

# Performance of Cutting and Grinding Wheel Based on Lignin-Phenolic Resin Matrix and Aluminum Oxide

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Grinding wheels made from an easily-prepared and industrialized thermosetting PFL resin (phenol, formaldehyde, and alkali lignin) with aluminum oxide particles were prepared (*i.e.*, PFL grinding wheel). The mechanical properties of these grinding wheels were characterized by their Brinell hardness, compression strength, and abrasiveness. The curing and heat resistance properties of the PFL resin were studied using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The results indicated that the new PFL resin with 30% of the phenol replaced by alkali lignin exhibited excellent heat resistance. When using alkali lignin to replace a portion of the phenol, the curing temperature of phenol-formaldehyde resin (PF) was increased. Scanning electron microscopy (SEM) of the PFL grinding wheel showed no pores and cracks in the composite when compared to laboratory prepared PF grinding wheels; PFL grinding wheels had high hardness and compression resistance. Furthermore, the PFL grinding wheel exhibited abrasiveness that was comparable to the PF grinding wheel during laboratory tests.

*Keywords:* Alkali lignin; Phenol-formaldehyde resin; Grinding wheel; Aluminum oxide particles

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## INTRODUCTION

The angle grinder is one of the most widely used cutting tools in the construction and fabrication fields. An important component of the angle grinder is the grinding wheel; this wheel typically is mainly composed of phenol-formaldehyde (PF) resin composite (Robie 1957; Rowse and Stinchfield 1959; Lin *et al.* 2015), carbon/carbon composite materials (Nasibulin 2017), or metal composite grinding materials (Zhang *et al.* 2017). However, carbon/carbon composite materials are expensive and have poor oxidation resistance. Metal composite materials are heavy and exhibit poor mechanical properties at high temperatures. PF resin composite materials are low cost and lightweight; however, these composites utilize toxic substances for the matrix and the resin requires a long time to cure fully. As a result of these limitations, research has been carried out to identify low-cost, lightweight, and low-toxicity grinding wheels. Fortunately, a large number of approaches have examined bioresources, such as tannin and lignin, for some low-cost resin components. Presently, tannin-based adhesive (Zhang *et al.* 2017) and grinding wheels (Lagel *et al.* 2015; Zhang *et al.* 2015) have been developed. In addition, the structure of lignin has some similarities to phenol (Hemmilä *et al.* 2017). Presently, lignin has been developed to replace phenol to react with formaldehyde for some biobased materials, such

as lignin-phenol-formaldehyde (PFL) wood adhesives (Yang *et al.* 2015; Zhao *et al.* 2016) and PFL foams (Qu *et al.* 2017). During the chemical pulping of lignocellulosic materials, the generated kraft and alkali lignin is burned as a biofuel to generate steam and to recover spent inorganic chemicals (Xue *et al.* 2014). Thus, the PFL-based resin for grinding wheel could be made using the dissolved alkali lignin obtained from chemical pulping.

In this paper, the possibility of preparing PFL resins and using them for making grinding wheel is examined. The PFL resin explored are based on the co-reactions of alkali lignin, phenol and formaldehyde under alkaline conditions. These PFL resins are combined with aluminum oxide particles to make new grinding wheels. The PFL-based grinding wheels are examined concerning their durability and abrasiveness, and such properties are compared to PF-only grinding wheels.

## EXPERIMENTAL

### Materials

Alkali lignin, which was obtained from soda (NaOH) pulping of lignocellulosic materials in the mild alkaline extraction of wheat straw residues, was obtained from the Long Live Biological Technology Co. (Shangdong, China). Furfuryl alcohol (98% purity) and lactic acid were purchased from Acros Organics (Geel, Belgium). Liquefied phenol (80% purity) and NaOH were purchased from Fisher Scientific (Loughborough, UK). Formaldehyde (37% aqueous solution) was purchased from Acros Organics (Geel, Belgium). Aluminum oxide particles, with a size of 0.4  $\mu\text{m}$ , and steel tube (diameter of 7 mm, inner diameter of 6 mm, and length of 0.8 m) were obtained from the Run Xin Materials Co. (Kunming, China). Commercial PF-based resin grinding wheel (“PFG”; diameter of 100 mm, center hole of 22.2 mm and thickness of 6.4 mm) was supplied by the Bosh Co. (Stuttgart, Germany).

