Chemical composition analysis and structural features of banana rachis lignin extracted by two organosolv methods

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ABSTRACT

Two organosolv methods involving Formic acid/Acetic (FA) acid and Sulphuric acid/Ethanol (SE) solvent mixtures were investigated for lignin extraction from banana rachis biomass residues. Different heating methods were also applied during each extraction process, respectively direct conduction heating and microwave heating. The chemical composition and structural features of the extracted lignin fractions were further analyzed by ATR-FTIR, TGA, elemental analysis and 13C NMR methods. SE extraction method showed a higher extraction yield (58.7%) and allowed also to obtain a lignin fraction with higher purity (76.5% Vs 71.0% for FA lignin). In addition, SE extraction method allowed a higher pulp yield which meant a better selectivity for lignin extraction thanks to the microwave heating method. SE lignin also showed a higher thermal stability due to its higher purity and higher density. The higher molecular weight found for FA lignin residues (7622.7g/mol Vs 5957.7g/mol for SEL) was suspected to be due to co-extracted carbohydrate residues bounded to extracted lignin macromolecules. These results allowed us to establish the SE extraction method (Sulphuric acid/Ethanol/water solvent with microwave heating) as effective for lignin extraction from banana rachis straw.

1. Introduction

Valorization of agro-industrial green waste by the biorefinery of their ligno-cellulosic fractions currently undergo intensive research. Amongst the main biomass agro-resources, residues from the banana industry have also been characterized (Cordeiro et al., 2007, 2004; Happi Emaga et al., 2008; Kamdem et al., 2011; Kamsonlian et al., 2011; Oliveira et al., 2006; Someya et al., 2002; Tiappi et al., 2015; Zuluaga et al., 2009). Banana (*Musa cavendish*) is an herbaceous annual plant, grown on an industrial scale and also by local populations in tropical areas. In 2016, more than 126 million tons of bananas were produced worldwide (FAOSTAT, last update May 2018). The generated organic residues from this important industry are of particular interest considering their availability and also their content in cellulose, hemicelluloses and lignin fractions. Banana rachis is among the most important residues from banana plant, generated during the harvesting process of banana fruits. The chemical composition analysis of that specific residue (Cordeiro et al., 2009; Tiappi et al., 2015) revealed that

their lignin content though lower in comparison to woody biomass, was found significant for potential valorization throughout the different platforms. Several lignin extraction methods from banana rachis including soda/dioxane solvent extraction method (Cordeiro et al., 2009) and steam explosion (Ibrahim et al., 2010) have been studied in the literature. However, solvent-based lignin extraction methods have not been fully explored for banana rachis.

Organosolv methods are among the most user-friendly and economically interesting because many of the solvents involved can be easily recycled. Moreover, the extracted lignin residues are of better quality and therefore offer a wider range of valorization pathways. Organosolv extraction method was originally developed by Delmas et al. (Boijsen et al., 1971) for the paper industry (bleaching of wood pulp) and was further applied for herbaceous biomass (Delmas et al., 2005; Nayeem Rumee et al., 2014; Manara et al., 2013; Sadeghifar et al., 2017; Sun et al., 1998, 2012, Vanderghem et al., 2012; Xu et al., 2006). The lignin extraction process consists in their chemical solubilization in the presence of an acid (proton donor). The process is

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generally carried out in hot conditions to promote hydrolysis reactions between the ligno-cellulosic sub-fractions (Harmsen et al., 2010). Water in the solvent extraction mixture facilitates homogeneous diffusion of heat in the reactor. Finally, the extracted lignin fractions are solubilized in a solvent solution (acetic acid or ethanol)

In the current work, two solvent based lignin extraction methods (acetic acid/formic acid/water and sulphuric acid/ethanol/water solvent mixtures) involving two different heating methods (direct conduction and microwave heating) were studied for banana rachis lignin extraction. The extracted lignin fractions were further characterized by physical (TGA/DTG, ATR-FTIR, ^{13}C-NMR, elemental analysis), and wet chemistry methods (Klason lignin, Size exclusion chromatography) and the impact of the extraction method was assessed.

