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Separation of syringol from birch wood-derived vacuum pyrolysis oil

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Abstract

A birch wood sample (*Betula papyrifera*) composed of 54% sapwood and 46% bark was pyrolyzed in a pilot plant reactor. The products were 53.9% condensates (pyrolysis oil including 8% reaction water), 24.0% gas and 22.1% wood charcoal (anhydrous initial feed basis). The pyrolysis oil was steam distilled and the recovery of phenols at various steam pyrolysis oil ratios was studied. A 14.9% by wt. of volatile pyrolysis oil fraction (based on the total feed oil) was recovered at a steam:oil ratio of 27. The distillate was analyzed by GC/MS after acetylation and showed 21.3% by wt. of phenolic compounds on the pyrolysis oil basis. The distillate was further distilled under a total pressure of 0.7 kPa. About 16 sub-fractions were recovered. The steam-distilled fractions were found to be chemically and thermally stable when subjected to further purification processes. The 2,6-dimethoxyphenol (syringol)-rich fraction was separated and purified. Syringol with a purity of 92.3% was obtained. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Birch bark; Oil; Phenols; Purification; Steam distillation; Syringol; Vacuum pyrolysis

1. Introduction

The forest industry produces large volumes of biomass such as bark and sawdust residues. These lignocellulosic residues have a low, if not a negative, value and are usually landfilled, incinerated or burned as cheap fuel [1]. By pyrolysis, biomass residues can be converted into wood charcoal, gas, and oil. Since time immemorial, wood has

been a primary source of chemicals. Acetic acid, acetone, creosote and methanol have been extracted from wood tar. With the alarming depletion of fossil fuel resources and with the public concern about global warming, increasing interest is now directed towards renewables for the production of bioenergy [2]. Current research efforts in the area of biomass pyrolysis concentrate on relatively basic aspects like thermal analysis and analytical studies [3], modeling and simulation [4], pyrolysis processes [2,5] and product research and development [6–8].

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Biomass-derived pyrolytic oils maintain to some extent the chemical identity of the source material. They represent a potential feedstock for chemicals and energy. Pyrolysis oils are highly oxygenated, complex mixtures, viscous, relatively unstable and susceptible to aging [9]. Various chemical classes are present in these oils in different quantities such as acids, alcohols, carbonyls, esters, furans, lactones, and phenols [10]. The yield of each group of compounds depends on the pyrolysis process and operating conditions as well as the nature of the source material [5].

In the past, the utilization of biomass pyrolytic oil has focused on fuel application after partial or major modification. According to Radlein [11], industrial production of bio-oil will focus in the short term on its utilization for the manufacture of higher value materials other than fuels. Due to the presence of a large number of compounds, complete isolation of every constituent of the oil is not possible. The best achievement that may be expected is to separate those compounds that are the most abundant. When separated and purified, some phenolic compounds can be used as a high commercial value chemical feedstock. Some phenols derived from biomass possess anti-diarrhea and anti-motility properties [12], germicidal activity [13], herbicidal and antiseptic properties [14,15]. They have been used in tanning of leather [16], as dyes [17], as thermal insulating material [18] and as food additives [19]. Examples of a

number of pure phenols, which are used in the chemical, food and pharmaceutical industries are shown in Table 1. Several chemical and physical methods have been tested to recover phenols from bio-oil matrices. Examples are alkaline extraction, partition into different solvents and adsorption on different packing materials [23].

The physico-chemical properties of biomass-derived pyrolytic oils, such as their complex composition and thermal instability [24], make conventional separation techniques like distillation less suitable, if not impractical [11]. Solvent extraction and chemical adsorption are frequently used. Phenolic compounds of pyrolytic oils are usually extracted with basic aqueous solutions of 8–15% by wt. of NaOH in soluble polar organic solvent media such as ethyl acetate and methyl isobutyl ketone. Phenol recovery by liquid-liquid extraction of pyrolytic oils has been achieved [7,23]. However, several authors have reported many practical problems associated with the extraction of phenols from pyrolysis oil such as the redistribution of phenols in both phases and precipitation between the organic phases [25]. Liquid chromatography has also been reported to be an efficient technique for the separation of pure phenols. However, high consumption of solvents and regeneration of the adsorbents render liquid chromatography tedious and uneconomical [6].

Steam distillation is a term applied to a process with open steam in direct contact with the mate-

Table 1
Applications of some valuable phenols

Compounds	Applications	References
Catechol	Antiseptic, photography and dyes, electroplating, antioxidant and speciality inks.	[20]
Guaiacol	Sweet aroma, burnt aroma, smokey odor used in synthetic flavors and fragrances and pharmaceutical	[20,21]
4-methylguaiacol	Smokey aroma, smokey test, used in food (candy and baked goods)	[21]
4-ethylguaiacol	Soya sauce flavor, used in non-alcoholic beverages, ice cream, gelatines and puddings	[21]
Syringol	Woody, medicinal and smoky aroma used in food (in meat, soups and seafood), pharmaceutical (platelet aggregation, anti-dermatophyte activity)	[21,22]

rial to be distilled in either a batch or in a continuous operation. Generally, any distillation process conducted in the presence of an inert gas such as nitrogen, carbon dioxide or flue gas is governed by the same fundamental relationship as steam distillation. Steam or inert distillation is usually applied either to separate a small amount of a volatile fraction from a large amount of non-volatile or heat-sensitive materials. Also, steam distillation is often used for the production of essential oils and for the analysis of food and agricultural products.