### Preparation of PFL and PF Resins

The PFL or PF resins were synthesized at 94 °C using 1 molar of phenol (80% purity) mixed with 2.2 molar of formaldehyde (37% aqueous solution). For PFL resins, alkali lignin was used to replace part of the phenol at the substitution ratios of 30%, 40%, and 50%. Formaldehyde was divided into four equal parts, and each part was added at 15-minute intervals. Initially, the first portion of formaldehyde was mixed with the phenol (or phenol with alkali lignin) at 94°C; the mixture was refluxed with continuous stirring. Afterwards, the second, third, and fourth parts of formaldehyde were added, and the mixture was continuously stirred. Throughout the process, the mixture pH was maintained at 10 by adding drops of concentrated NaOH solution (40% aqueous), and the mixture temperature was controlled at 94 °C.

### Preparation of Grinding Wheels

Initially, the PFL or PF resins were mechanically stirred with or without lactic acid in a beaker for 20 s. Next, the aluminum oxide particles ( $\text{Al}_2\text{O}_3$ ) were mixed with these resins. The mass ratio of  $\text{Al}_2\text{O}_3$ -to-PFL that was used was 1.5:1 and 2:1, which was based on the previous work by Lagel *et al.* (2015). Afterwards, the mixtures were put in a mold (100 mm of diameter) with three layers of fiber glass. The molds were placed in a press for 1.5 h at 150 °C under a pressure of 7.8 MPa for curing. After curing, the grinding wheels were cooled and 22 mm diameter holes were drilled at their centers. The various

compositions for the grinding wheels tested are shown in Table 1.

**Table 1.** Composition of Different Grinding Wheels Tested in This Study

Sample	Alkali lignin (g)	Phenol (mL)	Substituted ratio of phenol with alkali lignin (%)	Formaldehyde (mL)	Mass ratio of Al <sub>2</sub> O <sub>3</sub> :resin	Lactic acid (mL)
S1	0	20	0	38	1.5:1	0
S2	6	14	30	38	1.5:1	0
S3	8	12	40	38	1.5:1	0
S4	10	10	50	38	1.5:1	0
S5	6	14	30	38	1.5:1	5
S6	6	14	30	38	2:1	5

In order to be compared with the commercial PF grinding wheel, Sample S7 was prepared and the composition amount of S7 was the same with the sample of S5, But compared with sample of S5, S7 was prepared at an higher press temperature (250 °C) and pressure (12 MPa) according to the preparation process of commercial PF grinding wheel (Robie 1957; Rowse and Stinchfield 1959).

The solids levels of the S1 to S4 bonded resins were determined by measuring the weight of the resins before and after drying at 120±1 °C for 2 h in an oven. The viscosities of the various resins were measured using a tu-4 viscosity cup at 25 °C, and the resin curing time was determined in accordance to Chinese National Standard GB/T14074 (2006). The characteristics of different resins are shown in Table 2.

**Table 2.** Characterization of Laboratory PF and PFL Resins

Adhesive Type	Colors	Solid content (%)	Viscosity (s)	Curing time (s)
PF-S1	pink	70.2	75	134
PFL-S2	brown	72.5	72	168
PFL-S3	black-brown	77.3	83	205
PFL-S4	black	82.4	96	258

### Scanning Electron Microscopy (SEM)

A scanning electron microscope (model S 4800, Hitachi, Tokyo, Japan) was used to observed these surfaces of PF and PFL resins based grinding wheels, which were cut into 5 mm × 5 mm × 4 mm, at 100x magnification.

### Electrospray Ionization Mass Spectrometry (ESI-MS)

The spectra were recorded in a negative ion mode (ion energy of 0.3 eV and scan range of 0 to 1000 Da) using a Waters Xevo Triple Quadrupole-MS (Waters, Milford, MA, USA) with an electrospray ionization source (ESI). First, the sample of LPF resin was dissolved in chloroform at a concentration of about 10 µL/mL. After that, the mixture was injected into the ESI source, in combination with an ion trap mass spectrometer (Bruker Daltonics Inc., Billerica, MA, USA) *via* a syringe at a 5 µg/s flow rate.

## Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA)

The curing behaviors of resins were studied using a DSC analyser (model DSC 204 F1, Netzsch, Germany) with a heating rate of 15 °C/min under a nitrogen atmosphere. The temperature range was 30 °C to 250 °C. The thermal properties of resins were evaluated using a thermogravimetric analyzer (TGA) (model TG 209 F3, Netzsch, Germany). The tests were carried out with temperatures ranging from 30 °C to 900 °C using a rate of 20 °C/min under a nitrogen atmosphere.

## Compression and Brinell Hardness Measurements

The compression test was implemented by the strength analysis instrument (Instron model 1193, Norwood, MA, USA) in accordance to ASTM D695-10 (2010) with the rate of loading of 2 mm/min. Each result was the average value of five specimens.

The Brinell hardness measurement following the GB/T 231.1-2009 (2009) standard was conducted using the strength analysis instrument (Instron model 1193, Boston, USA) with a 10 mm steel ball diameter and 2452 N loading. The rate of loading was 2 mm/min. The Brinell hardness number,  $H$  (in daN/mm<sup>2</sup>), was calculated using Eq. 1,

$$H = \frac{3.18}{1000} \times \frac{F}{P} \quad (1)$$

where  $F$  is the force that determined the retained depression (in N) and  $P$  is the depth of depression of the ball (in mm). Each result was the average value of five specimens.

## Abrasiveness Measurement

The abrasiveness of the grinding wheels was determined by using an angle grinder (Bosch GWS 1400) with a rotational speed of 11,000 rpm. For all of the grinding wheels, the steel tube was cut off with a grinding wheel along the vertical direction of the tube length. The size of steel tubes for different grinding wheels was the same (diameter of 7 mm, inner diameter of 6 mm, and length of 0.8 m). In the process of cutting, the cutting time and mass loss of grinding wheels were measured. In addition, the commercial grinding wheels (PFG) were compared with alkali lignin based grinding wheels. The percentage of mass loss from the grinding wheels was calculated by weighting the grinding wheels before and after the abrasiveness test. The mass loss was calculated using Eq. 2,

$$m = \frac{m_1 - m_2}{m_1} \times 100\% \quad (2)$$

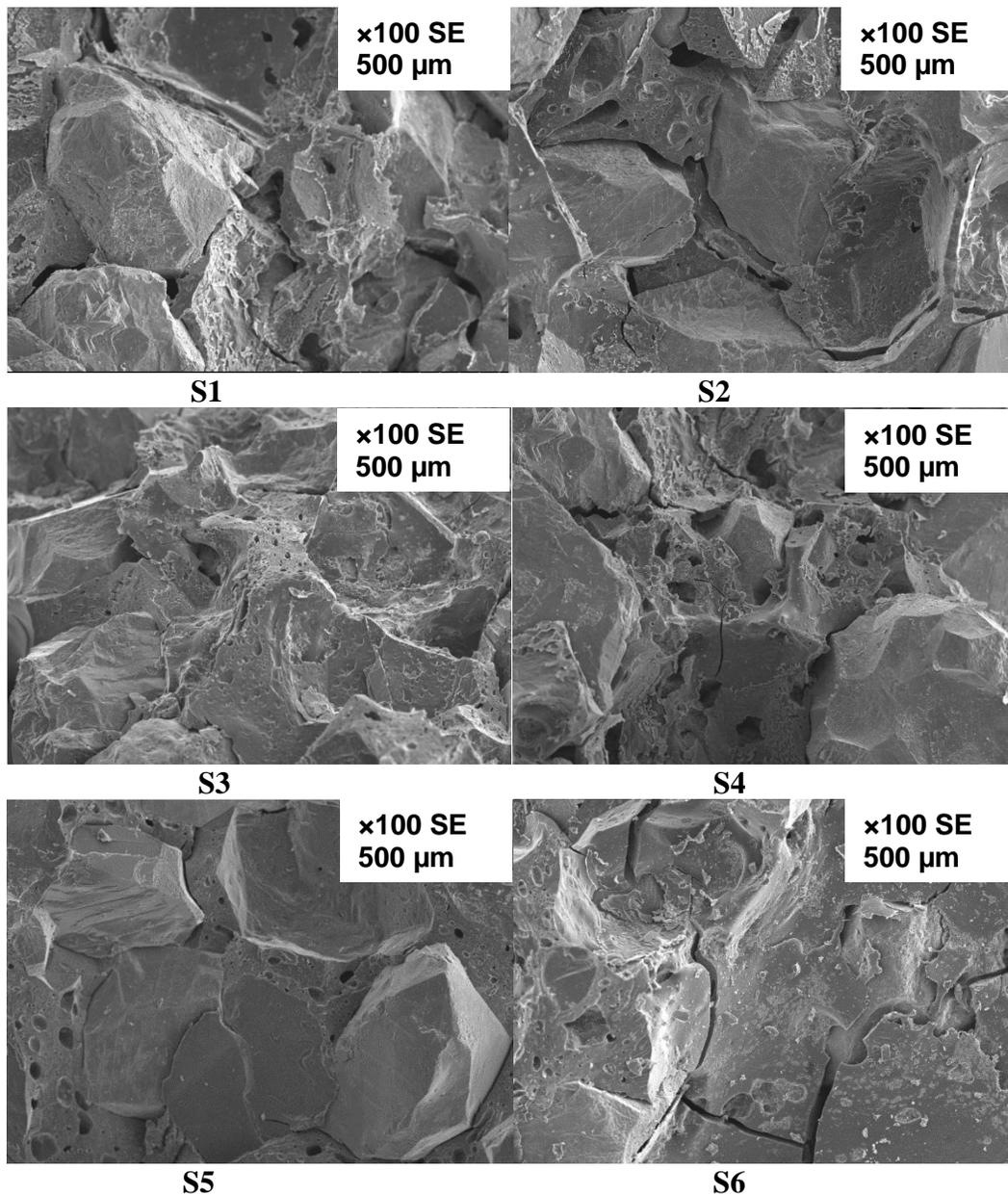
where  $m$  is the mass loss (in %),  $m_1$  is the mass of the grinding wheel before steel cutting (in g), and  $m_2$  is the mass of the grinding wheel after steel cutting (in g). Each result was the average value of four specimens.