2. Material and methods

2.1. Material

Banana rachis residues were collected from an industrial farm in Cameroon (Central Africa). Before treatment, fresh banana rachis were chopped, air dried until constant weight and coarsely grinded. The chemical composition of banana rachis is provided in Table 1. (Tiappi Deumaga et al., 2018)

2.2. Lignin extraction methods

2.2.1. Lignin extraction with Formic acid/Acetic acid/water

The FA lignin extraction parameters was adapted from the method by Vanderghem et al. (Quoc Lam et al., 2001) and are summarized in Table 2. Pre-treatments were performed in a 500 mL double-necked boiling flasks and heated by heating plates (direct conduction). At first, the dried banana rachis straw was soaked at 50 °C for 30 min with the extraction solvent mixture at a solid to liquid ratio of 1/25. After the soaking phase, the temperature was set to 107 °C and the mixture was cooked for 3 h (lignin extraction phase). The extraction reaction was stopped by removing the flasks from the heating plates, and the pulps were directly filtered with a vacuum filter (40–100 μm pore size). The pulps were further washed with hot water. The liquor and washes were collected and diluted with distilled water to reach a pH around 2 in order to allow precipitation of the extracted lignin. Finally the FA lignin (FAL) was recovered by centrifugation, washed with acidified water (pH 2), vacuum filtered (1–1.5 μm pore size) and freeze-dried.

2.2.2. Lignin extraction with sulphuric acid/ethanol/water

The lignin extraction parameters with microwave heating are summarized in Table 2. The dried banana rachis straw was mixed with the extraction solvent mixture made of Sulphuric acid/Ethanol/Water (5.4 / 92 / 2.6, %v/v) with a solid to liquid ratio of 1/20. The mixtures were realized just before the launch of the microwave extraction process to avoid influence of pre-hydrolysis reactions. Microwave extractions were carried out inside 80 mL reactors (EasyPrepPlus 80 mL Teflon vessels, max 12 vessels/run) at 1200 W power in a Mars6 microwave device (One Touch Technology, CEM, equipped with IR and optic fiber thermal probes). The extraction reaction holding time at the

Table 1
Chemical composition of crude dried rachis from Grande naine (Musa spp.) banana variety.

Chemical component	%w/w in crude material
Total ash	28.5
Total proteins	4.3
Extractives* (water & ethanol)	9.7
Cellulose (Van Soest)	35.3
Hemicelluloses (Van Soest)	17.9
Acid insoluble lignin (Klason)	6.0

Table 2
Lignin extraction parameters by Formic/Acetic acid (FA) and Sulfuric acid/Ethanol (SE) extraction methods.

Extraction parameters	FA Method	SE Method
Lignin extraction Solution	Formic acid / Acetic acid / Water 30 / 50 / 20	Sulfuric acid / Ethanol / Water 5.4 / 92 / 2.6
Extraction temperature (plateau)	50 °C–107 °C	161 °C
Extraction duration	30mins–180mins	10 min.
Solid to liquid ratio (S/L)	1/25	1/20

plateau temperature (161 °C) was 10 min. after which the microwave treatment was stopped. After a cooling period of about 10 min., the pulp mixtures were filtered with a vacuum filter (40–100 μm pore size) and washed with an ethanol/water mixture solution (92/8, %v/v). The ethanol fraction in the collected liquid was then removed by vacuum evaporation at maximum 45 °C. The residual solution was diluted with distilled water to reach a pH around 2 in order to allow precipitation of the extracted lignin. Finally the extracted SE lignin (SEL) was recovered by centrifugation, washed with acidified water (pH 2), vacuum filtered (1–1.5 μm pore size) and freeze-dried.

2.3. Klason Lignin analysis and structural characterization

Acid insoluble lignin also known as Klason lignin was determined after the two steps hydrolysis method (1 h pre-hydrolysis with 72% sulfuric acid solution followed by a 1 h 4% sulfuric solution treatment) as described by Sluiter et al. (2010).

Thermogravimetric degradation analyses were performed with a TGA/DSC1 instrument from Mettler-Toledo (Greifensee, Switzerland). The pyrolysis was conducted under a nitrogen flow of 20 mL/min. The experiments were conducted in triplicate using 70-μl alumina pan containing 10 mg of dried banana rachis straw. The thermal degradation analysis was carried out over a temperature range comprised between 40 and 600 °C at 10 °C/min heating rates. DTG graph was determined as the degradation rate over the temperature increment.

For ATR-FTIR structural analysis, about 5 mg of the extracted lignin residues (freeze-dried) were mixed with potassium bromide in a ratio of 1/100 and then mechanically pressed in order to form a pellet. The pellets were then analyzed by infrared spectroscopy and the FTIR spectra were recorded in the 4000–500cm^{-1} range with a resolution of 4 cm^{-1} on a Bruker IFS 48 spectrometer.

