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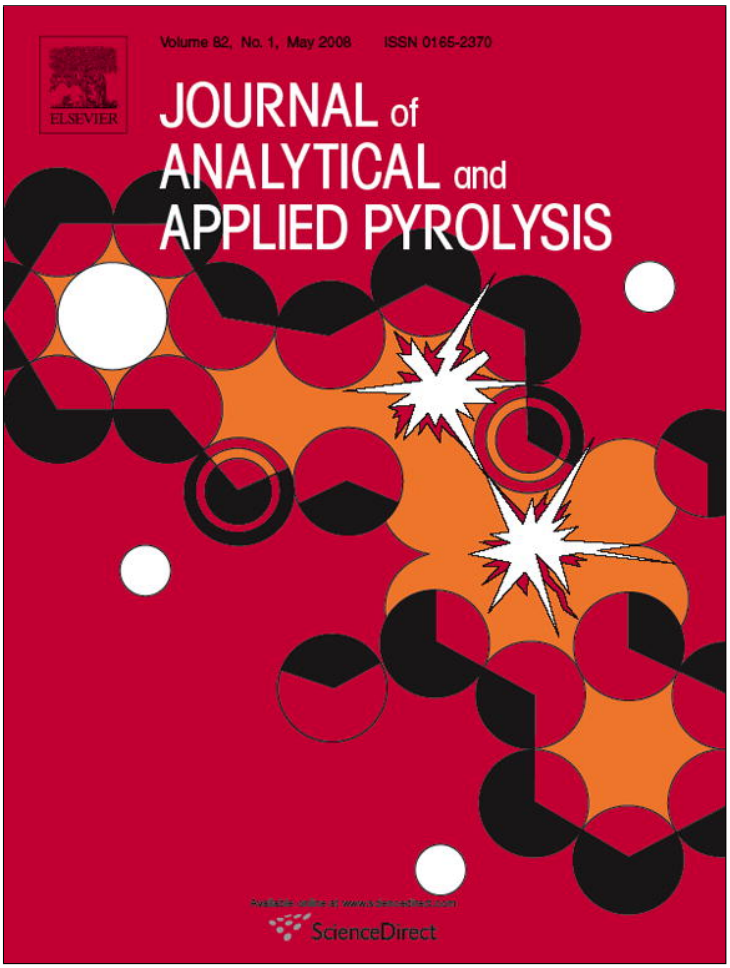

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Understanding the pyrolysis of CCA-treated wood Part II. Effect of phosphoric acid

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ABSTRACT

The pyrolysis process of chromated copper arsenate-treated wood impregnated with phosphoric acid (2–6 wt.%) has been investigated using a Kugelrohr short-path distillation apparatus. The presence of phosphoric acid is shown to change the overall pyrolysis kinetics and yields of products resulting in a decrease in the amount of volatile products and an increase in the char formation. The yield of levoglucosan was found to decrease in the presence of relatively higher concentrations of phosphoric acid, while the yield of levoglucosenone increased. The pyrolysis of chromated copper arsenate-treated wood in the presence of phosphoric acid gave rise to 22% (on water-free tar basis) levoglucosenone. Such data demonstrates the potential for a new pathway toward the rational utilization of chromated copper arsenate-treated wood waste.

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1. Introduction

Waterborne chromated copper arsenate (CCA) has been used to preserve wood against fungi and insects for many years [1]. CCA-treated wood is usually used for outdoor applications, such as telephone poles, decks, fence posts, boat docks, and wooden playgrounds. In recent years, CCA-treated wood has been phased out for any residential use due to the potential health risks associated from the leaching of arsenic-laced preservatives. Environmental friendly recovery and/or disposal technologies for CCA-treated wood have been developed [2–5]. Low-temperature pyrolysis has shown to be a promising approach to this growing disposal problem [2], with the purpose of minimizing metal release and maximizing production of pyrolysis oils. Pyrolysis oils can be used as a source of valuable chemical products [6,7], such as levoglucosan (LG), levoglucosenone (LGO), hydroxyacetone, hydroxyacetaldehyde, isoeugenol, guaiacol, etc.

Both LG and LGO are carbohydrate thermal decomposition products. LGO is a highly functionalized compound owing to its carbon–carbon double bond, the ketone group and the glycosidic anhydro linkage. LG is also an optically active compound. Thus, LG and LGO are attractive chiral raw materials for application in organic synthesis and the pharmaceutical industry. There have been many studies on the formation mechanisms of LG and LGO [8–12]. To obtain high yields of LG and LGO, various factors need to be considered: chemical composition of cellulosic materials, the addition of inorganic catalysts, and the conditions of pretreatment and pyrolysis.