## RESULTS AND DISCUSSION

### Macroscopic Observations

SEM micrographs of the PF and PFL grinding wheels at the macroscopic level are shown in Fig. 1. All sample surfaces exhibited some pore spaces between the inorganic particles and the polymeric resin matrix. These results indicated that some water remained in PF resin after the curing process. Thus, these pores are the consequence of the water

present in the resin. In addition, for samples S3 and S4, it was apparent that some pores were present. Compared with them, the sample of S2 showed less pores. The S1, S2, S3, and S4 samples presented rough surfaces. In contrast, samples S5 and S6, which had lactic acid added, exhibited smooth surfaces. There were obviously large cracks observed with the S6 sample surface. These observations indicated that the addition of lactic acid could decrease the brittleness of the PFL resin. After the resin curing process, the surfaces of S5 and S6 were smoother than the other samples prepared without lactic acid addition. Sample S6 had a mass ratio of  $\text{Al}_2\text{O}_3/\text{PFL}$  that was 2-to-1, which was higher than that of sample S5; the higher mass ratio of S6 resulted in the  $\text{Al}_2\text{O}_3$  particles that were not as well bonded within the resin matrix as with S5. Hence, it is clear that compared with other grinding wheels, sample S5 exhibited a better surface appearance.



**Fig. 1.** The SEM photos of PF and PFL grind wheels with aluminum oxide particles

### Brinell Hardness and Compression

Test results from the Brinell hardness measurements of PF and PFL grinding wheels are shown in Table 3. The results showed that PFL samples that contained different levels of alkali lignin were harder than the sample S1, which was prepared with PF resin only. The structure of alkali lignin is more complex than phenol. High carbon content yielded higher resin hardness with the PFL-based samples. The differences in hardness among samples S2, S3 and S4 were attributed to the amount of alkali lignin incorporated into the PF resin. The reaction between alkali lignin and formaldehyde occurred at the unsubstituted aromatic positions and at other reactive sites (Qu *et al.* 2017). It was clear that sample S2 was harder than S3 and S4. Moreover, sample S2 was harder than sample S5, but not as hard as sample S6. These results can be explained by the high amount of aluminum oxide particles, which increased the hardness of S6 versus S2. Meanwhile, under the same PFL resin amount, the sample of S5 prepared with lactic acid was more flexible compared with sample of S2, but on the contrary, the hardness of S5 can be decreased with the addition of lactic acid.