In the petroleum industry, steam distillation plays an important role during the steam flooding of the light oils. Yields ranging from 12 to 56% of the initial volume of oil at a distillation temperature of 193°C and a pressure of 1.38 MPa have been reported by Wu and Elder [26]. They correlated the yields at a fixed steam to oil ratio with various properties of the original crude oils such as viscosity and molecular weight evolution. Their results were confirmed in a study of ten different crude oils conducted by Duerksen and Hsueh [27]. A few years ago, a model for predicting the increasing hydrocarbon distillation yield with increasing temperature of distillation has been developed [28].

Saturated steam in contact with pyrolysis oil provides heat to the oil, reducing the viscosity and facilitating the vaporization of a portion of the oil into the vapor phase which will be subsequently transferred with steam. In such a case, each volatile compound contained in the oil exerts a partial pressure, which is equal to its vapor pressure. Vasalos et al. [29] performed steam distillation of the acidic fraction of a pyrolytic oil sample after neutralization with 8% NaOH and extraction with CH_2Cl_2 . The authors demonstrated the potential of steam distillation as a recovery method of light phenolic compounds from a biomass-derived pyrolytic oil.

The objective of the present investigation is to demonstrate the potential merits of the steam distillation for the recovery of syringol, a potentially valuable compound [21,22], from *Betula papyrifera*-derived vacuum pyrolysis oil.

2. Experimental

2.1. Vacuum Pyrolysis

The birch wood sample (*B. papyrifera*) used in this work was obtained from Scierie John Lewis, a sawmill in the province of Quebec, Canada. The sample composed of 54% (w/w) sapwood and 46% bark with 8.5% moisture content, was pyrolyzed under vacuum in a Process Development Unit at a throughput capacity of 22 kg/h (run # H-34). The operating conditions were as follows; the total pressure inside the reactor was lower than 3 kPa, the final pyrolysis temperature was 500°C and the heating rate was 10°C/min. The final pyrolysis temperature was held constant for at least 30 min. The pyrolytic oil obtained was stored at 4°C for future analysis. A description of the equipment used can be found elsewhere [30]. The products yields were as follows; 53.9% condensates (i.e. 45.9% anhydrous pyrolysis oil and 8% of miscible reaction water), 24.0% gas and 22.1% wood charcoal (wt.% feedstock moisture-free basis). The total mass of feedstock treated was 116 kg. The mass balance closure was 3.2%. The losses were distributed at pro rata of the pyrolysis product yields.

2.2. Steam distillation

A 100 g of the pyrolytic oil sample was steam distilled batchwise. Steam was generated in a 5 l glass flask and injected at 100°C at a rate equivalent to 5 ml/min of cold water. The pyrolytic oil was kept at 130°C in a mineral oil bath during the distillation to avoid any accumulation of water in the oil flask. The distillate obtained at a temperature of 105°C was condensed using cold water and collected in a glass flask, and then decanted. The aqueous phase was extracted once with an equivalent volume of ethyl acetate, then twice with its half volume of ethyl acetate. The solvent was evaporated and the residual oil was combined with the oil, which was obtained after decantation. The steam-to-pyrolysis oil ratio ($Q_s: Q_o$), the distillation yield and the recovery of phenols were calculated.

