



# Thermal behavior of extracted and delignified pine wood flour

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## ABSTRACT

To investigate the effect of extractives and lignin on the thermal stability of wood flour (WF), thermogravimetric analysis was used to determine thermal degradation behavior of extracted and delignified mixed pine WF. The contribution of lignin to thermal stability was greater than that of extractives. Removing extractives resulted in improved thermal stability by increasing onset temperature  $T_0$ , and solvents with greater polarity contributed more to increasing  $T_0$ . However, solvent extraction had less effect on decomposition temperature ( $T_2$ ) than did delignification, which significantly decreased  $T_2$ . A shoulder peak was observed only in delignified WF. Acetone/water extraction, hot-water extraction, and delignification are all significant model terms for variation in  $T_0$ ; delignification is the most significant factor affecting  $T_0$  and  $T_2$ .

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## 1. Introduction

Wood is a natural polymeric composite consisting of cellulose, hemicellulose, lignin, and extractives. Extractives account for only a few percent of the overall composition, but they significantly affect the quality of wood and wood-containing products, such as wood–plastic composites (WPCs) [1,2]. Interest in using wood as a filler in plastics keeps growing because of obvious economic, technical, and ecological benefits [3], primarily high relative strength and stiffness, flexibility during processing, and little mechanical harm to equipment [4,5]. Its use also provides an outlet for waste wood resources, thus decreasing adverse environmental effects [6]. Properties of WPCs depend on many factors, including matrix characteristics, chemical and physical characteristics of the wood filler, overall composition, interaction between wood filler and matrix, and processing conditions.

One issue affecting use of WPCs is their low thermal stability related to thermal degradation at elevated temperatures [7–9]. Thermal degradation of both wood filler and plastic matrix contribute to thermal degradation of WPCs [10,11]. In recent years, several studies on performance and improvement of WPCs have been published [12–19], but none has been found that reports the effects of extractives and lignin on thermal stability of WPCs. A

fundamental understanding of the chemical characteristics of wood filler is essential to improving utilization of lignocellulosic resources in the production of WPCs [20].

In this study, thermal stability of mixed pine wood flour (WF), both control and after treatment using solvent extraction and/or delignification was evaluated by thermogravimetric analysis (TGA). The objective is to promote a fundamental understanding of the effect of extractives and lignin on thermal stability of pine WF for use in manufacturing WPCs.

## 2. Materials and methods

### 2.1. Wood flour

The supplied mixed pine WF (AWF 4020, American Wood Fibers, Schofield, WI) was nominal 40 mesh, contained primarily ponderosa pine (*Pinus ponderosa*), and had a moisture content of 8.0%. After further screening, the WF particles that remained on a 40-mesh screen (0.425 mm) and fell through a 60-mesh screen (0.250 mm) were removed, and the remaining WF was sealed in a polyethylene bag for storage.

### 2.2. Extraction and composition determination

Solvent extractions and delignification were based on a 2<sup>4</sup> full-factorial experimental design (Table 1). Each extraction process was performed for 24 h in a Soxhlet apparatus to ensure removal of

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**Table 1**  
Experimental design.

| WF sample   | Extraction solvents <sup>a</sup> |    |    |                    |
|-------------|----------------------------------|----|----|--------------------|
|             | TE                               | AW | HW | NaClO <sub>2</sub> |
| Control     | 0                                | 0  | 0  | 0                  |
| TE          | 1                                | 0  | 0  | 0                  |
| AW          | 0                                | 1  | 0  | 0                  |
| TE–AW       | 1                                | 1  | 0  | 0                  |
| HW          | 0                                | 0  | 1  | 0                  |
| TE–HW       | 1                                | 0  | 1  | 0                  |
| AW–HW       | 0                                | 1  | 1  | 0                  |
| TE–AW–HW    | 1                                | 1  | 1  | 0                  |
| LR          | 0                                | 0  | 0  | 1                  |
| TE–LR       | 1                                | 0  | 0  | 1                  |
| AW–LR       | 0                                | 1  | 0  | 1                  |
| TE–AW–LR    | 1                                | 1  | 0  | 1                  |
| HW–LR       | 0                                | 0  | 1  | 1                  |
| TE–HW–LR    | 1                                | 0  | 1  | 1                  |
| AW–HW–LR    | 0                                | 1  | 1  | 1                  |
| TE–AW–HW–LR | 1                                | 1  | 1  | 1                  |

