* Arn. subsp. *pallasiana* (Lamb.) Holmboe] was used in this study as a biomass feedstock. Anatolian black pine is one of the major construction materials in Turkey.

The proximate analysis was carried out according to the ASTM standard test methods for measuring moisture contents, combustible matter, and ash contents, which are ASTM D 4442-92, ASTM E 897-88 and ASTM D 1102-84,

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respectively (American Society for Testing and Materials, 1997, 1983, 2004). The higher heating value (HHV) of the ash wood sawdust was measured based on the Dulong–Berthelot correlation (Channiwala and Parikh, 2002). The chemical composition of feedstock was performed according to Wise and John (1952), Rowell et al. (2005), and TAPPI standards T-222 and T-204 (Technical Association of the Pulp and Paper Industry, 1997, 2002). The main characteristics of the pine wood are given in Table 1.

Synthetic polymers such as synthetic, polyurethane, and polyester varnishes were used for the finishing process of the samples. During the varnishing process, to avoid interior effects on the layer performance, the emulsion ratios were carefully calculated on the basis of the product specifications. The technical specifications of the varnishes are given in Table 2. Varnished and unvarnished samples were ground and sieved to give fractions with particle diameters in the range of 0.4–1 mm. They were dried in an oven for 12 h at 105 °C before the experiments were performed.

2.2. Thermal analysis

The thermal decomposition behavior of pine wood sawdust was examined using a thermogravimetric analyzer (HITACHI STA 7300). For thermogravimetric analysis,

approximately 10 mg of sample was used with a 5 °C/min heating rate. Nitrogen was used as the carrier gas, with a flow rate of 25 mL/min. After that the sample was heated from 20 °C to 700 °C.

2.3. Pyrolysis experiments

Pyrolysis experiments were carried out using a fixed-bed reactor (6 cm in diameter and 21 cm long) in nitrogen atmosphere as the carrier gas. A schematic diagram of the pyrolysis apparatus is shown in Figure 1. Experiments were performed in two sets. For the first set of the experiments, to determine the effect of the pyrolysis temperature on pyrolysis yields, 50 g of unvarnished wood sawdust was weighed and placed into the reactor, which was heated by an electric furnace. During the experiments, the heating rate and pyrolysis temperature were controlled with a proportional–integral–derivative (PID) controller. The temperature was controlled by a K-type thermocouple inside the bed. The pyrolysis temperatures were 400, 450, 500, 550, and 600 °C with a heating rate of 15 °C /min and were maintained for 30 min. The liquid was condensed in a collector to be weighed. The bio-char yield collected in the reactor was weighed at the end of the experiment. The gas yield was then calculated by the difference. Experiments were repeated at least three times with experimental

Table 1. Main characteristics of the feedstock.

Characteristics	Method	Value
Proximate analysis ^a (%)		
Moisture content (%)	ASTM D 4442-92	6.54
Volatiles	ASTM E 897-88	74.44
Ash	ASTM D 1102 - 84	0.20
Fixed carbon	Calculated from difference	18.82
Ultimate analysis ^a (%)		
Carbon		48.16
Hydrogen		6.15
Oxygen	Calculated from difference	45.69
HHV (MJ/kg)	Calculated from Dulong–Berthelot correlation	19.34
Component analysis ^a (%)		
α-Cellulose	Rowell et al., 2005	48.61
Holocellulose	Wise and John, 1952	70.53
Lignin	TAPPI T 222 om-02	29.67
Extractive	TAPPI T 204 cm-97	3.06
a: Weight percentage on dry basis.		

Table 2. The technical specifications of the varnishes.

Technical properties	Varnish type		
	Synthetic	Polyester	Polyurethane
Density (g/cm ³)	0.94–0.95	-	0.95–0.96
Viscosity (seconds/DIN CUP 4 mm/20°)	18	18	16
Amount applied (g/m ²)	100	200–300	125
Drying type	Physical	Chemical	Chemical
Drying time (20 °C)	6–8 h	4–6 h	2–3 h

error of less than ±0.5%. In the second set of pyrolysis experiments, three different types of varnished wood sawdust were pyrolyzed at the optimum temperature for investigating the effect of varnishes on pyrolysis yields.