Table 3 also shows the compression strength of all grinding wheel samples studied. Sample S5 had the highest compression strength of all the laboratory prepared LPF-based samples that were prepared with the same temperature and pressure. Moreover, it had higher compression strength than sample S1, which was the laboratory PF only resin (116.52 MPa). Furthermore, from the same LPF-based grinding wheels of S2 and S5 it was observed that the addition of lactic acid made the resin matrix more flexible, which improves the tool's compressive resistance. Meanwhile, the hardness ( $7.88 \pm 0.038$  daN/mm<sup>2</sup>) and compressive strength ( $139.78 \pm 1.55$ ) values of commercial PFG were higher than that of S1, S2, S3, S4, S5, and S6. Indeed, some sources (Robie 1957; Rowse and Stinchfield 1959) report that PFG samples were pressed at high pressure (10 to 35 MPa) and temperature ( $\geq 200$  °C). The PFL-based resin grinding wheels of this study, on the other hand, were pressed at 7.8 MPa and 150 °C. However, the value of hardness and compression strength of PFG were lower than that of sample S7. This indicated that the addition of lignin in PF resin can increase hardness and improve compression properties of a PF grinding wheel.

**Table 3.** Brinell Hardness Number and Compression Strength of PF and PFL Grinding Wheels

	S1 (PF)	S2 (PFL)	S3 (PFL)	S4 (PFL)	S5 (PFL)	S6 (PFL)	S7 (PFL)	PFG (PF)
Brinell Hardness No. (daN/mm <sup>2</sup> )	6.16 $\pm 0.032$	6.74 $\pm 0.049$	6.38 $\pm 0.027$	6.21 $\pm 0.028$	6.32 $\pm 0.021$	7.05 $\pm 0.033$	8.23 $\pm 0.029$	7.88 $\pm 0.038$
Compressive strength (MPa)	116.52 $\pm 1.52$	119.43 $\pm 1.62$	117.43 $\pm 1.26$	111.52 $\pm 1.31$	128.35 $\pm 1.33$	92.43 $\pm 1.74$	151.11 $\pm 1.93$	139.78 $\pm 1.55$

Note:  $\pm$  indicates the standard deviation

### Abrasiveness of Grinding Wheels

The results shown in Table 4 indicated that sample S5 prepared with PFL resin exhibited better wear resistance when compared to other PFL samples prepared in the laboratory using the same press temperature and pressure. Sample S5 had lower mass loss than sample S2 at the same phenol substitution level (*i.e.*, 30% phenol replaced with alkali lignin). When compared to sample S5, sample S6 had a higher mass loss over a shorter period of cutting time. Increasing the Al<sub>2</sub>O<sub>3</sub>/PFL mass ratio to 2:1 improved the wearability

of S6, which had a shorter cutting time (3 s). However, the SEM micrographs of sample S6 showed cracks, which resulted in high mass loss of this grinding wheel. Furthermore, under the same press temperature and pressure, these samples prepared with PFL resins (S2 to S6) exhibited higher mass losses when compared to the laboratory PF resin-based grinding wheel (S1). Apparently, the structure of the alkali lignin was more complex than that of phenol. The mass loss of S5 (6.5%) was close to that of S1 (6.3%). Consequently, sample S5 had good abrasiveness and was competitive with the PF resin-based grinding wheel. The cutting time of S5 was the same as that of the commercial PF grinding wheel (PFG). However, the mass loss of the PFG was twenty to eighty times lower than that of sample S5. Meanwhile, sample of S7 showed a better cutting property compared with sample S1 to S6. This may have been due to the different conditions used for the resin curing process. For the commercial PFG and S7, the resin curing process was conducted at a higher press temperature and pressure. Moreover, the mass loss of S7 was higher than that of PFG. The result indicated that some resin binders were mixed with PF resin of the commercial PFG to improve the matrix's bonding strength with the inorganic particles.