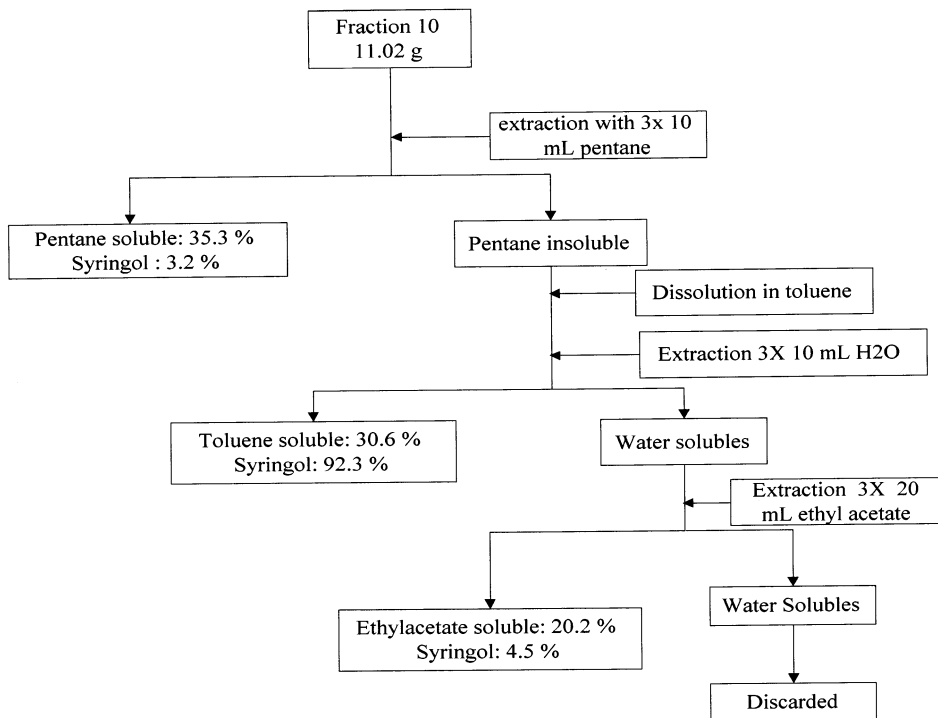


Fig. 1. Syringol purification scheme.

2.3. Flash distillation

The pyrolytic oil was distilled in continuous mode at 200°C under a total pressure of 1.5 kPa. A description of the equipment used can be found elsewhere [23]. The distillate was analyzed by GC/MS for its phenolic content.

2.4. Fractional vacuum distillation

The oil fraction obtained from the steam distillation was evaporated under vacuum at 30°C to eliminate any trace of solvent before the fractional vacuum distillation step. A 440.2 g sample of the oil thereof was further distilled under a total pressure of 0.67 kPa. A column with a height of 850 mm and a diameter of 20 mm was packed with Goodloe™ wire-mesh structured material. About 16 different distillation fractions were collected at a temperature ranging between room temperature and 135°C. All fractions obtained were then submitted to GC/MS analysis.

2.5. Purification of phenols

Fraction # 10 from the vacuum distillation, contained about 33.7% syringol which was further treated according to the following procedure; 10 ml of fraction 10 was extracted three times with 5 ml of HPLC grade pentane. The pentane soluble fraction was concentrated under vacuum. The pentane insoluble fraction was dissolved in 20 ml of toluene and extracted three times with 10 ml of distilled water. The syringol-rich fraction was obtained after the evaporation of toluene. The water fraction was extracted three times with 10 ml of ethyl acetate. The solvent was evaporated off under vacuum and water was discarded (see Fig. 1). The pentane, toluene, and ethyl acetate soluble fractions were submitted to GC/MS analysis.

2.6. Analysis of phenols

Using standard compounds, the identification and quantification of the phenols were carried out

by gas chromatography coupled to a mass spectrometer (GC/MS). The pyrolysis oil was acetylated before analysis. A sample of well-mixed pyrolytic oil (200–300 mg) and the distillate (50–120 mg) were mixed separately with approximately 5 ml of acetic anhydride and two drops of pyridine. The sample mixture was heated in a sealed vial in a water bath at 60°C for 90 min. The derivatized oil solutions were purified on about 2 g of silica gel using 100 ml of 80% CH₂Cl₂ in hexane solution. The eluates were concentrated under vacuum. A 100 µl sample of a solution containing 25 mg anthracene dissolved in 10 ml of ethyl acetate was added to the pre-concentrated solutions as an internal standard. Three sets of derivatized standard phenols diluted in 1:1, 1:2 and 1:3 vol of ethyl acetate were used for the determination of the relative response factors of the acetylated phenols with respect to anthracene.

The GC/MS analysis was performed on a HP-5890 gas chromatograph with split injection at 290°C. The column was a 30 m × 0.25 mm i.d. HP5-MS fused silica capillary with 0.25 µm film thickness from Hewlett Packard. Helium was the carrier gas with a flow rate of about 1 ml/min. The GC initial oven temperature was set at 50°C for 2 min, then programmed to increase to 210°C at 5°C/min and then to 290°C at 10°C/min. The oven temperature was held at 290°C for 10 min. The end of the column was introduced directly into the ion source of a HP-5970 series quadrupole mass selective detector. The transfer line was set at 270°C and the mass spectrometer ion source was set at 250°C with a 70 eV ionization potential. A volume of 1 µl of sample was injected into the GC using a HP-7673 automatic sampler. Data acquisition was carried out with a PC base G1034C Chemstation software and a NBS library database. The mass range of $m/z = 30\text{--}350$ Dalton was scanned every second.

3. Results and discussion

3.1. Steam distillation

The yields of distilled phenols depended on the relative proportion of steam used. Steam co-distills

with phenols and other compounds according to their volatility. At any operating temperature, the components of the pyrolytic oil have different vapour pressures. The volatile components with high vapor pressure tend to concentrate in the vapor phase. Thus, their concentration was reduced from the heavy fraction progressively. Consequently, the highly volatile compounds were distilled first followed by the semi-volatiles and the less volatile compounds. The steam distillate appeared yellow colored and the residue was black and semi-solid at room temperature. The phenolic composition of the various steam distillates obtained is given in Table 2.