2.4. Characterization of bio-oils

The bio-oils selected for characterization were obtained under experimental conditions that gave maximum oil yield. Liquid products were extracted with an equal quantity of diethyl ether. The elemental and compositional properties of the bio-oil were characterized using analytical techniques such as elemental analyzer and gas chromatography/mass spectroscopy (GC/MS). GC/MS analysis has often been used to identify various compounds present in bio-oil. A capillary column (HP-5, 30 m × 0.25 mm i.d. × 0.25 μm) was employed to separate organic mixtures. Diethyl ether was used as a solvent to dilute the dehydrated liquid product to a concentration appropriate for analysis. The carrier gas was helium at a flow rate of 1.2 mL/min. The GC oven temperature was programmed to start at 40 °C, held for 10 min, then raised at a rate of 2 °C to 170 °C, held for 5 min, then raised to 250 °C at a rate

of 8 °C, held for 15 min, then raised to 300 °C at a rate of 15 °C, and held at this final temperature for 10 min. The data acquisition system used was G1035A software with the NIST library.

The elemental analyses of the bio-oils were performed using an elemental analyzer (LECO CHNS 932). The oxygen content was calculated by the difference. The HHV of the bio-oils was calculated based on the Dulong–Berthelot correlation (Channiwala and Parikh, 2002). The chemical composition of the bio-oils was analyzed by GC/MS (Agilent 6890). A capillary column (HP-5, 30 m × 0.25 mm i.d. × 0.25 μm) was employed to separate organic mixtures. The carrier gas was helium at a flow rate of 1.2 mL/min. The GC oven temperature was adjusted to start at 40 °C, held for 10 min, then raised at a rate of 2 °C to 170 °C, held for 5 min, then raised to 250 °C at a rate of 8 °C, held for 15 min, then raised to 300 °C at a rate of 15 °C, and held at this final temperature for 10 min. The end of the column was directly introduced into the ion source of the Agilent 5973 series mass selective detector operated with electron impact ionization mode. The data acquisition system used was G1035A software with the NIST library.

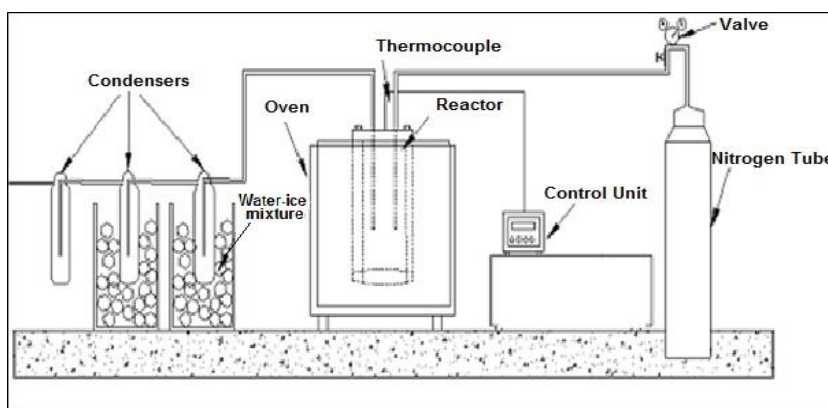


Figure 1. Schematic diagram of the pyrolysis apparatus.