Various additives have been investigated to change the pyrolysis product distribution and convert cellulosic materials to valuable added products. Acid catalysts are known to promote dehydration reactions and favor the formation of LGO. Phosphoric acid has proved to be an efficient catalyst to enhance the yield of LGO from the pyrolysis of cellulosic materials [13–15]. In industrial materials, phosphoric compounds are used as flame retardants for wood, textile finish and polymeric fibers in order to reduce flammability and smoke generation by lowering their weight loss rate and increasing their char yield [16,17]. Since LGO was detected through the thermal degradation of cellulose in the presence of acidic additives such as phosphoric acid in the 1970s [8], this research area has attracted much interest. Phosphoric acid has specific effects on cellulose. Phosphoric acid is known to induce less catalytic degradation to cellulose than other mineral acids [13] and it has lower volatility than that of sulfuric acid or hydrochloric acid [18]. It is also well known for its swelling property resulting in easy penetration into interfibrillar reaction centers [19]. Thus, phosphoric acid promotes diffusion and chemical processes. During pyrolysis, phosphoric acid can change the depolymerization and dehydration mechanisms of cellulose. It has been observed [20–25] that the presence of phosphoric acid in cellulosic materials lowered decomposition temperatures, suppressed volatiles production and increased char yields.

The objective of the present study is to investigate LGO and LG formation from pyrolysis of CCA-treated wood that has been

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pretreated with phosphoric acid, with the purpose of developing a new pathway in the rational utilization of CCA-treated wood waste. In addition, thermogravimetric analytical (TGA) data of pretreated wood samples is also provided.

2. Experimental

2.1. Materials

Southern pine wood and its CCA-treated equivalent (Type C) were used in this study. The active ingredients in this CCA preservative are 47.5% CrO_3, 18.5% CuO, and 34.0% As_2O_5 (based on a 100% oxide). The wood was milled to a powder that had a particle size of 0.1–0.2 mm, and the moisture content was 8%. Chemically pure 85% phosphoric acid was used for impregnation.

2.2. Impregnation with phosphoric acid

Solutions of phosphoric acid (2 wt.%, 4 wt.%, 5 wt.%, 6 wt.% based on dry wood) in methanol were mixed with untreated wood and CCA-treated wood (wood/acid solution = 1:5). Methanol was evaporated under vacuum and the impregnated wood powders were dried over a phosphorous pentoxide desiccator under vacuum.

2.3. Pyrolysis

The pyrolysis of pretreated wood samples was carried out under heat in a Kugelrohr short-path distillation apparatus (Fig. 1). The sample weight was 3 g for each trial. The vacuum pressure was controlled at 7 mmHg. The thermal treatment was carried out at 350 °C for 30 min. The end product—char was collected in the first flask, whereas the tar was collected in two consequently connected receiving flasks (B in Fig. 1) and condensed with ice water (0 °C) for further analyses. The volatiles were condensed with dry ice–acetone at −70 °C in the first trap (E in Fig. 1).

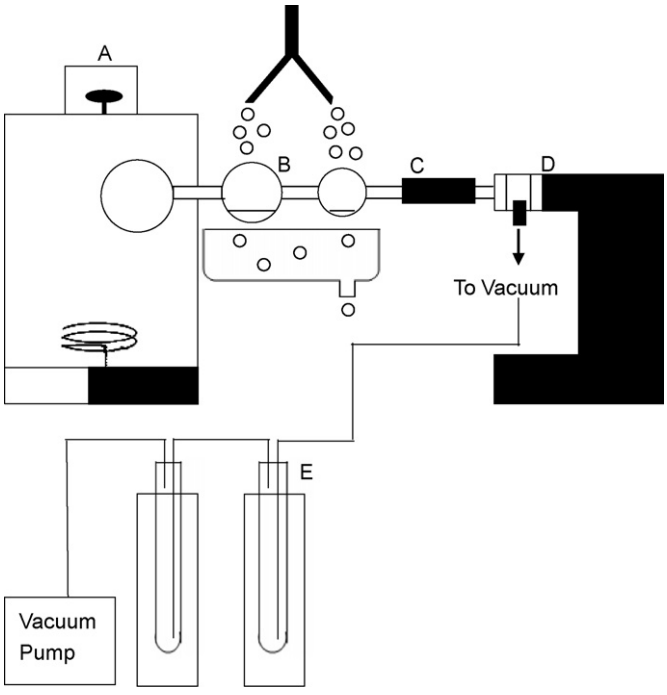

Fig. 1. Kugelrohr short-path distillation apparatus. *Legend*: (A) heated air bath; (B) receiving flasks; (C) joint connection and a hose connector; (D) motor drive; (E) traps.

