# SUNLIBB

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**"Production of high value phenolic compounds from C4 biomass"**


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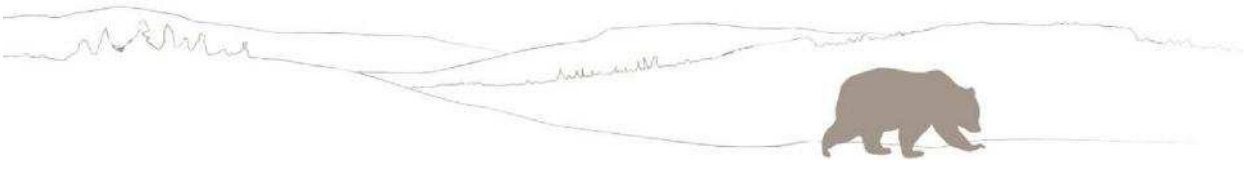

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Title **Production of high value phenolic compounds from C4 biomass**

Objective

Lignin breakdown products will be studied as a source of valuable aromatic building blocks for chemical synthesis and these are being analysed in Task 4.2. P3 has existing technology for converting wood lignin into vanillin and other valuable products and will adapt this technology to work with lignin from C4 grasses. New technologies will be considered aiming at commercialising inventive and high value-added applications for those lignins in both existing and new markets.

Conclusion

The samples mainly contained different phenols of lignin origin and carbohydrate derived pyrolysis products. Also polyaromatic compounds and fatty acids could be found. No component was found to be of unique enough character and high enough concentration that any further separation could be justified.

The analysis shows that the complex starting materials give mainly complex product mixtures. The samples analysed here are not significantly different from other pyrolysis samples we have analysed.

The use of microwave pyrolysis is an effective tool for generating higher value products from lignin, however further technologies need to be developed to effectively separate molecules into commercially important fractions. The pyrolysis and fractionation techniques used for these samples were not able to fine tune the decomposition of the biomass in to a product mixture which is commercially interesting. Both the pyrolysis and fractionation techniques need to be altered to provide fractions which are more homogenous in composition. Inhomogenous samples, such as these, will not give any more information than that the sample is a complex mixture and thus not commercially interesting.

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# 2 Introduction

**Microwave pyrolysis was highlighted as a technology for the processing of lignin into higher value products. Pyrolysis samples of maize, bagasse and Miscanthus from the University of York have been analysed by GC-MS in order to identity of the main components in the samples and highlight areas of commercial interest.**

# 3 Results and

# Discussion 3.1 Maize

**Three maize samples were analysed, the samples are listed in Table**

**3.1. Table 3.1 Maize samples.**

| Sample | Sample Name | Dilution |
|---|---|---|
| SP-2693-1 | Maize tusk | 104.8 mg/ 10 ml |
| SP-2693-2 | Maize stem | 88.8 mg/ 10 ml |
| SP-2693-3 | Maize leaves | 90.2 mg/ 10 ml |

**The GC chromatograms of the three samples were similar with only minor variations, as seen in Figure 3.1 to Figure 3.3. There were however some variations in the intensities of the individual peaks.**