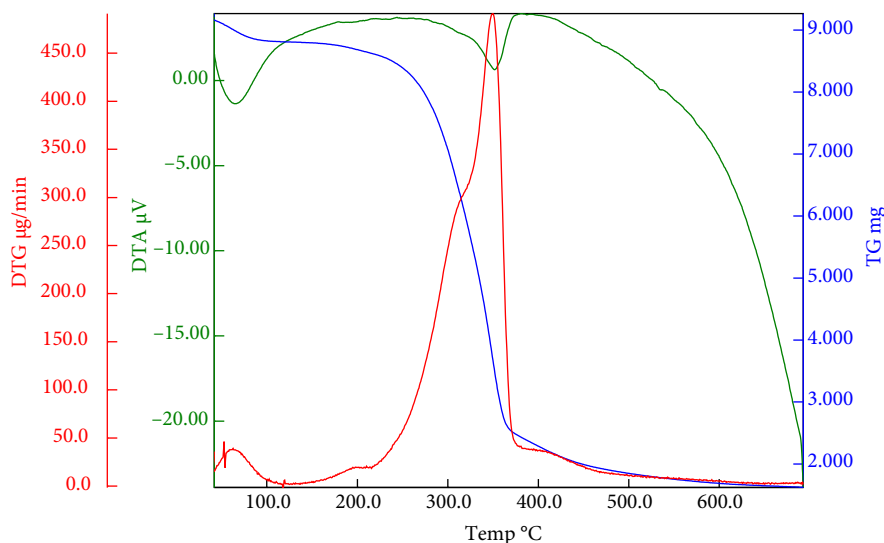


Figure 2. TGA and DTG curves of pine wood sawdust

3. Results and discussion

3.1. Thermogravimetric analysis of the pine wood

The TGA and derivative thermogravimetric (DTG) curves of pine wood sawdust are shown in Figure 2. It can be seen that the moisture in the pine wood was removed at up to 100 °C and the main decompositions of pine wood started around 275 °C and had a rapid incline to around 600 °C. Tutuş et al. (2010) stated that thermal degradation of Scots pine occurred between 300 and 500 °C. TGA is one of the most common techniques used to evaluate thermal behavior during the pyrolysis of wood and other woody biomasses (Ertaş and Alma, 2010a; Özbay, 2015).

3.2. Pyrolysis products from unvarnished and varnished wood

The first set of pyrolysis experiments was carried out at varying final pyrolysis temperatures between 400 and 600

°C. The experimental results are given in Figure 3. At 400 °C, char yield reached its highest value (31.5%). The char yield decreased to 24.9% as the temperature increased from 400 to 600 °C. The bio-oil yield was 42.2% at the pyrolysis temperature of 400 °C, and it reached a maximum of 45.1% at the final temperature of 500 °C. The significant change that we observed was the increase in gas yield for all tested temperatures. The yield of gas increased from 26.3% and 32.1% at temperatures of 400 and 600 °C, respectively. Jahirul et al. (2012) reported that the secondary cracking of molecules produced smaller molecules that enrich the gaseous fraction at higher temperatures. These results were consistent with previous studies (Fagbemi et al., 2001; Phan et al., 2008; Ertaş, 2010; Mourant et al., 2013; Özçifçi and Özbay, 2013).