<sup>a</sup> 1 and 0 denote solvent used and not used, respectively.

the extractives. Toluene/ethanol (2:1 by volume) (TE), acetone/water (9:1 by volume) (AW), hot water (HW), and various combinations of these solvents were used. Delignified WF (LR) was obtained by the method of Wise et al. [21]. A 100 g sample of air-dried WF was dispersed (under constant stirring) in 1 L of deionized water containing 30 g of NaClO<sub>2</sub> (80%), 10 mL of acetic acid, and 100  $\mu$ L of octan-2-ol (antifoaming agent, Acros Organics), and heated to 70 °C for 1 h. A further aliquot of 30 g of NaClO<sub>2</sub> (80%) and 10 mL of acetic acid was added, and the reaction was continued for another 1 h. Addition of NaClO<sub>2</sub> and acetic acid was repeated three more times for a total reaction time of 5 h before the mixture was allowed to cool to room temperature. The liquid layer was siphoned off, and the delignified WF was rinsed with 1 L aliquots of reverse-osmosis (RO) water until a neutral pH was attained. The wood flour slurry was filtered on a Buchner funnel lined with filter paper. The filter cake was rinsed with RO water for several times to remove Na<sup>+</sup> and Cl<sup>−</sup> thoroughly, followed by 500 mL of 95% ethanol, 500 mL of absolute ethanol and 1 L of acetone. The filter cake was air-dried under suction for 30 min. All WF samples, including the control, were vacuum-dried before further experimentation.

Contents of TE, AW, and HW extractives were determined by the method based on Tappi standard T204 cm-07 [22]. Klason lignin content was measured according to Tappi T222 om-83 [23]. Cellulose content was measured by the method of Allen et al. [24].

### 2.3. Thermogravimetric analysis

Thermogravimetric analysis of WF samples was based on global mass loss measured with a PerkinElmer TGA 7 thermogravimetric analyzer (Shelton, CT). WF samples were evenly and loosely distributed in an open sample pan; initial sample weight was about 5–7 mg. Temperature change was controlled from 25 to 600 °C, with a heating rate of 20 °C/min, in a nitrogen environment to prevent unwanted oxidative decomposition. Onset degradation temperature  $T_0$ , mild weight loss temperature  $T_1$  and rapid weight loss temperature  $T_2$  were evaluated according to the degradation curves. Degradation temperatures in this study were the parameters of primary concern. In the manufacture of WPC, degradation temperatures of the wood fiber are the most important parameters to be considered. Char residues, although they are also important, were not discussed in this study because the temperatures for WPCs manufacturing are not so high as to cause formation of char residues.

### 2.4. Statistics

An attempt was made to model thermal stability using full-factorial statistical analysis. Four treatments (TE, AW, HW, LR) and their combinations were chosen, resulting in a 2<sup>4</sup> factorial design (Table 1). Design Expert 7.0.0 software (Stat-Ease, Minneapolis, MN) was used to design the experiment and analyze data. Derived equations were reported in terms of actual factors. In this design, 0 represents no treatment, 1 represents treatment.

## 3. Results and discussion

### 3.1. Extractives content

Differences in thermal stability among wood species can be attributed to variation in chemical composition [25]. Degradation of wood is closely related to traits of individual wood components. Composition of the mixed pine WF used in this study is presented in Table 2. Extractives contributed only a few percent of the overall composition.

Wood extractives can be divided into three major classes: (1) aliphatic compounds (fatty acids, fatty alcohols, waxes and fats, gums, glycosides), (2) terpenes and terpenoids, (pinene, cadinene, abietic acid), and (3) phenolic structures (phenolic acids, tannins, flavonoids, lignans, stilbenes). Various solvents or combinations of solvents, including ethanol, water, benzene, acetone, chloroform, or a mixture of ethanol/benzene, can remove different types of extractives without damaging the wood structure [22].

Hot water removes the polar extractives (tannins, gums, sugars, starches, coloring substances [26]), acetone the less polar extractives, and toluene the nonpolar extractives (benzaldehyde and numerous terpenic compounds, such as cedrol, agathadiol, epimanol, bornyl acetate,  $\alpha$ -cedrene, and  $\beta$ -cedrene). As shown in Table 2, HW removed the greatest percentage of extractives, AW the least. The lower extraction capabilities of AW and TE are attributed to their lower polarity indexes and their resulting lower abilities to extract the more polar compounds. Removing different types of extractives is expected to affect thermal stability of WF.