The evolution of phenols with respect to the ratio of steam throughput quantity (Q_s) over the initial oil quantity (Q_o) is illustrated in Fig. 2. Both, the quantity of distillate and yield of phenols level off at a Q_s/Q_o of about 27. At $Q_s/Q_o = 27$, the distillation yielded approximately 14.9% distillate (wt.%, initial pyrolysis oil basis). The distillate contained 21.3 phenols or 3.17% by wt. of the pyrolysis oil. Pyrolysis oil before the steam distillation step contained 3.6% phenols. Hence, the total phenol recovery efficiency is 88.2%. The GC/MS analysis of the residue obtained after steam distillation showed a concentration of about 0.08% by wt. of residual phenols in the pyrolysis oil which included trace amounts of catechol, 4-methyl- and 3-methylcatechol, syringol, 4-methoxycatechol, 4-methylsyringol, 4-allyl- and 4-propenylsyringol.

Fig. 3a and b illustrate the evolution of phenol, guaiacol, 4-methylguaiacol, syringol, catechol and 4-methylsyringol at various Q_s/Q_o ratios. The evolution of phenols was found to be dependent on their nature and volatility. Volatile phenols such as phenol and guaiacol were easily removed at the early stage of distillation, using a low Q_s/Q_o ratio. 4-methylguaiacol as an intermediate volatile phenol, reached a maximum recovery at a Q_s/Q_o ratio of about 10. Due to their relatively low volatility, syringol, catechol and 4-methylsyringol (Fig. 3b) require higher Q_s/Q_o ratios compared with phenol and guaiacol. Fig. 4 illustrates the recovery of a number of individual phenols at various Q_s/Q_o ratios.

Table 2
Phenolic compounds distribution in the fractional vacuum distillates (wt.% in the distillate)