2.4. Analytical methods

2.4.1. GC/FID analysis

Quantitative tar analysis was performed on a Hewlett Packard HP6890 series GC system, using a DB-1701 (J&W) capillary column (60 m × 0.25 mm i.d. and 0.25-μm film thickness). The temperature program was an isothermal temperature profile at 45 °C for the first 4 min, followed by a 3 °C/min temperature gradient to 270 °C and finally an isothermal period at 270 °C for 7 min. Helium was used as a carrier gas, with a split ratio of 1:20 and a constant flow of 1.0 mL/min. The temperature of the detector and injector were maintained at 280 °C and 250 °C, respectively. For the sample preparation, 70 mg of tar was weighed and dissolved in 5 mL of acetone. Fluoranthene was used as an internal standard. Detailed signal assignments of the emerging gas chromatograms of the obtained pyrolysis tars can be located in Ref. [26].

2.4.2. Metal analysis

The chromium and copper content of pyrolysis char were determined by atomic absorption spectrometer PerkinElmer 2380. Analytical wavelengths were 357.9 nm for chromium and 324.8 nm for copper. The arsenic content of pyrolysis char was determined by inductively coupled plasma (ICP) spectrometer, using a PerkinElmer Optima 2000DV. The samples for metal analysis were prepared according to the reflux method provided by Helsen et al. [27]. This methodology causes the complete sample dissolution in nitric acid without loss of any material. 0.1 g of pyrolysis char was placed into a conical flask equipped with a reflux condenser, followed by the addition of 10 mL of 65% HNO_3. Then the flask was heated on a heating plate under reflux until the sample dissolved completely (approximately 2 h). The concentration of chromium, copper and arsenic in pyrolysis char from the CCA-treated wood pretreated with 4% H_3PO_4 were 82.7%, 87.5% and 74.5%, respectively.

2.4.3. Thermogravimetric analysis

TGA was carried out using a TA Q500 Instrument. The wood powder sample, approximately 10–15 mg in weight, was introduced into a platinum sample pan and heated from room temperature to 600 °C at a heating rate of 50 °C/min under a nitrogen flow of 60 mL/min.

3. Results and discussion

3.1. Quantitative analysis of tar products

Yields of pyrolysis fractions (at 350 °C) of untreated wood and CCA-treated wood pretreated with phosphoric acid are shown in Table 1. The total recovery (the sum of the yields of char, tar and volatiles) from pyrolysis was around 90%. Phosphoric acid pretreatment on wood leads to a considerable change of the thermal decomposition mechanism, which is demonstrated by a decrease in the amount of the volatile products. The tar obtained from the pyrolysis of CCA-treated wood was higher than that obtained from untreated wood, from which it may be concluded that the CCA treatment accelerated the formation of tar. Correspondingly, the amount of char increased with the increase in the concentration of phosphoric acid. This demonstrates that the impregnation of wood powder with phosphoric acid promotes dehydration reactions resulting in the formation of condensed structures, which cannot therefore degrade further to monomeric volatile products [13]. It was reported that metal ions in CCA-treated wood could be extracted by phosphoric acid [28]. This is also one of the options to recycle the CCA components when managing CCA-treated wood waste [1]. In our study, the same amount of phosphoric acid was used to pretreat untreated and

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Table 1
Yields of pyrolysis fractions of H_3PO_4-treated original wood and CCA-treated wood at 350 °C (wt.% based on dry wood)

Fractions (%)	0% H_3PO_4		2% H_3PO_4		4% H_3PO_4		5% H_3PO_4		6% H_3PO_4	
	CCA	Untreated	CCA	Untreated	CCA	Untreated	CCA	Untreated	CCA	Untreated
Char	30.2	28.5	34.7	40.9	41.3	49.8	43.7	49.3	46.2	52.0
Tar	57.7	59.6	40.7	36.4	36.3	19.1	26.3	13.8	20.3	12.1
Volatiles	4.6	3.0	14.7	18.0	13.2	26.6	20.1	24.6	25.6	29.8
Total recovery	92.5	91.1	90.1	95.3	90.8	95.5	90.1	87.7	92.1	93.9

CCA-treated wood. A certain amount of metal ions can be removed by phosphoric acid, which would consume some phosphoric acid. That is to say, less acid will further react with CCA-treated wood material compared to untreated wood. Dehydration reactions would be reduced compared to those for untreated wood. Thus, the formation of condensed structures in CCA-treated wood is less than that in untreated wood. The char from CCA-treated wood was therefore lower than that from untreated wood.