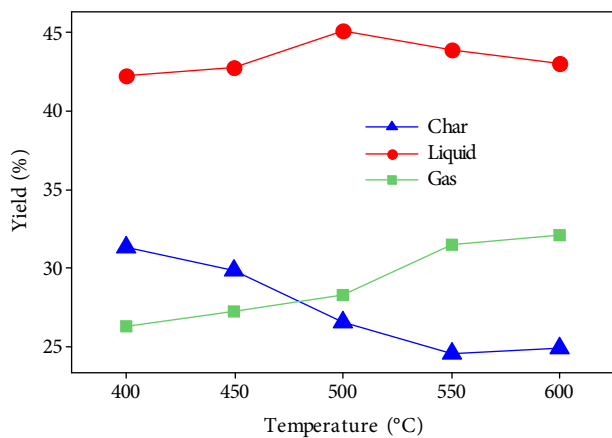


Figure 3. Yields of pyrolysis products

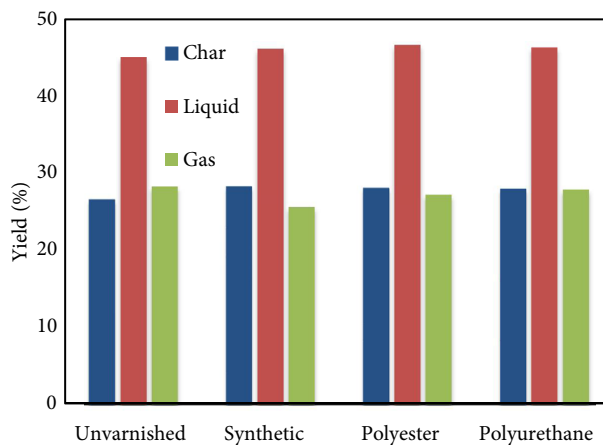


Figure 4. Effects of varnishes on the pyrolysis products

The second set of experiments was performed at a constant pyrolysis temperature of 500 °C. Figure 4 shows the influence of synthetic, polyurethane, and polyester varnishes on the pyrolysis products. All three varnishes increased yields of char and liquid products slightly while yields of gaseous products decreased as compared with pyrolysis of unvarnished wood sawdust at the same temperature. According to Figure 4, higher liquid yield was derived from pyrolysis of wood waste containing polyester varnish (46.7%). Conesa et al. (2009) carried out pyrolysis of various wastes including polyurethane-based varnish, which was obtained from the furniture industry. They reported that high yields of the liquids can be obtained at moderate temperatures.

3.3. Characteristics of bio-oil

The bio-oils obtained from pyrolysis of unvarnished and varnished wood waste were characterized by elemental and GC/MS analysis. The elemental composition and heating values of the bio-oils are listed in Table 3. As can be seen from Table 3, the elemental composition of the bio-oils was clearly affected by containing synthetic, polyurethane, and polyester varnishes during the pyrolysis of wood waste. The presence of polyester varnish caused the significant reduction of oxygen content in the bio-oil (28.68%). The carbon content increased from 56.82% to 64.50% and the HHV increased from 24.60 MJ/kg to 27.59 MJ/kg when compare to unvarnished bio-oil. These differences could be related to the carbon content and oxygen content of the bio-oils. Similar behaviors were observed for bio-oils derived from wood waste containing synthetic and polyurethane varnishes.

Demiral and Ayan (2011) reported that bio-oil has high oxygen content. The high oxygen content is indicative of the presence of many highly polar groups leading to high viscosities and boiling points, as well as relatively poor chemical stability.

Table 3. Elemental composition of the bio-oils.^a

	C	H	O ^b	HHV (MJ/kg)
Unvarnished	56.82	7.20	35.98	24.60
Synthetic varnished	63.26	6.92	29.82	27.15
Polyester varnished	64.50	6.82	28.68	27.59
Polyurethane varnished	62.97	6.50	30.53	26.49

a: Dry basis, b: calculation from difference.

Figure 5 shows the chromatograms of GC/MS analysis of the bio-oils from pyrolysis of waste wood containing different types of varnishes. More than 35 compounds were determined, and the main compounds were listed in Table 4. All the determined products were classified into seven groups including aldehydes, ketones, phenols, acids, benzenes, alcohols, and N-containing compounds, as presented in Figure 6. As listed in Table 4, some very interesting trends were observed. First of all, the compositions of the bio-oils varied depended on the type of varnishes, while most of the identified compounds were found to be similar. We found that phenols, including 2-methoxy-phenol, 2-methoxy-4-methyl-phenol, 4-ethyl-2-methoxy-phenol, 2-methoxy-4-propyl-phenol, and 2-methoxy-4-(1-propenyl)-phenol, were the dominant compounds in the bio-oils. The presence of polyurethane varnish significantly increased the levels of phenols. During the thermal degradation of lignin it produces phenolics that are valuable chemicals and can be used as intermediates in the synthesis of pharmaceuticals, for the production of adhesives, and in the synthesis of specialty polymers (Roy et al., 2000; Bu et al., 2012). Therefore, most of the prior studies were based on phenol production from biomass (Ku and Mun, 2006; Windeisen and Wegener, 2008; Ertaş and Alma, 2010b; Žilnik and Jazbinšek, 2012; Özbay et al., 2013; Trinh et al., 2013).