Fraction number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Temperature cut (°C)	25–45	45–55	55–60	60–65	65–70	70–80	80–85	85–90	90–95	95–100	100–105	105–110	110–115	115–120	120–130	130–135
Phenol	13.34	12.09	3.24	–	–	–	–	–	–	–	–	–	–	–	–	–
<i>o</i> -cresol	3.98	7.60	3.47	0.54	–	–	–	–	–	–	–	–	–	–	–	–
<i>m</i> -cresol	–	5.43	7.67	5.40	0.68	–	–	–	–	–	–	–	–	–	–	–
<i>p</i> -cresol	0.41	10.47	14.30	9.77	1.09	–	–	–	–	–	–	–	–	–	–	–
2,4-xyleneol	10.11	1.53	0.71	–	–	–	–	–	–	–	–	–	–	–	–	–
2-ethylphenol	–	1.30	1.17	0.80	–	–	–	–	–	–	–	–	–	–	–	–
2,5-xyleneol	–	0.55	1.73	3.13	0.59	–	–	–	–	–	–	–	–	–	–	–
2,3-xyleneol	–	1.11	4.72	9.71	2.24	0.44	–	–	–	–	–	–	–	–	–	–
3-ethylphenol	–	–	–	0.49	3.13	1.07	–	–	–	–	–	–	–	–	–	–
4-ethylphenol	–	–	–	2.11	15.02	5.28	0.43	–	–	–	–	–	–	–	–	–
Guaiacol	10.41	9.92	3.79	0.18	–	–	–	–	–	–	–	–	–	–	–	–
3,5-xyleneol	–	–	–	2.06	1.47	–	–	–	–	–	–	–	–	–	–	–
3,4-xyleneol	–	–	–	–	1.53	2.51	0.30	–	–	–	–	–	–	–	–	–
Trimethylphenol	–	–	1.25	1.59	0.33	1.33	–	–	–	–	–	–	–	–	–	–
Trimethylphenol (isomer)	–	–	–	0.47	1.84	3.31	–	–	–	–	–	–	–	–	–	–
Methylguaiacol	03.6	0.79	0.39	0.44	–	–	–	–	–	–	–	–	–	–	–	–
4-propenylphenol	–	–	–	–	–	1.59	0.86	–	–	–	–	–	–	–	–	–
Trimethylphenol	–	–	–	–	–	0.96	0.38	–	–	–	–	–	–	–	–	–
4-propylphenol	–	–	–	–	–	–	1.72	0.61	–	–	–	–	–	–	–	–
Methylguaiacol (isomer)	–	–	0.47	0.82	–	–	0.45	–	–	–	–	–	–	–	–	–
Trimethylphenol (isomer)	–	–	–	–	1.78	–	–	–	–	–	–	–	–	–	–	–
4-methylguaiacol	–	8.21	14.95	14.10	3.00	0.82	–	–	–	–	–	–	–	–	–	–
4-allylphenol	–	–	–	–	–	0.50	0.75	–	–	0.53	–	–	–	–	–	–
Catechol	–	–	–	–	–	3.27	16.46	35.33	19.82	12.94	0.71	–	–	–	–	–
Trimethylphenol (isomer)	–	–	–	–	–	–	–	–	–	–	–	–	0.51	0.52	0.26	–
Rersorcinol	–	–	–	–	–	–	–	–	–	–	–	–	4.37	3.68	0.80	–
4-ethylguaiacol	–	–	0.62	3.52	9.78	4.72	0.31	–	–	–	–	–	–	–	–	–
3-methylcatechol	–	–	–	–	–	–	1.58	4.50	7.58	9.23	1.31	0.22	0.17	–	0.31	–
Syringol	–	–	–	–	–	–	–	0.93	11.40	33.72	12.09	0.26	–	–	–	–
4-methylcatechol	–	–	–	–	–	–	–	–	0.92	0.75	16.72	14.19	0.65	–	–	–
Methylresorcinol	–	–	–	–	–	–	–	–	–	–	–	–	1.30	1.39	0.49	–
Eugenol	–	–	–	–	–	3.68	3.17	1.64	–	0.66	0.47	–	0.13	0.31	0.35	–
Propylguaiacol	–	–	–	–	–	–	–	–	–	–	–	–	0.18	–	–	–
4-ethylcatechol	–	–	–	–	–	–	–	–	–	–	–	5.23	0.49	–	–	–
Ethylcatechol (isomer)	–	–	–	–	–	–	–	–	–	–	–	2.32	6.84	4.29	0.82	–
Isoeugenol	–	–	–	–	–	–	2.23	2.61	9.00	0.62	–	–	–	–	–	–
4-methylsyringol	–	–	–	–	–	–	–	–	–	–	14.48	25.92	1.50	0.35	–	–
Methoxycatechol	–	–	–	–	–	–	4.68	10.18	–	1.63	–	1.74	–	–	–	–
Dimethylresorcinol	–	–	–	–	–	–	–	–	–	–	–	–	0.60	–	0.19	–
Dimethylresorcinol (isomer)	–	–	–	–	–	–	–	–	–	–	–	–	2.00	1.64	0.44	–
Propenylmethoxyphenol	–	–	–	–	–	–	–	–	–	–	2.26	–	–	–	–	–
Trimethylresorcinol	–	–	–	–	–	–	–	–	–	–	–	–	0.64	0.57	–	–
Trimethylresorcinol (isomer)	–	–	–	–	–	–	–	–	–	–	–	–	0.81	0.46	–	0.27
Isopropylguaiacol	–	–	–	–	–	–	–	–	–	–	–	–	3.76	3.97	1.43	–
Trimethylresorcinol (isomer)	–	–	–	–	–	–	–	–	–	–	–	–	2.94	2.14	0.61	–
Methylmethoxycatechol	–	–	–	–	–	–	–	0.2	0.78	0.55	–	–	4.80	1.95	0.22	–
Propylbenzenediol	–	–	–	–	–	–	–	–	–	–	–	–	1.0	1.5	1.3	0.0
Allylsyringol	–	–	–	–	–	–	–	–	–	–	–	–	8.7	7.6	2.7	0.0
Propylsyringol	–	–	–	–	–	–	–	–	–	–	–	–	0.4	0.1	0.1	–
Dimethylmethoxyresorcinol	–	–	–	–	–	–	–	–	–	–	–	–	2.8	1.2	0.7	–
Isopropylsyringol	–	–	–	–	–	–	–	–	–	–	–	–	1.1	1.5	1.7	–
Propenylsyringol	–	–	–	–	–	–	–	–	–	–	–	–	2.8	4.9	6.2	1.0
Total phenols in distillate	29.61	58.99	58.47	55.14	40.71	33.00	32.21	55.71	49.49	60.63	48.04	49.89	48.96	38.15	18.99	2.87
Distillate yields ^a	13.91	9.59	9.60	5.05	4.54	5.85	4.56	4.53	6.60	6.95	2.68	5.50	4.71	3.05	2.08	1.88

^a wt% on total oil basis.

A progressive decline in the recovery of heavy phenols is also illustrated in Fig. 4. However, the recovery of phenols can be improved by increasing the quantity of steam introduced into the system and/or by increasing the operating temperature. At a steam-to-oil ratio of 12, about 90% of light phenols, such as phenol and guaiacol, were recovered. Approximately 94% of the 4-methylguaiacol was extracted at a Q_s/Q_o of 16. Heavy phenols, such as syringol, catechol and 4-methylguaiacol, followed different recovery patterns. Approximately 80% of the syringol can be recovered using a Q_s/Q_o ratio of 27. But, only 20% of the 4-propenylsyringol was recovered under those operating conditions.

Operating at atmospheric pressure with saturated steam, requires more steam per unit of heavy phenols for their complete extraction. High pressure, on the other hand, enables a considerable saving in the amount of steam required for the process. However, a high operating temperature is less suitable for the stability of the pyrolytic oil and subsequent changes in the chemical composition may occur [9].