### 3.2. Thermal stability of wood flour

Fig. 1 presents thermal degradation curves of WF samples before and after extraction and/or delignification. Mass loss of the WF started at approximately 220 °C and was nearly complete at approximately 500 °C. Thermal degradation curves (Fig. 1) reveal degradation of wood components and suggest three phases of thermal degradation: (1) at 200–300 °C, a small mass loss is primarily due to evaporation of volatile substances and water; (2) at 300–450 °C, a more dramatic mass loss is primarily due to degradation of cellulose, hemicellulose, and lignin; and (3) at greater than 450 °C, mass loss is gradual and attributed to further degradation of the residue.

**Table 2**  
Lignin, cellulose, hemicellulose and TE, AW, and HW extractives in pine WF.

| Items          | Content (%) |
|----------------|-------------|
| TE extractives | 4.69        |
| AW extractives | 4.15        |
| HW extractives | 5.26        |
| Lignin         | 28.86       |
| Cellulose      | 53.12       |
| Hemicellulose  | ND          |

ND denotes not determined.

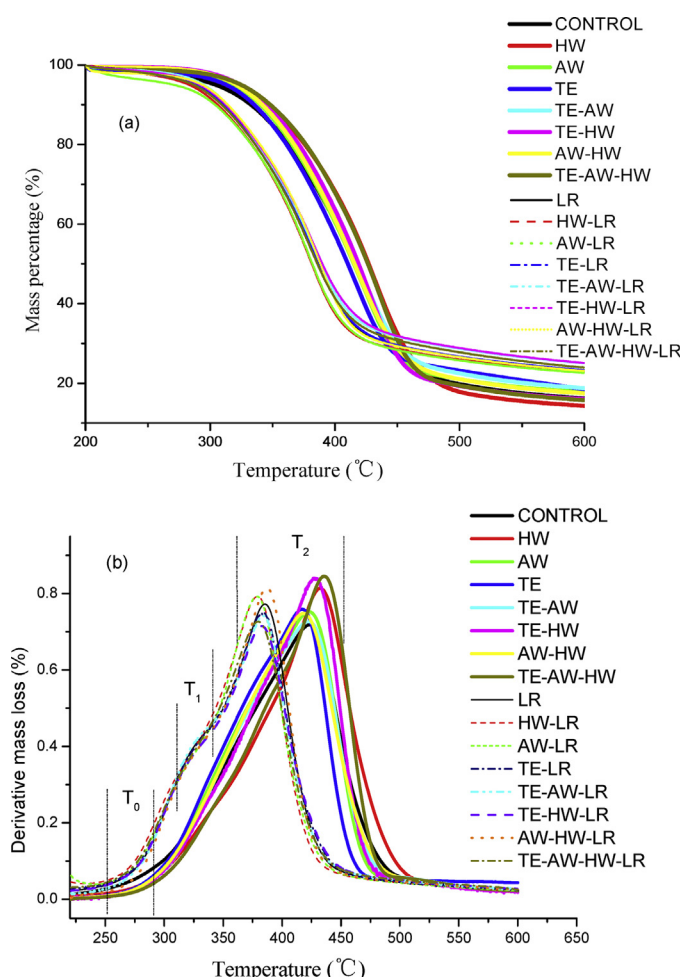


Fig. 1. TGA (a) and DTG (b) curves of extracted and delignified WF samples.

### 3.3. Thermal stability of extracted and delignified WF

Fig. 1 implies that thermal stability of treated WF follows one of two patterns, depending on whether the treatment involved delignification. Extracted WF follows a thermal stability pattern similar to that of control WF, except that control WF showed a small early rise in mass loss compared with extracted WF. This suggests that lignin may contribute more to variation in thermal stability of pine WF than extractives do. Composition of wood affects thermal stability: cellulose is highly crystalline, which contributes to its thermal stability; hemicelluloses and lignin are amorphous and tend to degrade at lower temperature than does cellulose [27]. Hemicelluloses are the least thermally stable wood component, in part due to acetyl groups in its structure [28]. In the DTG spectra, the shoulders around 325 °C are due to degradation of hemicellulose [29]. Lignin degrades over a wide temperature range, starting at relatively low temperatures [30].