The aldehydes in the bio-oils ranged from 7.49% to 12.45%. The aldehydes were mainly composed of furfural and 5-methyl-2-furancarboxaldehyde. The varnishes were also responsible for the production of N-containing compounds, such as pyrazine and pyridine. Heo et al. (2010) noticed that waste furniture has various additives, such as adhesives, coating materials, and dyes, because the resulting bio-oil composition may be somewhat different from that of fresh wood. Our results were in agreement with the results of previous studies (Chiaramonti et al., 2007; Shen and Gu, 2009; Lu et al., 2011; Ren et al., 2012; Bu et al., 2013; Kantarelis et al., 2013).

3.4. Conclusion

The effects of varnishes on product yields and compositions during the pyrolysis of wood waste were investigated. From the experimental results obtained, varnishes affected both yield and composition of the bio-oil. The maximum bio-oil yield of 46.7% was obtained from pyrolysis of wood waste containing polyester varnish at a final pyrolysis temperature of 500 °C. The bio-oils were composed of more than 35 low-molecular-weight compounds, mainly phenols, aldehydes, acids, ketones, alcohols, benzenes, and N-containing compounds. The presence of polyurethane

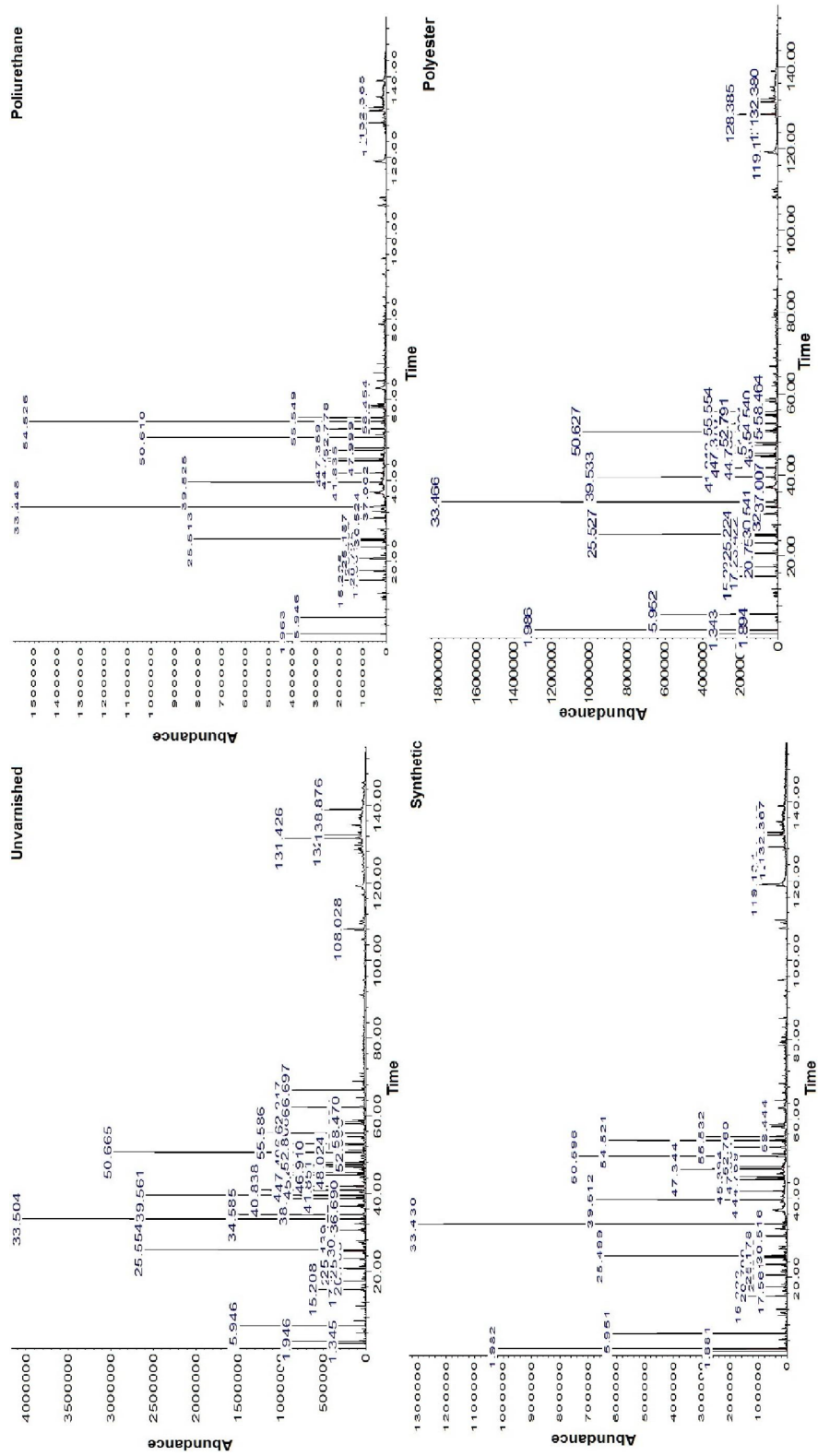


Figure 5. The chromatograms of the bio – oils

Table 4. Main organic components of bio-oils.^a

RT (min)	Name of compound	Area (%)				Category
		Unvarnished	Synthetic varnish	Polyester varnish	Polyurethane varnish	
5.94	Furfural	2.31	4.92	3.36	2.28	Aldehyde
15.21	5-Methyl-2-furancarboxaldehyde	1.52	2.14	2.22	2.01	Aldehyde
17.47	Phenol	0.85				Phenol
17.67	Pyridinamine	-	1.08	1.96	1.24	N-con. Comp.
20.71	3-Methyl-1,2-cyclopentenone	1.93	3.78	2.64	3.18	Ketone
23.35	Benzenemethanol	1.62	-		-	Alcohol
23.39	2-Methyl-phenol	-	1.48	1.87	1.58	Phenol
25.14	4-Methyl-phenol	0.83	2.57	4.02	2.80	Phenol