Steam distillation produced 14.9% distillate compared with 30.2% obtained by flash distillation at 10 kPa and 200°C. Both distillates contained, respectively 21.3 and 9.8% phenolic compounds, which represent approximately 3.2%

by wt. of the initial pyrolysis oil. Flash distillation yielded more heavy compounds and the final residue obtained was thick. Steam distillation appeared to be more selective, easy to control and yielded light compounds. Due to its low viscosity, the steam distillation residue was easier to handle compared with the residue from the flash distillation process.

3.2. Fractional distillation under vacuum

The vapor pressure of various phenols was plotted as a function of temperature in Fig. 5. The temperature gradient with respect to the pressure variation is more significant at 20 kPa and lower. Thus, the distillate obtained from steam distillation was further distilled into 16 fractions under total pressure of 0.67 kPa within the temperature range of 25–135°C. The distillation curve is presented in Fig. 6. The distillation yielded 93.1% distillate and 6.2% residue. The balance was considered as loss. The distribution and composition of the phenols in the distillates are given in Table 2. The syringol distribution in various distillates is illustrated in Fig. 7, as an example. A high percentage of syringol was obtained in fractions 8–12 in the distillation temperature range of 85–110°C. Heavy syringylic compounds such as 4-methylsyringol, 4-allylsyringol, and 4-propenylsyringol

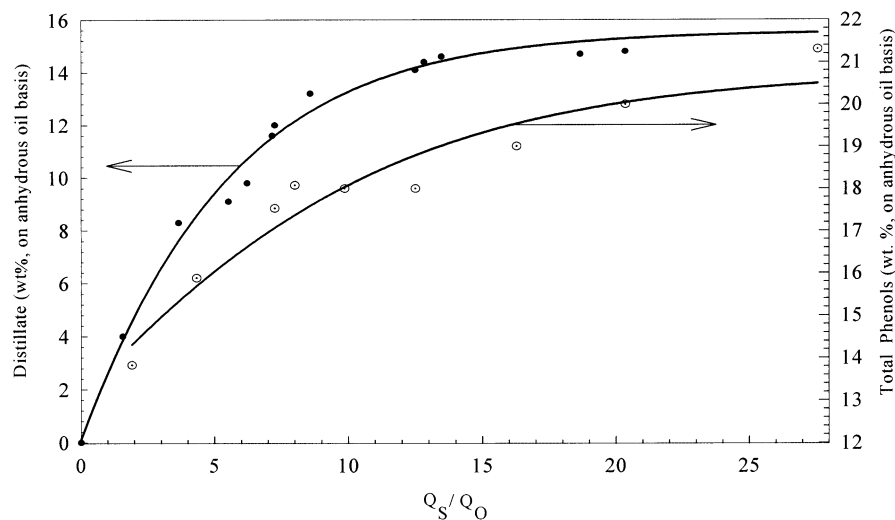


Fig. 2. Evolution of distillates and total phenols as a function of various steam-to-oil ratios.