Molecular mass distribution of the extracted lignin sub-fractions was determined by Gel Permeation Chromatography (GPC, Agilent Technologies, 1200 series, equipped with Ultraviolet (UV) and Refraction Index detectors (RI)). Sodium polystyrene sulphonates molecules with molecular weight varying from 4.3 to 32 KDa were used to generate the correlation equation (Retention time Vs Molecular weight).

^{13}C NMR analyses were performed using a Varian Unity 600 MHz NMR analysis instrument. About 75 mg of each extracted lignin samples was dissolved in 0.75 mL of DMSO. Samples were then transferred in 5 mm SHIGEMI NMR micro-tubes and analyzed (50 °C/90° pulse angle/ 0.865 s acquisition time/2 s relaxation).

3. Results and discussion

3.1. Lignin extraction results

Results of lignin extraction by the FA and SE methods from banana rachis and characterization data are summarized in Table 3.

The extraction yield obtained for SE extraction method (58.7%) was higher than FA extraction method (45.3%). However, these results were higher than those obtained for lignin extraction from banana rachis by

Table 3

Results of lignin extraction by Formic/Acetic acid (FA) and Sulfuric acid/Ethanol (SE) extraction methods.

	FAL	SEL
Lignin extraction yield (%)	45.3	58.7
Pulp Yield (%)	43.6	61.3
Insoluble lignin (Klason) in material residue (%)	10.0	2.2

the soda/dioxane solvent extraction method (33%) and from banana stem by FA solvent extraction method (38%) (Cordeiro et al., 2009; Delmas et al., 2005). The higher extraction yield observed for SEL could be explained by a better selectivity of this method for the hydrolysis of covalent lignin-carbohydrates linkages compared to the FAL extraction method.

On the other hand, the pulp yield obtained for FA extraction method was less (43.6%) than the pulp yield for the SE extraction method (61.3%). Also, a higher lignin content in residual pulp from FA extraction method (10.0% Vs 2.2%) was observed. The most probable hypothesis to explain this phenomenon could be a significant solubilization of celluloses and hemicelluloses fractions during the FAL extraction process, resulting in a relatively higher lignin content in residual pulp. This pattern has been observed during lignin extraction by steam explosion treatment (Ibrahim et al., 2010).

3.2. Chemical composition of the extracted lignin

The chemical analysis of the insoluble lignin extracted by the FA and SE extraction methods was carried out and the results are displayed in Table 4. It was observed that the Klason lignin content in the SE lignin (76.5%) was higher than the FA Klason lignin content (71.0%). That difference could be explained by the comparatively harsher hydrolytic conditions of the SE method thanks to the use of a strong acid as proton donor as well as the higher temperature, and also by its higher selectivity. In general, the significant purity values obtained with both extraction methods were comparable to those obtained by Vanderghem et al (Quoc Lam et al., 2001) for the lignin extraction by the FA method from Miscanthus gigantus (80%). Argyropoulos et al. (2002) demonstrated that extraction of higher purity lignin (> 90%) specifically for analytical purposes required additional acidolytic and enzymatic treatments. We therefore considered that the purity of the lignin fractions extracted both by the simple FA and SE methods was significant. However, the use of sulphuric acid as a hydrolysis promoter was potentially the main drawback for SEL as additional neutralization purification treatments could be required in some lignin application.

3.3. Elemental analysis of extracted lignin

The results of the elemental analysis in Table 4 show a higher proportion of carbon in SE lignin (63.8%) relatively to FA lignin (58.6%). The carbon atoms content in lignin fractions can be correlated to the amount of aromatic carbons from lignin sub-units (p-hydroxyphenyl, guaiacyl, and syringyl). The higher carbon content observed

Table 4

Chemical composition of FA and SE lignin fractions.

Chemical component	FAL	SEL
Insoluble lignin (Klason) of extracted lignin residue (%)	71.0	76.5
Soluble lignin (Klason) of extracted lignin residue (%)	3.2	4.6
Molecular Weight (g/mol)	7622.7	5957.7
C / H / N Elementary analysis (%)		
Carbon	58.6	63.8
Hydrogen	6.6	7.3
Nitrogen	3.7	2.5
Final residue	31.0	26.4

for SE lignin can therefore be explained by its higher purity relatively to FA lignin (see Table 4).