Table 3 shows transition temperatures of control, extracted, and/or delignified WF. Onset temperature  $T_0$  is the temperature at which degradation begins (defined as 5% mass loss) and is considered to be the temperature at which hemicelluloses begin to degrade [31].  $T_0$  of WF samples increased after solvent extraction, suggesting increased thermal stability of WF with removal of extractives. The effects of combined extractions were more pronounced than those of individual extractions, suggesting that more complete removal of extractives further enhanced thermal stability. The WF samples subjected to extraction degrade gradually over a wide temperature range [30], from 130 to

Table 3

Thermal degradation temperature of extracted and delignified WF samples.<sup>a</sup>

| Samples     | $T_0$ (°C)   | $T_1$ (°C)   | $T_2$ (°C)   |
|-------------|--------------|--------------|--------------|
| Control     | 263.3 (±0.7) | –            | 424.6 (±2.4) |
| TE          | 280.1 (±1.5) | –            | 416.2 (±2.1) |
| AW          | 275.6 (±1.2) | –            | 422.3 (±1.8) |
| TE-AW       | 290.3 (±0.9) | –            | 422.4 (±1.1) |
| HW          | 276.0 (±2.6) | –            | 434.1 (±1.7) |
| TE-HW       | 290.1 (±1.3) | –            | 428.3 (±2.9) |
| AW-HW       | 290.7 (±1.0) | –            | 424.3 (±3.2) |
| TE-AW-HW    | 294.3 (±0.7) | –            | 436.6 (±1.7) |
| LR          | 244.2 (±0.7) | 325.2 (±1.3) | 385.2 (±2.1) |
| TE-LR       | 244.2 (±0.8) | 331.3 (±1.3) | 383.1 (±1.4) |
| AW-LR       | 258.1 (±1.6) | 316.7 (±1.5) | 378.3 (±1.7) |
| TE-AW-LR    | 244.5 (±0.8) | 323.3 (±1.1) | 383.7 (±1.7) |
| HW-LR       | 260.3 (±0.8) | 312.2 (±0.8) | 378.3 (±2.3) |
| TE-HW-LR    | 244.7 (±0.7) | 325.6 (±0.8) | 382.8 (±1.6) |
| AW-HW-LR    | 261.3 (±1.1) | 323.3 (±0.6) | 387.1 (±1.9) |
| TE-AW-HW-LR | 261.3 (±1.1) | 314.2 (±0.7) | 380.3 (±2.2) |

<sup>a</sup> Average (±standard deviation) of five replicates.

550 °C, overlapping the temperature range for thermal decomposition of hemicelluloses.  $T_0$  is important to WPC manufacturing because it limits processing temperatures, which can influence the mixing efficiency of WF and plastic matrix. Extractives can also significantly affect performance of WPCs with respect to wettability, Young's modulus, density, durability, shrinkage, and permeability [11].