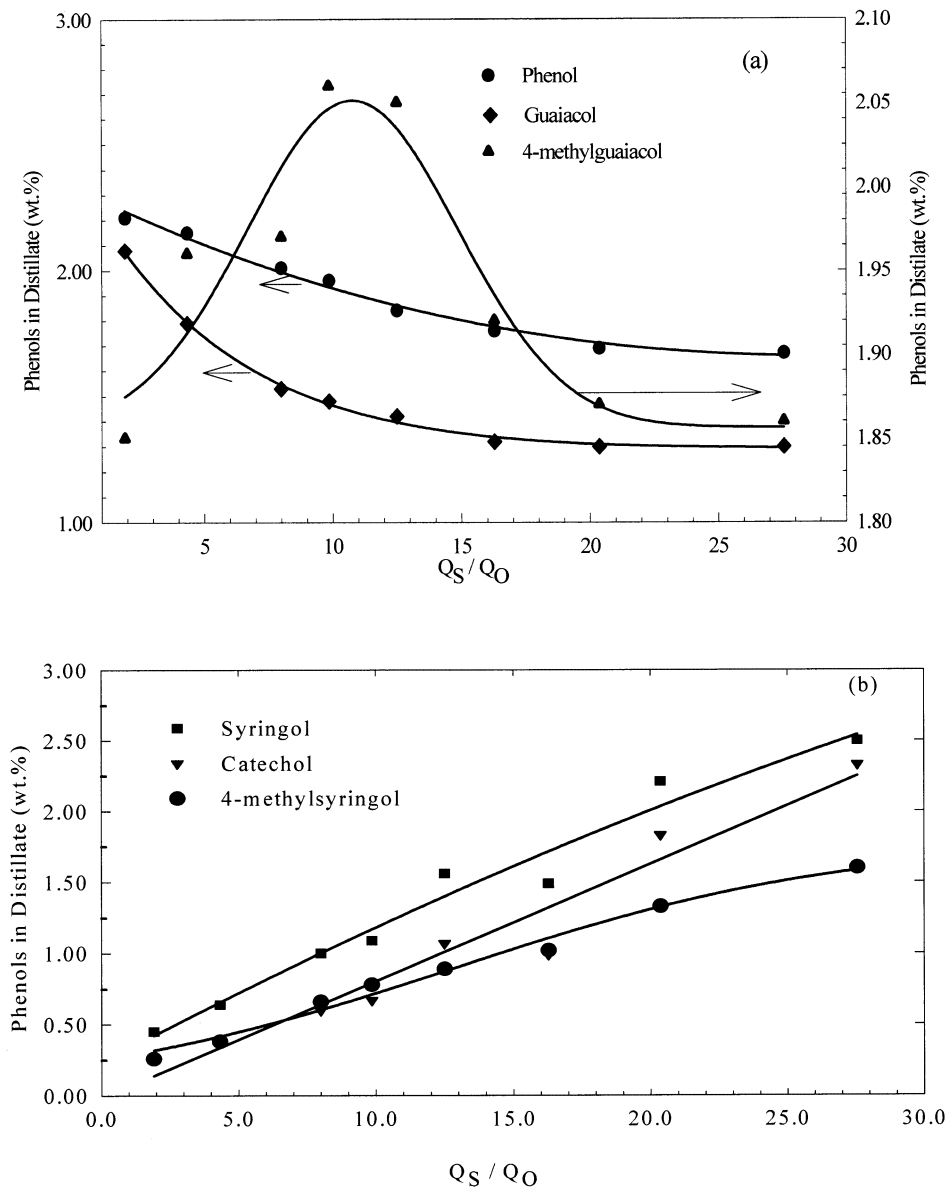


Fig. 3. a and b: Evolution of some selected phenols as a function of various steam-to-oil ratios.

were recovered in fractions 11–16, corresponding to 100–135°C distillation cut. The boiling temperatures of 4-allylsyringol and 4-propenylsyringol have been reported by Pearl [31] in the temperature range of 123–125°C under a pressure of 0.27 kPa, and 107–108°C at 0.007 kPa, respectively.

The syringol concentrated fraction # 10 (Table 2) was further purified following the

scheme outlined in Fig. 1, yielding a product with a purity of 92.3%. The extraction efficiency was 81.1%. The major impurities identified were 2-ethyl-3-hydroxy-(4*H*)-pyran-4-one, 3-methylcatechol, 4-methylcatechol, 2,3-dihydro-(1*H*)-inden-1-one, dimethylhydroquinone, 3,4-dimethoxyphenol, allylmethylphenol, propenylmethylphenol, methyltrimethoxybenzene, isoeugenol, (2-pro-

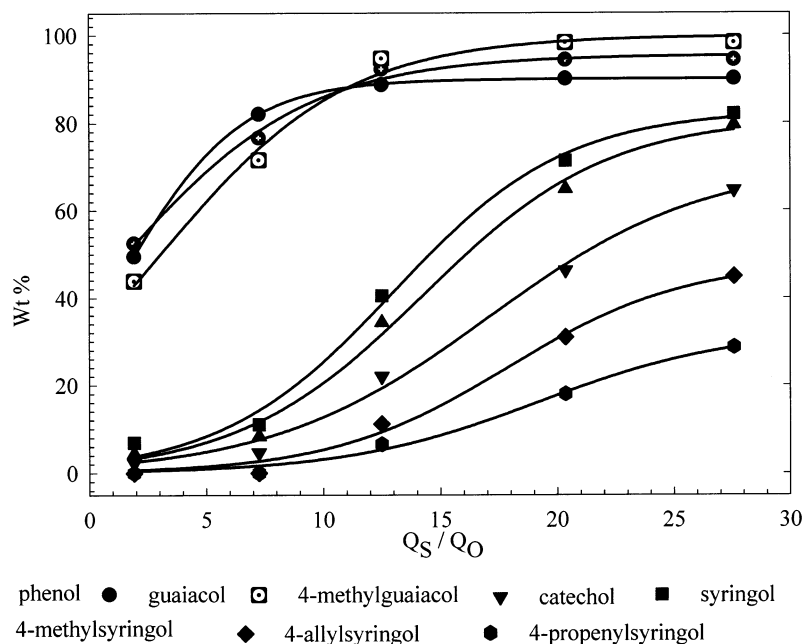


Fig. 4. Recovery of some selected phenols as a function of various steam-to-oil ratios.

penyl)-4-1,2-dimethoxybenzene, fluorene and methyl dibenzofuran. Further purification of the syringol sample might be required depending on the ultimate end-use.

4. Conclusions

Steam distillation of birch wood-derived vacuum pyrolysis oil enables the separation of light compounds from heavy compounds at a low temperature. No significant modification of the chemical composition of the pyrolytic oil was observed. The light steam distilled fraction was concentrated with phenolic compounds. The amount of distillate increased with the relative proportion of steam.