The quantified pyrolysis tar products from untreated wood and CCA-treated wood pretreated with phosphoric acid are listed in Table 2. The yields of some carbohydrate degradation products in the pyrolysis tars are shown in Fig. 2. As can be seen in Fig. 2, the LG yield reached a maximum for both wood materials at the addition of 2% H_3PO_4. For CCA-treated wood, the amount of LG increased to 17.5% and then decreased to 0.6% (based on water-free tar) as the phosphoric acid content rose from 0% to 6%. The untreated wood showed a similar trend on the yield of LG. The amount of LG derived from CCA-treated wood was higher than that from untreated wood over the entire phosphoric acid concentration range. LGO is the main product of catalytic depolymerization resulting from the dehydrated glucopyranose cycles [13]. Its yield rose and reached a maximum of 26.4% (based on water-free tar) for untreated wood at the amount of 4% phosphoric acid. For CCA-treated wood, the amount of LGO increased from 0.9% to 22.3% (based on water-free tar) as the phosphoric acid content rose to 6%. Due to the extraction of some chromium, copper and arsenic from CCA-treated wood by phosphoric acid, the yield of LGO was low at the initial addition of phosphoric acid. Then, at higher phosphoric acid concentration, the yield of LGO increased up to 22.3% via the acidic catalysis of cellulose dehydration process. Because the yield of tar decreased with the addition of phosphoric acid, the yield of LGO (based on dry wood) for untreated wood decreased. But for CCA-treated wood, the yield of LGO (based on dry wood) increased until it leveled off at 3.4%.

For other carbohydrate degradation products, the yield of hydroxyacetaldehyde and furan-(5*H*)-2-one decreased drastically

Table 2
Yields of tar compounds from the pyrolysis of H_3PO_4-treated original wood and CCA-treated wood (wt.% based on water-free tar)