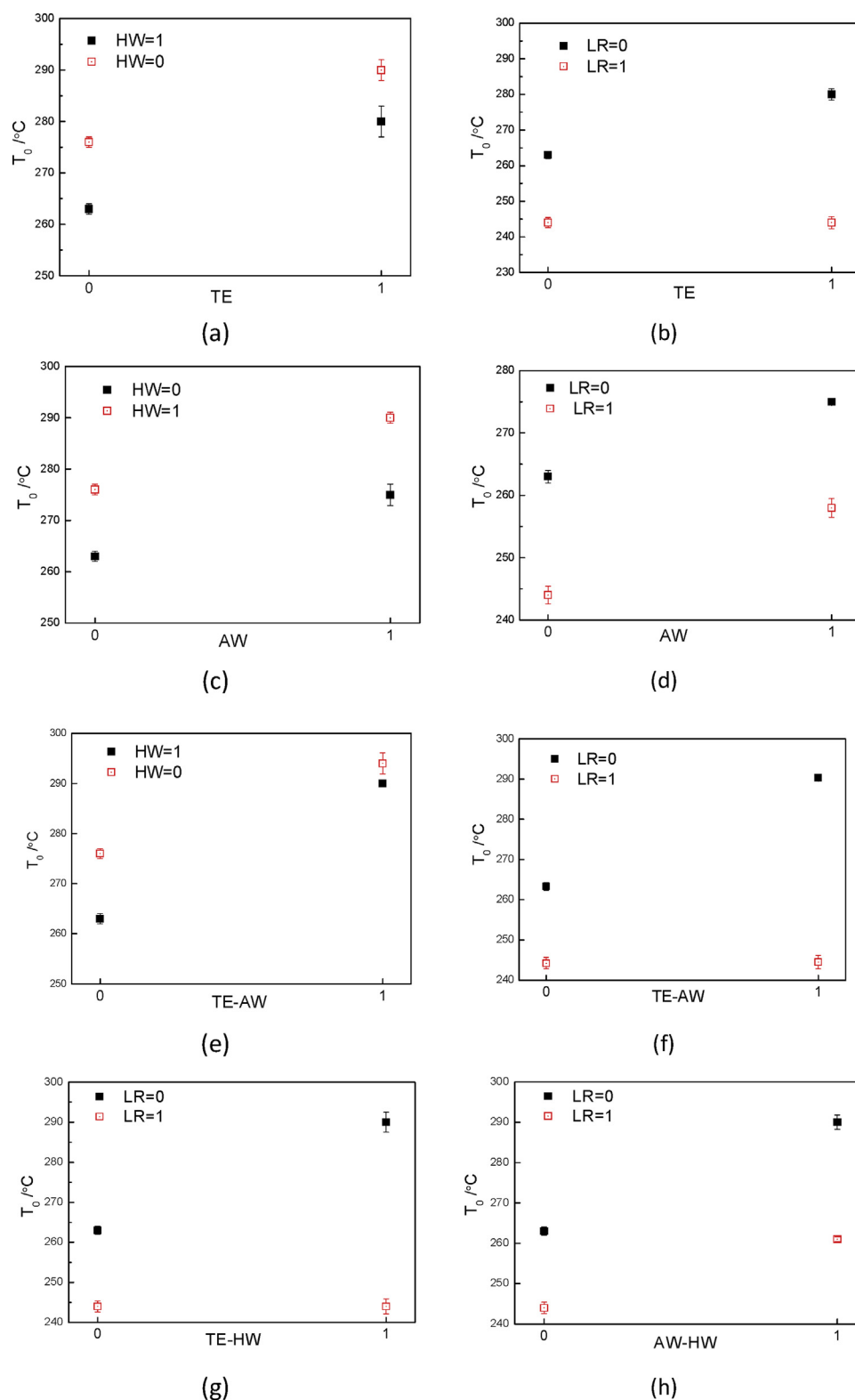
$T_1$  and  $T_2$  are decomposition temperatures, seen as shoulders and peaks, respectively, in Fig. 1b. No obvious variations in  $T_2$  were evident after solvent extractions. The greatest value of  $T_2$ , reflecting slightly improved thermal stability, was obtained after extraction with HW, which has the highest polarity among the solvents used in the study. Differences in  $T_2$  can be attributed to variation in cellulose conversion, which might be modified when the wood flour was subjected to different solvents extraction or delignification, a relationship that helps in understanding the effect of extractives removal on thermal properties of WPCs.

Fig. 1 and Table 3 show that decomposition temperatures, both  $T_0$  and  $T_2$ , of delignified WF decreased compared with the control. Decrease in thermal stability of delignified WF varied with the various extraction steps involved and might be due to the exposure of hemicelluloses following the removal of lignin. Hemicelluloses are the least thermally stable wood components and decompose quickly [26,27]. Consequently, the shoulder peaks ( $T_1$ ) of delignified WF samples were attributed to hemicelluloses.

Fig. 2a and b shows interactions between TE-HW and TE-LR, respectively, and Fig. 2a clearly shows that extraction with TE followed by extraction with HW increases  $T_0$  significantly beyond the increase observed with TE or HW extraction alone. Extraction with TE followed by delignification resulted in a decrease in  $T_0$  (Fig. 2b). A similar trend occurred for AW-HW and AW-LR extractions (Fig. 2c and d, respectively). In these interaction graphs,  $T_0$  increases with each single solvent extraction, with the combination of two solvents increasing  $T_0$  by a value close to the sum of the two individuals values (Fig. 2a and c). Extraction with TE-AW followed by HW resulted in an increase in  $T_0$  (Fig. 2e). Extraction by a combination of solvents followed by delignification resulted in an overall decrease in  $T_0$  (Fig. 2f–h).