**Table 4.** Cutting Properties of Different Grinding Wheels

	S1 (PF)	S2 (PFL)	S3 (PFL)	S4 (PFL)	S5 (PFL)	S6 (PFL)	S7 (PFL)	PFG (PF)
Mass loss (%)	6.3±0.1	7.2±0.1	9.6±0.1	11.5±0.1	6.5±0.1	23.7±0.1	0.6±0.1	0.3±0.1
Cutting time (s)	4	4	4	5	4	3	4	4

Note: ± indicates the standard deviation

### ESI-MS Analysis

To study the reaction between lignin, phenol, and formaldehyde, the ESI-MS spectra of alkali lignin and PFL samples (S5) based resin was examined (Figs. 2 and 3). Phenol and lignin contain many phenolic hydroxyl groups, and these are easy to ionize. Such structures were easily detected in the negative ion mode. The structure of free phenol (93 Da) and dihydroxy phenol (153 Da) shown in Fig. 3 indicated that some phenol and dihydroxy phenol were not involved in the LPF reaction system. Meanwhile, from Fig. 3 one can see a peak at 271, which was attributed to the condensation of two molecules of hydroxymethyl phenol and 377 Da corresponded to the polycondensation structure of three hydroxymethyl phenol molecules. These peaks were attributed to the self-polycondensation of phenol. The 195 Da peak (shown in Fig. 2 and 3) was attributed to the structure of guaiacyl of lignin. Moreover, the peak of 301 Da (Fig. 3) may be the condensation structure between guaiacyl unit and hydroxymethyl phenol, which was shown in Eq. 3. Meanwhile, the 319 Da peak (the different value range with 301 Das was 18) shown in Fig. 3 was attributed to the complex ion peak of phenol and guaiacyl unit. Moreover, 407 Da (the different value range with 301 Das was 106) was attributed to the condensation structure of the two molecules, hydroxymethyl phenol and the guaiacyl unit. Hence, the reaction of lignin, phenol, and formaldehyde under alkaline condition was established.