3.4. Thermogravimetric analysis

Thermal degradation graphs (TGA & DTG) of FA and SE lignin fractions are displayed in Fig. 1. The analysis of the DTG graph allowed us to distinguish 4 degradation phases. The first phase from 35 °C to 125 °C was related to the residual moisture elimination from the rachis straw samples. The second phase from 125 °C to 280 °C was mainly related to the destruction of carbohydrates (mainly hemicelluloses) co-extracted with lignin and also the destruction of low molecular weight lignin. The third and fourth phases from 280 °C to 430 °C and from 430 °C to 480 °C respectively was associated mainly to the degradation higher molecular weight and high density lignin fraction. Finally, the last phase from 480 °C to 600 °C was associated to the degradation of residual crystalline cellulose (Wu et al., 2009). These lignin fractions degradation profiles were similar to those obtained by Vanderghem et al. and Ibrahim et al. (Ibrahim et al., 2010; Quoc Lam et al., 2001). It was also observed that the degradation rate of the SEL fraction was higher than the FAL fraction during the carbohydrate degradation phase (125 °C to 280 °C). This could be due to the presence of lower molecular weight hemicelluloses or lower molecular weight lignin in the SE lignin fraction due to the harsher hydrolytic conditions as subjected above. However, during the lignin degradation phase (from 280 °C to 480 °C), a reverse trend was observed: the degradation rate of SE lignin was slower than FA lignin. The slower degradation of SEL could be explained by its higher purity and its potentially higher lignin density (Ibrahim et al., 2010) which resulted in a greater resistance to thermal degradation.

3.5. Molecular weight analysis

The molecular weight analysis of the extracted lignin (see Table 4) showed that SE lignin had a lower molecular weight (5957.7 g/mol) than FA lignin (7622.7 g/mol). This result could be related to harsher hydrolytic conditions of the SE extraction method leading to greater depolymerization reactions of the extracted lignin macromolecules. This hypothesis can also be justified by the higher fraction of soluble lignin (low molecular weight lignin) in the SEL fraction (4.6%) compared to FAL lignin (3.2%). On the other hand, the higher molecular weight found for FA lignin residues could be due to the contribution of co-extracted carbohydrates residues and bounded to lignin macromolecules.

3.6. Infrared spectroscopy analysis

The infrared spectroscopy analysis of the extracted lignin is shown in Fig. 2. Identification of the lignin moieties absorption wavelengths was based on literature (Ibrahim et al., 2010; Liu et al., 2010; Manara et al., 2013; Quoc Lam et al., 2001). It appeared that for the aromatic C–H group's absorption region (1121 & 1032 cm^{-1}) and also for the aromatic carbon atoms absorption region (1510–1460 cm^{-1}), absorption intensity was higher for the SEL fraction. That could be explained by the higher purity of the SEL fraction in comparison to FAL fraction. Similarly, the absorption intensity at 1332 cm^{-1} and 1220 cm^{-1}, representative of synapyl and conyferyl unit respectively was greater for the SEL fraction. In addition, the higher absorption intensity for aliphatic C–H groups (1370 cm^{-1} and 2850-2920 cm^{-1}) for SE lignin could be related to methoxyl groups of synapyl and conyferyl units. A higher content in synapyl and conyferyl units for the SEL fraction could justify the hypothesis of its higher density compared to FAL fraction. In fact, the high substitution degree of synapyl and conyferyl aromatic rings (-methoxy groups principally and also -ethoxy groups from ethanol) renders their reactive positions sterically less available for polymerization and thus contributing to a higher macromolecule
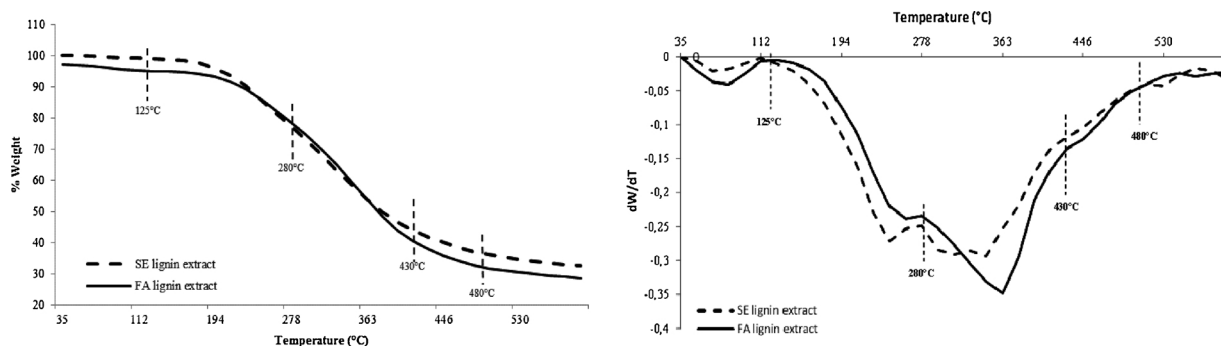
Fig. 1. TGA and DTG analysis of FA and SE lignin fractions.

density (Tondi et al., 2016).