25.55	2-Methoxy-phenol	8.27	8.93	9.74	9.16	Phenol
30.48	3,5-Dimethyl-phenol	0.64	1.71	2.07	1.82	Phenol
32.27	3-Methyl-4-methylenecycloheptanone	-	-	1.03	-	Ketone
33.50	2-Methoxy-4-methyl-phenol	15.72	20.29	20.95	20.17	Phenol
34.59	1,2-Benzenediol	7.33	-	-	-	Phenol
36.69	5-(hydroxymethyl)-2-furancarboxaldehyde	1.44	-	0.54	-	Aldehyde
38.73	3-Methyl-1,2-benzenediol	2.49	-			Phenol
39.56	4-Ethyl-2-methoxy-phenol	7.44	9.88	9.73	9.65	Phenol
40.84	4-Methyl-1,2-benzenediol	4.49	-		-	Phenol
41.82	Pyrazine	-	1.34	2.41	1,35	N-con. Comp.
41.85	2-Methoxy-4-vinylphenol	1.20				Phenol
44.77	2-methoxy-4-(2-propenyl)-phenol	-	-	1.94	2.58	Phenol
44.80	1,3-Difluoroazulene	2.91	-		-	Benzene
45.43	2-Methoxy-4-propyl-phenol	2.08	2.77	2.76	2.69	Phenol
46.90	4-Acetyl-1,5-dimethylpyrazole	2.19	-	-	-	Ketone
47.41	3-Hydroxy-4-methoxy-benzaldehyde	2.85	5.39	3.30	3.20	Aldehyde
50.67	2-Methoxy-4-(1-propenyl)-phenol	10.04	11.50	10.03	12.55	Phenol
52.51	4-Propyl- 1,3-benzenediol	0.65	-	-	-	Phenol
52.81	1-(4-Hydroxy-3-methoxyphenyl)-ethanone	2.54	2.90	2.62	2.78	Ketone
54.52	2,6-bis(1,1-dimethylethyl)-4-methyl-phenol	-	3.45	0.27	10.42	Phenol
54.73	3,5-Dimethoxy-4-hydroxybenzaldehyde	-	-	0.62	-	Aldehyde
55.58	1-(4-Hydroxy-3-methoxyphenyl)-2-propanone	3.50	4.34	3.63	4.48	Ketone
58.03	4-((1E)-3-Hydroxy-1-propenyl)-2-methoxyphenol	1.23	-	1.11	1.16	Phenol
58.47	3-Methoxy-4-hydroxyphenone- propane	0.81	0.80	0.69	0.84	Ketone
62.22	Coniferyl alcohol	3.31	-	-	-	Alcohol
66.70	4-Hydroxy-2-methoxycinnamaldehyde	2.60	-	-	-	Aldehyde
108.03	Dihydrostibene	1.89	-	-	-	Benzene
119.14	Dehydro-abietic acid	-	4.96	2.56	1.27	Acid
128.50	7-Oxodehydroabietic acid, methyl ester	-	1.20	-	-	Acid
131.43	2-(2,3-Dimethoxyphenyl)methyl benzoic acid	2.64	1.41	2.76	1.21	Acid
132.36	2-(3,4-Dimethoxyphenyl)-7-hydroxy-4H-1-benzopyran-4-one	1.18	0.95	0.89	1.09	Ketone
TOTAL		98.50	99.13	98.13	99.51	