### 3.4. Analysis of variance (ANOVA)

The analysis of variance was performed to find the statistical significance of the variables and their interactions on the  $T_0$  and  $T_2$ . To compare the effect of extraction and delignification on thermal stability of WF samples, an orthogonal test was employed. The Design Expert software was used in the ANOVA, and the results of



**Fig. 2.** Variation in  $T_0$  as a function of two-factor interactions between (a) TE-HW, (b) TE-LR, (c) AW-HW, (d) AW-LR, (e) TE-AW-HW, (f) TE-AW-LR, (g) TE-HW-LR, and (h) AW-HW-LR (1 and 0 denote solvents used and not used, respectively).

the sum of square due to each source, the degrees of freedom associated with each source (df), the mean squares of each source, the F-statistic and  $p$ -value are presented in Table 4. Large differences correspond to large values of the F-statistic and small  $p$ -value. The models for  $T_0$  and  $T_2$  are both significant according to

$p$ -value and  $F$ -value in Table 4. Table 4 shows that AW, HW, and LR are all significant terms to  $T_0$ . Delignification (LR), with  $p < 10^{-4}$ , is the most significant factor among the overall treatment methods. HW extraction, with the lowest  $p$ -value among all extraction methods, is the second most significant factor, perhaps related to

**Table 4**  
Analysis of variance table for  $T_0$  and  $T_2$ .

| $T_0$     |                |    |             |         |                     |
|-----------|----------------|----|-------------|---------|---------------------|
| Source    | Sum of squares | df | Mean square | F-value | p-value<br>prob > F |
| Model     | 4389.50        | 4  | 1097.3750   | 21.0207 | <0.0001             |
| TE        | 25.00          | 1  | 25.0000     | 0.4789  | 0.5033              |
| AW        | 324.00         | 1  | 324.0000    | 6.2064  | 0.0300              |
| HW        | 380.25         | 1  | 380.2500    | 7.2838  | 0.0207              |
| LR        | 3660.25        | 1  | 3660.2500   | 70.1136 | <0.0001             |
| Residual  | 574.25         | 11 | 52.2045     |         |                     |
| Cor total | 4963.75        | 15 |             |         |                     |

| $T_2$     |                |    |             |          |                     |
|-----------|----------------|----|-------------|----------|---------------------|
| Source    | Sum of squares | df | Mean square | F-value  | p-value<br>prob > F |
| Model     | 7737.75        | 4  | 1934.4375   | 71.4054  | <0.0001             |
| TE        | 0.25           | 1  | 0.2500      | 0.0092   | 0.9252              |
| AW        | 0.25           | 1  | 0.2500      | 0.0092   | 0.9252              |
| HW        | 81.00          | 1  | 81.0000     | 2.9899   | 0.1117              |
| LR        | 7656.25        | 1  | 7656.2500   | 282.6133 | <0.0001             |
| Residual  | 298.00         | 11 | 27.0909     |          |                     |
| Cor total | 8035.75        | 15 |             |          |                     |

the relatively complete removal of the highly polar components. However, LR is the only term with significant effects on  $T_2$ .

#### 4. Conclusion

Thermal degradation temperature of pine WF is in the range of 220–500 °C. After various extractions and/or delignification, thermal stability of WF follows one of two patterns, depending on whether the WF was solvent-extracted or delignified. Delignification resulted in the largest decrease in thermal stability, whereas solvent extraction gave mixed results; some solvent systems (such as TE–AW–HW) led to improved thermal stability, and others had little effect compared with the control WF.  $T_0$ , an important parameter in WPCs manufacturing, increased after solvent extraction; the more polar solvents contributed more to the increase in  $T_0$  than did the less polar ones.  $T_2$  changed slightly after solvent extraction but changed significantly after delignification. Decomposition temperature of WF samples decreased after delignification. A shoulder peak ( $T_1$ ) was seen only in delignified WF samples and was attributed to decomposition of hemicellulose.  $T_2$  shifted to a lower temperature after delignification. Data from the orthogonal analysis shows that AW, HW, and LR all have a significant effect on  $T_0$ . Delignification (LR) has the most significant effect on  $T_0$  and  $T_2$ .

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