A steam-to-pyrolytic oil ratio of 27 enabled the recovery of 88.2% of the total phenols present in the pyrolytic oil. Heavy phenols such as allylsyringol can be recovered at a high distillation temperature and a high steam-to-oil ratio. The steam-distilled fraction, which contained mainly the volatile compounds (14.9% of the total oil), was successfully redistilled under vacuum into vari-

ous fractions. Pentane and water treatment of the syringol-concentrated fraction yielded syringol with 92.3% purity.

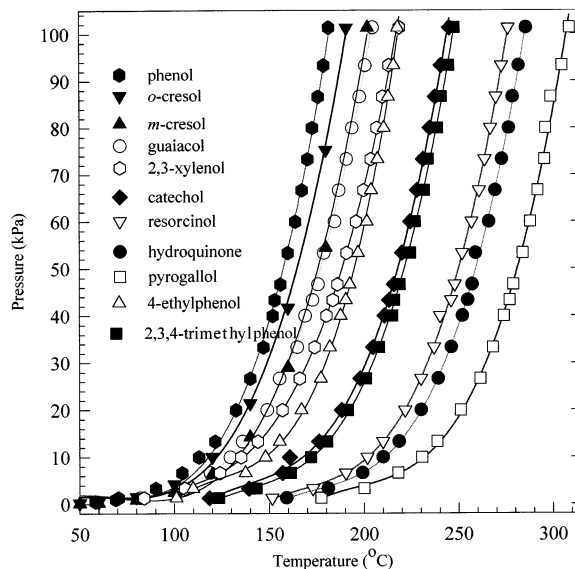


Fig. 5. Vapour pressure of some selected phenols at various temperatures.

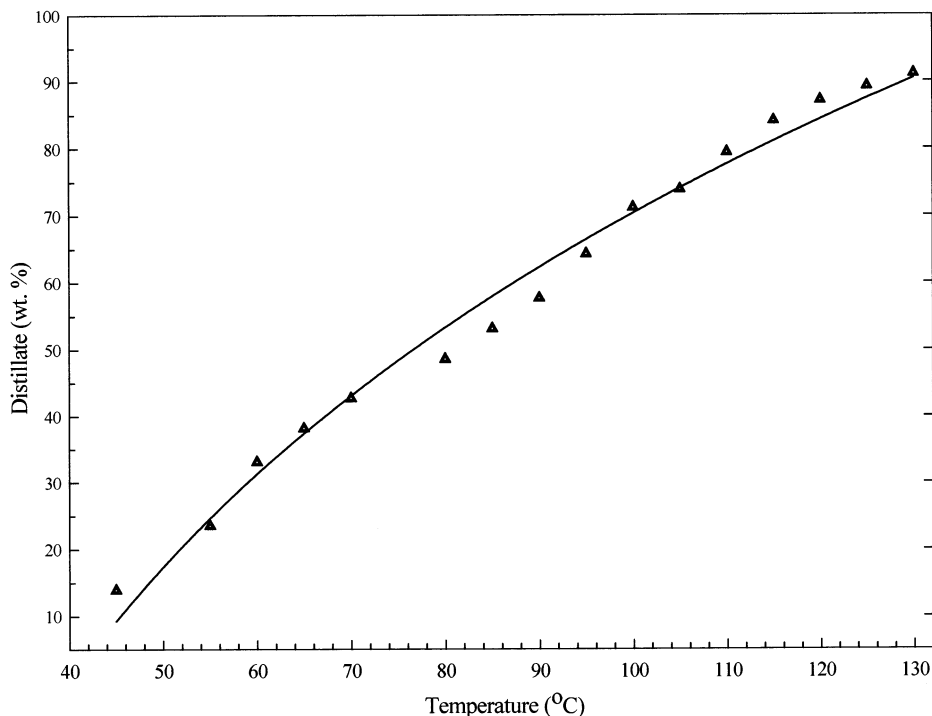


Fig. 6. Vacuum distillation profile of a steam distilled pyrolytic oil.

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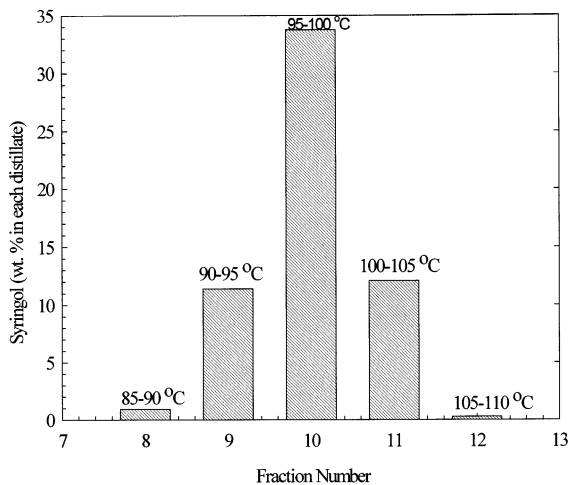


Fig. 7. Distribution of syringol in the fractional vacuum distillates.

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