3.7. C NMR analysis

The ^{13}C NMR spectrum of FAL & SEL fractions are presented in Figs. 3 and 4. Chemical movement analysis was conducted using literature databases (Cordeiro et al., 2009; Liu et al., 2010; Quoc Lam et al., 2001; Sun et al., 2011). The area between δ_C 100 and δ_C 165 ppm was representative of aromatic carbons from monolignols structural units. The specific peak at 165.8 ppm observed in the FAL fraction was characteristic of acetate groups in residual acetic acid molecules from the extraction solution.

Moreover, similar signals were also identified both in FAL and SEL fractions, notably the NMR signals for lignin's C_5-C_5 bonds (130.8–132.8 ppm) as well as signal for the lignin's C_6 carbon in *p*-coniferyl units (118.9 ppm). The main structural difference between the two lignin residues were found in the aliphatic area with the presence of β-O-4 bonds signals in the SEL fraction (62.6–73.0 ppm). The presence of these signals in the SEL residues could explain the higher polymerization degree and thus higher density of SE lignin extract. This assumption was also consistent with a higher density of the SE lignin extract.

3.8. Influence of the heating method

Microwave and direct conduction heating methods were studied in the current research work for lignin extraction with sulphuric acid/ethanol/water and formic acid/acetic acid/water solvent mixtures respectively. Considering the above results, the microwave extraction technique was demonstrated to be the most effective in term of extraction yield and lignin purity. These result were consistent with the research by Ruiz et al. (2017) describing the potential of microwave heating in biorefinery. In fact, while providing fast and cost effective

processing of biomass with low generation of degradation compounds, microwave heating processing allows selective hydrolysis reactions for dipolar chemical bounds. In the case of lignin extraction, mainly ester and ether covalent bounds within the lignin polymer and also between lignin and hemicellulose residues are targeted. Those hydrolysis reactions are also promoted by the higher temperature obtained with the microwave heating compared to direct conduction where the reaction temperature is limited by the solvent boiling point.