Compound name	0% H_3PO_4		2% H_3PO_4		4% H_3PO_4		5% H_3PO_4		6% H_3PO_4	
	CCA	Untreated	CCA	Untreated	CCA	Untreated	CCA	Untreated	CCA	Untreated
Hydroxyacetaldehyde	1.34 ± 0.07	3.07 ± 0.10	0.35 ± 0.02	0.31 ± 0.05	0.27 ± 0.05	0.19 ± 0.02	0.19 ± 0.01	0.17 ± 0.03	0.16 ± 0.02	0.15 ± 0.02
Acetic acid	1.92 ± 0.07	1.87 ± 0.14	0.49 ± 0.06	0.83 ± 0.04	0.36 ± 0.05	0.29 ± 0.02	0.91 ± 0.04	0.31 ± 0.03	0.99 ± 0.05	0.22 ± 0.02
2-Furaldehyde	0.70 ± 0.03	0.34 ± 0.01	0.27 ± 0.03	0.49 ± 0.02	0.24 ± 0.02	0.14 ± 0.01	0.43 ± 0.02	0.08 ± 0.01	0.35 ± 0.01	0.06 ± 0.01
Furan-(5*H*)-2-one	0.40 ± 0.01	1.10 ± 0.04	0.14 ± 0.02	0.14 ± 0.01	0.13 ± 0.02	0.12 ± 0.02	0.14 ± 0.01	0.15 ± 0.02	0.17 ± 0.02	0.13 ± 0.01
Phenol	0.04 ± 0.001	0.04 ± 0.001	0.05 ± 0.002	0.05 ± 0.001	0.06 ± 0.004	0.05 ± 0.001	0.05 ± 0.002	0.06 ± 0.002	0.07 ± 0.004	0.05 ± 0.001
Guaiacol	0.60 ± 0.04	0.41 ± 0.03	0.23 ± 0.02	0.24 ± 0.01	0.25 ± 0.01	0.22 ± 0.02	0.24 ± 0.01	0.20 ± 0.01	0.30 ± 0.01	0.18 ± 0.02
Levoglucosenone	0.87 ± 0.04	0.19 ± 0.04	8.17 ± 0.38	17.73 ± 0.72	14.14 ± 1.00	26.40 ± 0.53	18.29 ± 0.87	22.74 ± 1.10	22.25 ± 0.48	22.94 ± 0.44
Levoglucosenone[a]	0.40 ± 0.02	0.07 ± 0.02	2.93 ± 0.14	4.67 ± 0.19	3.49 ± 0.24	3.28 ± 0.07	3.46 ± 0.17	1.72 ± 0.08	3.33 ± 0.07	1.71 ± 0.03
2,4-Dimethyl phenol	0.09 ± 0.006	0.13 ± 0.01	0.10 ± 0.01	0.08 ± 0.002	0.08 ± 0.005	0.08 ± 0.001	0.07 ± 0.005	0.11 ± 0.006	0.07 ± 0.005	0.12 ± 0.01
4-Ethyl-guaiacol	0.10 ± 0.008	0.12 ± 0.005	0.07 ± 0.004	0.06 ± 0.006	0.07 ± 0.001	0.06 ± 0.01	0.08 ± 0.007	0.08 ± 0.007	0.08 ± 0.006	0.08 ± 0.004
Eugenol	0.21 ± 0.01	0.22 ± 0.03	0.09 ± 0.006	0.10 ± 0.005	0.10 ± 0.002	0.13 ± 0.01	0.11 ± 0.006	0.16 ± 0.01	0.12 ± 0.01	0.18 ± 0.01
5-(Hydroxymethyl)-furaldehyde-(2)	2.01 ± 0.10	0.99 ± 0.01	1.13 ± 0.06	1.08 ± 0.05	0.81 ± 0.03	1.19 ± 0.06	0.76 ± 0.02	1.30 ± 0.08	0.71 ± 0.02	1.12 ± 0.05
Isoeugenol	0.36 ± 0.02	0.51 ± 0.03	0.11 ± 0.02	0.11 ± 0.01	0.05 ± 0.003	0.03 ± 0.007	0.03 ± 0.003	0.05 ± 0.01	0.03 ± 0.004	0.05 ± 0.008
Vanillin	0.33 ± 0.01	0.35 ± 0.02	0.27 ± 0.01	0.18 ± 0.01	0.38 ± 0.02	0.25 ± 0.01	0.24 ± 0.01	0.34 ± 0.01	0.24 ± 0.01	0.35 ± 0.02
Acetoguaiacone	0.20 ± 0.005	0.23 ± 0.006	0.16 ± 0.005	0.06 ± 0.004	0.11 ± 0.001	0.08 ± 0.01	0.10 ± 0.003	0.11 ± 0.005	0.07 ± 0.002	0.13 ± 0.01
Guaiacyl acetone	0.36 ± 0.02	0.45 ± 0.04	0.26 ± 0.01	0.23 ± 0.03	0.24 ± 0.01	0.36 ± 0.03	0.23 ± 0.01	0.44 ± 0.04	0.23 ± 0.01	0.48 ± 0.03
Levoglucosan	14.66 ± 1.30	4.86 ± 0.20	17.46 ± 1.12	9.41 ± 0.56	7.23 ± 1.40	2.80 ± 0.17	2.77 ± 0.41	0.37 ± 0.03	0.62 ± 0.03	0.61 ± 0.10
Levoglucosan[a]	6.77 ± 0.45	1.88 ± 0.08	6.26 ± 0.40	2.48 ± 0.15	1.79 ± 0.34	0.35 ± 0.02	0.52 ± 0.08	0.03 ± 0.003	0.09 ± 0.01	0.05 ± 0.006

[a] Percentage based on dry wood.

Table 3
Characteristics of untreated wood and CCA-treated wood thermal decomposition in the presence of H_3PO_4

Sample	H_3PO_4 (%)	Initial temperature of mass loss (°C)	Temperature of maximal rate of mass loss (°C)	Mass loss up to 500 °C (%)
Untreated wood	–	250	386	82
	2	200	298	68
	4	170	276	62
	10	150	271	59
CCA-treated wood	–	250	359	78
	2	200	334	74
	4	175	286	64
	10	150	269	57