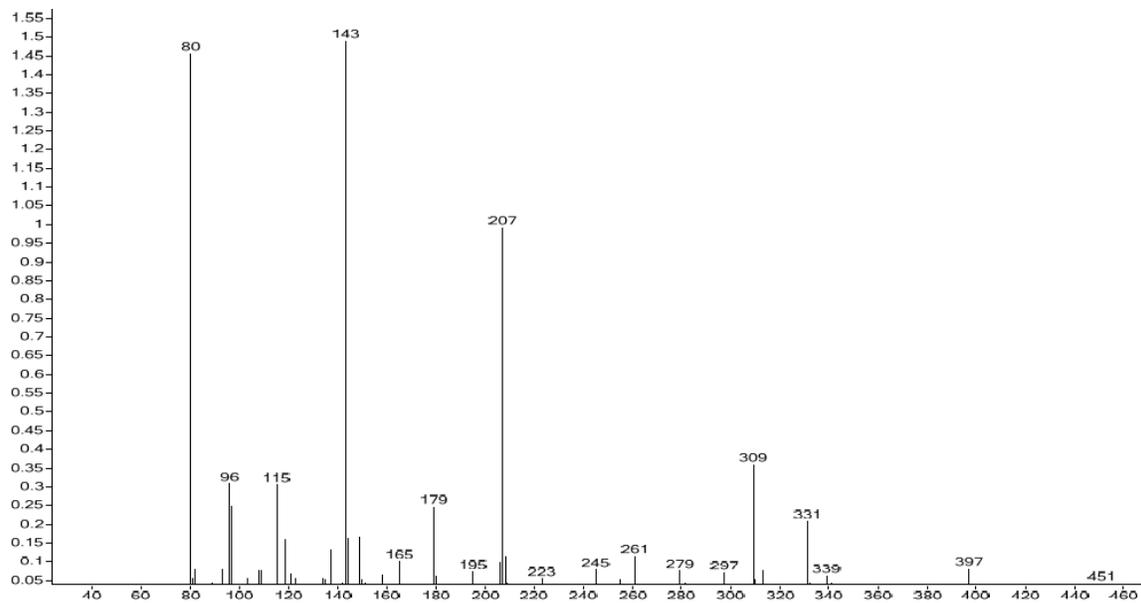


Fig. 2. ESI-MS spectra of alkali lignin

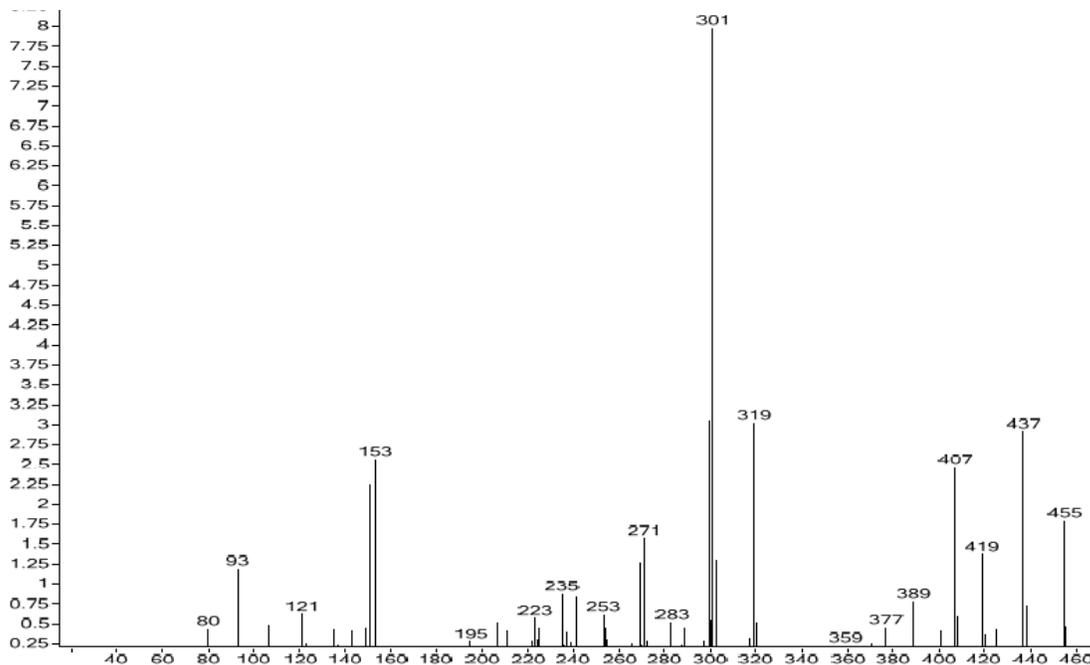
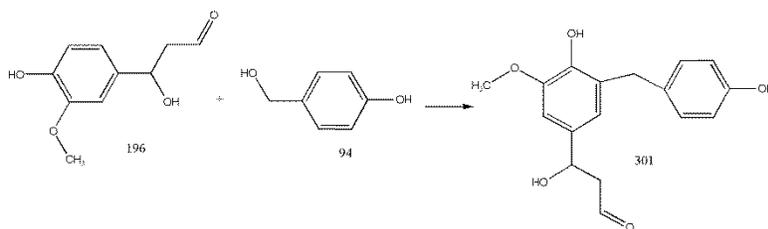


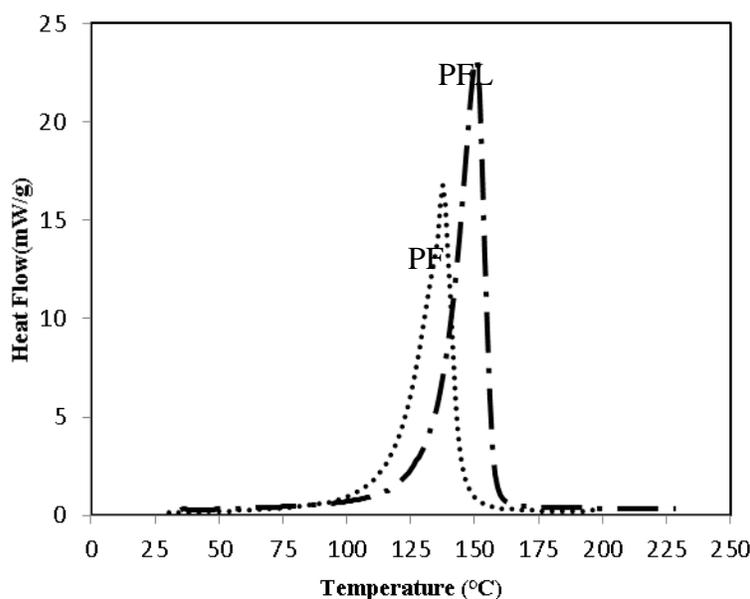
Fig. 3. ESI-MS spectra of LPF resin



### DSC and TG Analysis

To study the curing properties and heat resistances of PFL resins, one of the PFL samples (S5) was examined and compared to the laboratory prepared PF resin (S1).