4. Conclusion

Two organosolv pretreatment methods were investigated for lignin extraction from banana rachis straw. The first method involved Formic acid/Acetic acid/water (FA solvent) with a direct conduction heating and the second method involved Sulphuric acid/Ethanol/water (SE solvent) with microwave heating. SE extraction method showed a higher extraction yield (58.7% Vs 45.3% for FAL) and also a higher purity of the extracted lignin fraction (76.5% Vs 71.0% for FAL). In addition, SE extraction method allowed a higher pulp yield which meant a better selectivity for lignin extraction thanks to the microwave heating method. SE lignin also showed a higher thermal stability due to its higher purity and higher density. The higher molecular weight found for FA lignin residues (7622.7 g/mol Vs 5957.7 g/mol for SEL) was suspected to be due to co-extracted carbohydrate residues bounded to extracted lignin macromolecules. These results allowed us to establish the SE extraction method (Sulphuric acid/Ethanol/water solvent with microwave heating) as effective for lignin extraction from banana rachis straw. However, depending on the application of the extracted lignin, the presence sulphuric acid residues would require additional purification treatments.
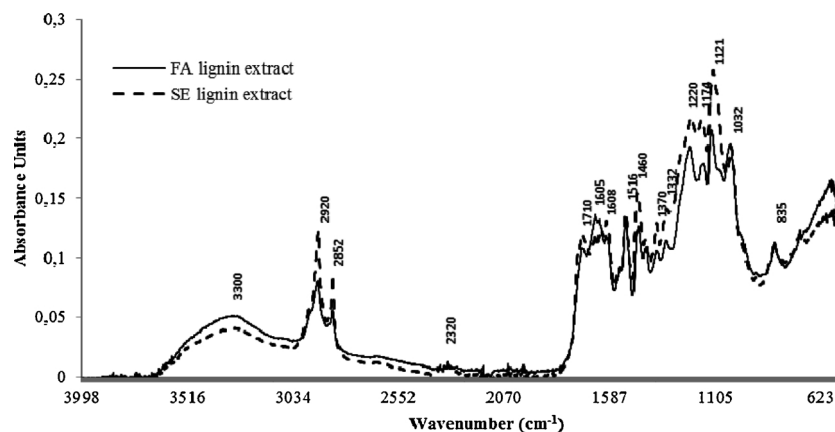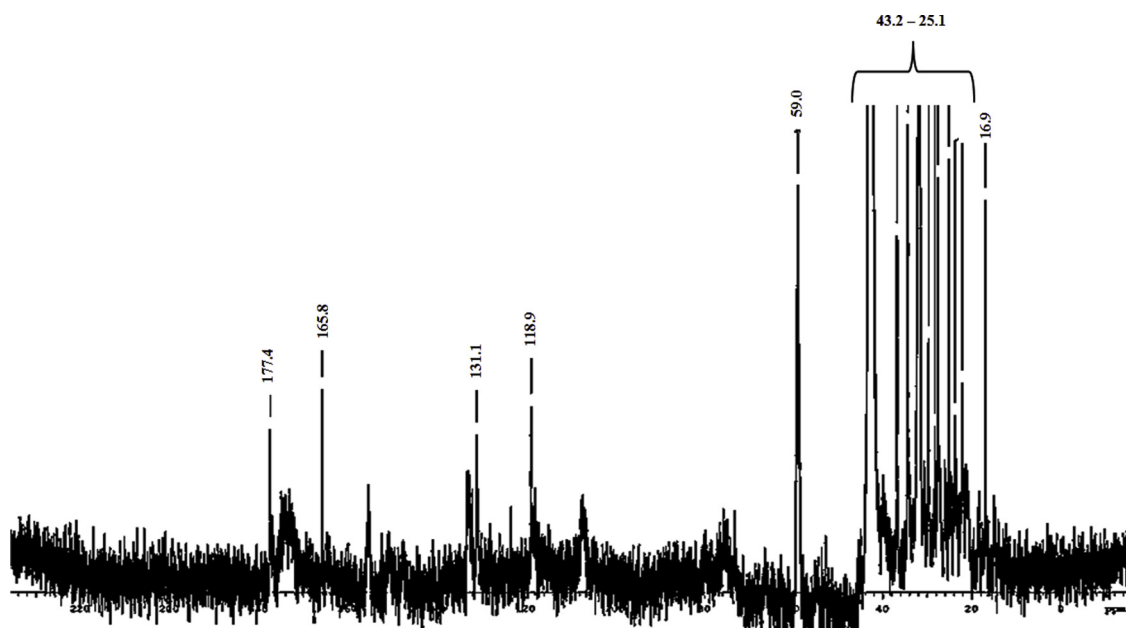

Fig. 2. ATR-FTIR analysis of FA and SE lignin fractions.

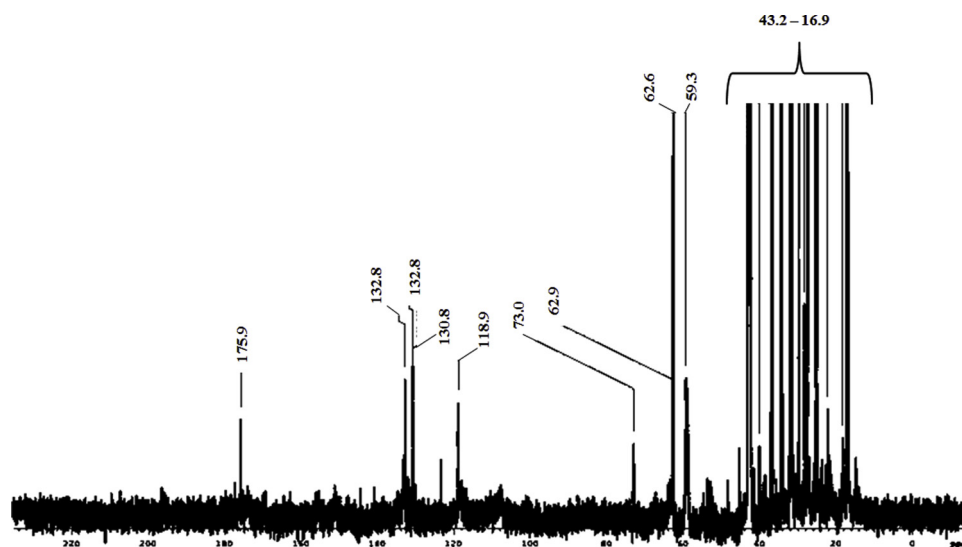**Fig. 3.** ^{13}C RMN analysis of FA lignin fraction.

Fig. 4. ^{13}C RMN analysis of SE lignin fraction.

Acknowledgments

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