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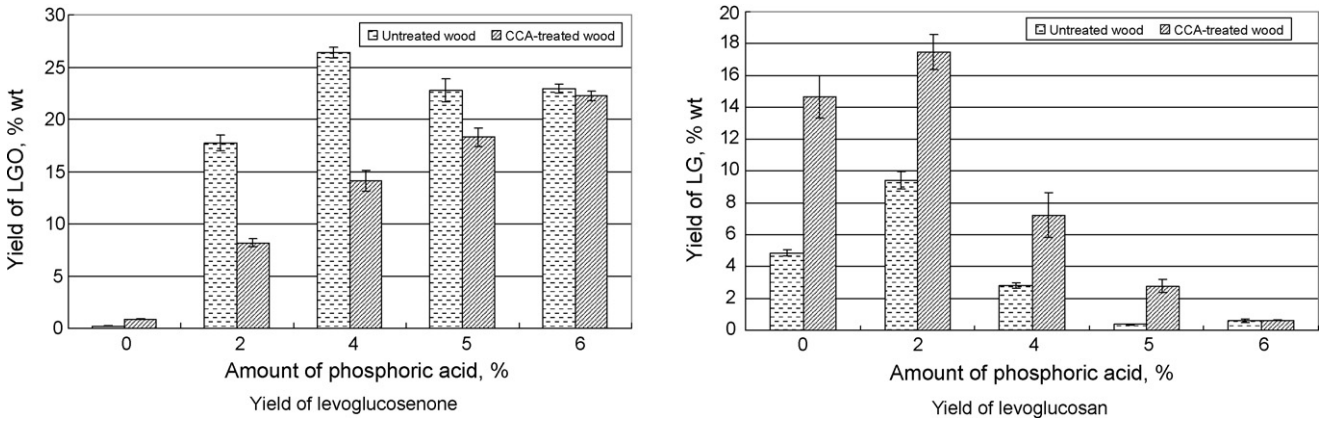

Fig. 2. Yield of some carbohydrate degradation products in pyrolysis tar from untreated wood and CCA-treated wood pretreated with H_3PO_4 (wt.% based on water-free tar).

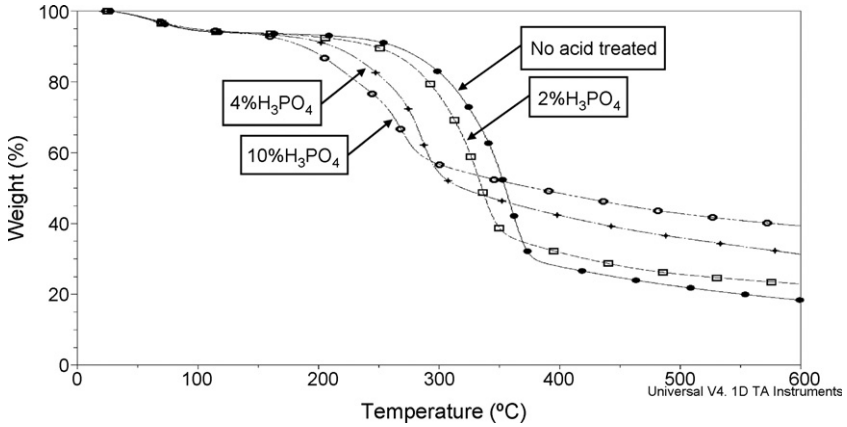

Fig. 3. TGA of H_3PO_4-treated CCA-treated wood in nitrogen at 50 °C/min.

with the addition of phosphoric acid (Table 2). Similar results were found for the lignin degradation products (Table 2). The yield of isoeugenol and guaiacol also decreased and indicates that phosphoric acid pretreatment favors LGO formation and suppresses the production of other chemicals.

3.2. TGA of untreated wood and CCA-treated wood pretreated with H_3PO_4

TGA revealed that the impregnation of wood in phosphoric acid altered the pyrolysis mechanisms. Pyrolysis proceeded mainly through dehydration reactions resulting in the decrease of the amount of volatile products. The onset temperatures and the temperatures corresponding to the maximal weight loss also decreased, which indicated that phosphoric acid accelerated the pyrolysis process (Table 3). As can be seen in Fig. 3, the curves were shifted to lower temperatures when the amount of phosphoric acid was increased. The final char yields were also increased with the addition of phosphoric acid. These results are consistent with the observed effect of phosphoric acid as a flame retardant. It is known that phosphoric acid can improve the fire retardancy of wood by lowering its weight loss rate and increasing its char yield [16].

4. Conclusion

The pretreatment of wood with phosphoric acid significantly alters the pyrolysis kinetics and products distribution leading to lower yields of tar and an increase in char formation. The yield of LG was found to decrease in the presence of relatively higher concentrations of phosphoric acid, while the yield of LGO increased. The pyrolysis of CCA-treated wood in the presence of phosphoric acid gave rise to 22% (on water-free tar) LGO, which demonstrates the potential for a new pathway in the rational utilization of CCA-treated wood waste.

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