Typical DSC curves for PF and PFL resins are shown in Fig. 4. Each curve exhibited one exothermic peak during resin curing. From these curves, it was observed that the PF curing temperature (130 °C) was lower than PFL (150 °C). Moreover, the PFL curve indicated that the co-reactions between phenol, formaldehyde, and alkali lignin had occurred and that they were exothermic reactions. The two sharp exothermic peaks indicated that the polymerization for PF reached its maximum conversion rate at a lower temperature than that for PFL. The addition of alkali lignin to the PF mixture under alkaline conditions increased the curing temperature of the resin compared to PF alone.



**Fig. 4.** DSC thermograms of the curing process of PF and PFL resins

The TG curves of cured PFL and PF resins are shown in Fig. 5. These curves were obtained at heating at a rate of 10 °C/min over the temperature range of 35 °C to 900 °C under a nitrogen atmosphere. The PFL with the substitution of 30% phenol by alkali lignin had higher heat resistance when compared to PF resin. PFL reached its 5% weight loss at 253 °C, which was higher temperature than that of PF resin, which reached its 5% weight loss at 83 °C. Furthermore, the PFL resin still retained 80% of its original mass at 500 °C; the weight loss of PFL and PF was 20% and 30%, respectively. In addition, the carbon yield of PFL at 900 °C reached as high as 56%. These observations indicated that replacing 30% of the phenol in PF with alkali lignin improve the heat resistance of the resulting resin. These results can be explained based on the fact that the alkali lignin has higher carbon content when compared to phenol; the complex structure of alkali lignin contributed to the high heat resistance of the PFL resin.

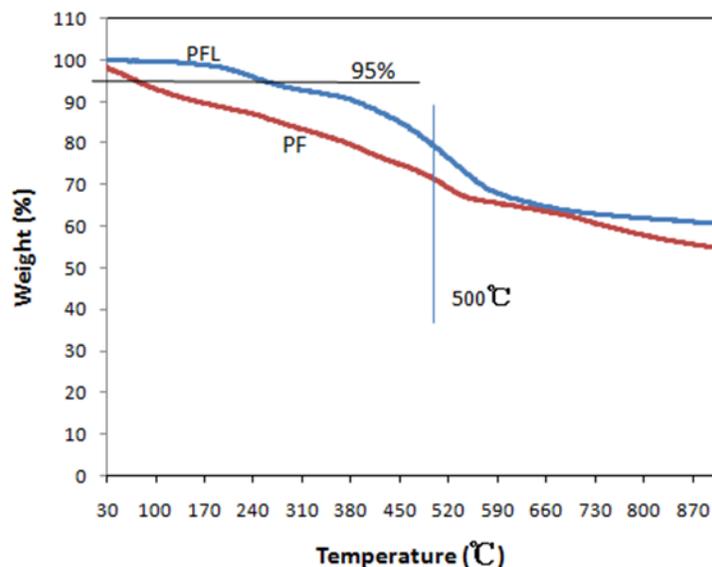


Fig. 5. TGA thermograms of the cured PF and PFL resins from 30 °C to 900 °C

## CONCLUSIONS

1. A phenol-formaldehyde-lignin (PFL) resin-based grinding wheel was prepared. The PFL resin (with 30% of the phenol replaced with alkali lignin) was made from 14 mL of phenol, 26 g of alkali lignin, 38 mL formaldehyde (37% aqueous solution), and 5 mL of lactic acid, which was reacted under alkaline conditions; aluminum oxide particles were added to this resin at 1.5:1  $\text{Al}_2\text{O}_3$ /resin ratio. Meanwhile, the mixture was cured in a heat press at 150 °C and 80  $\text{kg}/\text{cm}^2$  (7.8 MPa) for 1.5 h. The resulting composite exhibited no cracks and no bubbles as determined from SEM analysis.
2. Furthermore, the PFL grinding wheel was harder than the laboratory PF grinding wheel and had higher compression resistance. Moreover, in spite of the complex structure of lignin, the abrasiveness of PFL grinding wheel was still close to that of the laboratory PF grinding wheel. The metal tube was cut by PFL grinding wheel within 4 s. Moreover, the reaction between lignin, phenol, and formaldehyde was proven.
3. Finally, the PFL grinding wheel has high heat resistance. The carbon yield of PFL at 900 °C was as high as 56% during DSC/TGA tests. Meanwhile, by using the alkali lignin, the curing temperature of the PF resin can be increased. Thus, PFL resin could represent a prospective industrial product to replace PF resin, which is typically used in grinding wheel composites.

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