a: Obtained at 500 °C.

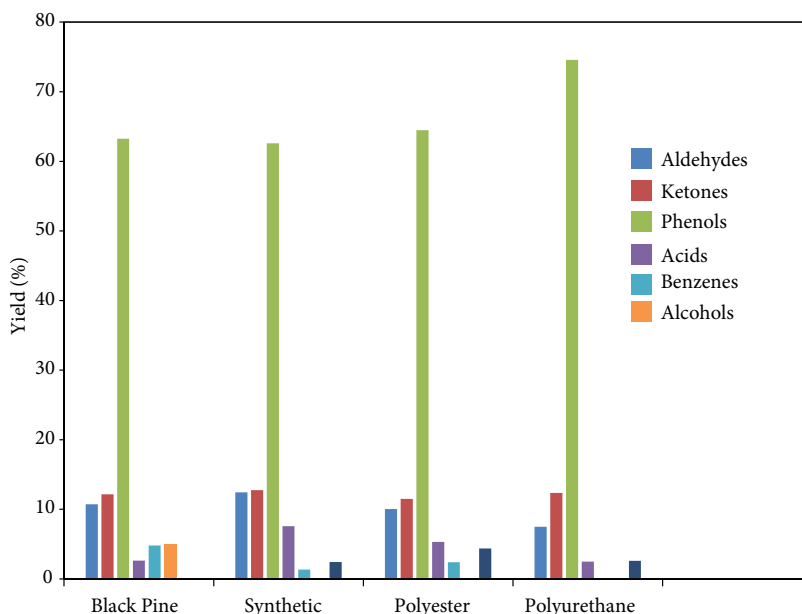


Figure 6. Chemical species of bio - oils^a (peak area %)

varnish significantly increased the levels of phenols. The phenols accounted for the largest amount of compounds in the bio-oils. The bio-oil obtained from varnish-containing wood waste could be used as valuable stock for production of bio-fuels and green chemicals.

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