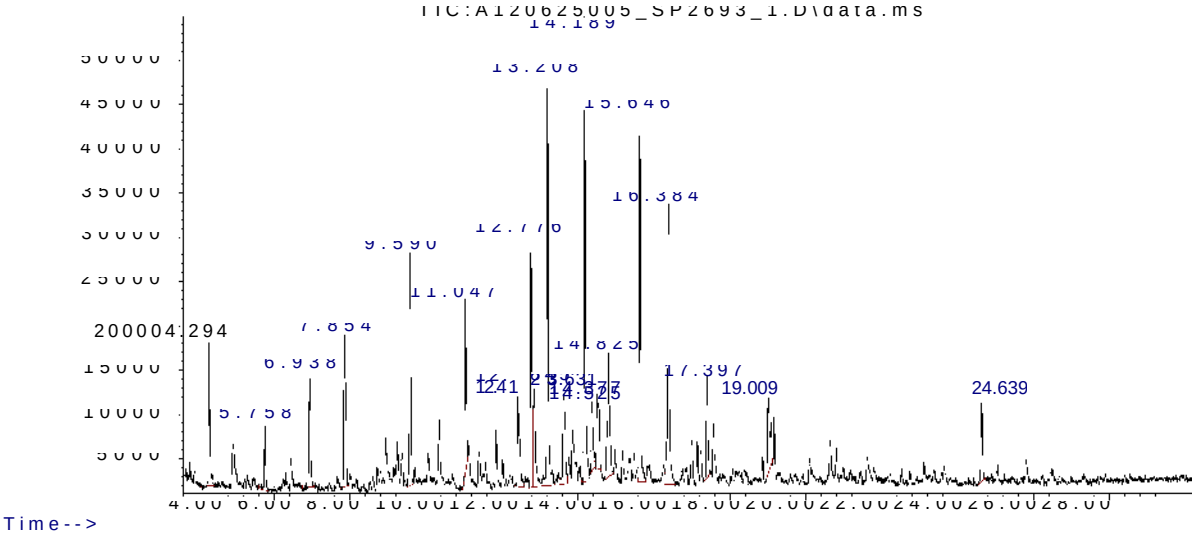



**Figure 3.1 GC chromatogram of Maize tusk.**

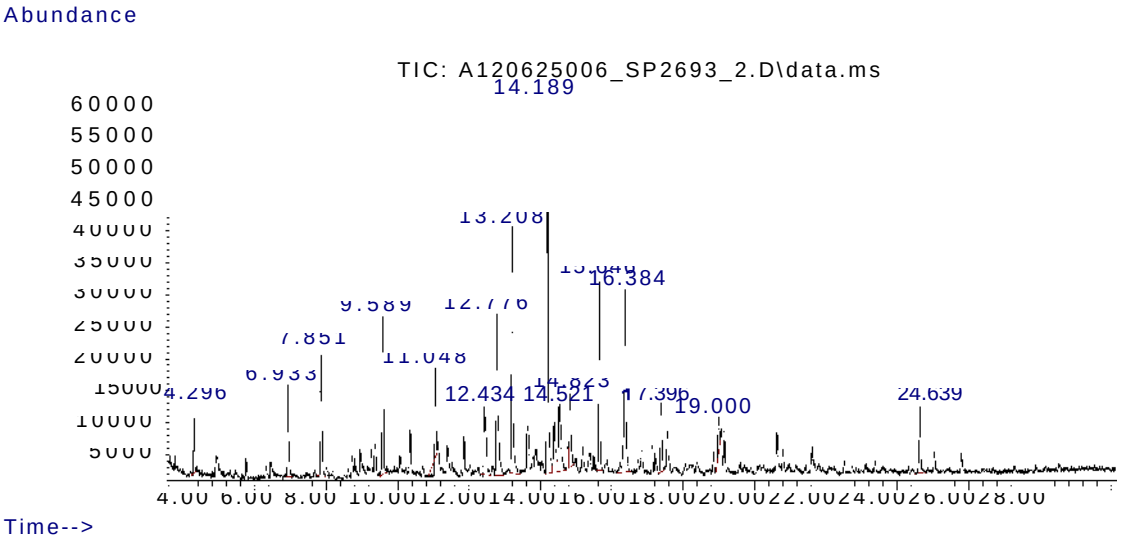



**Figure 3.2 GC chromatogram of Maize stem.**

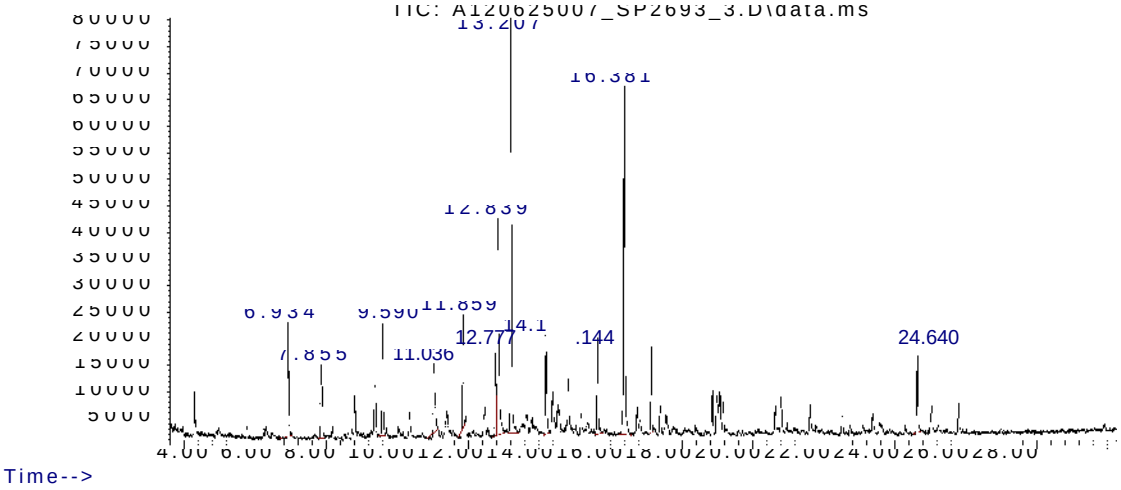



**Figure 3.3 GC chromatogram of Maize leaves.**

The MS analyses of the chromatographs are given in Table 3.2. The identity of the peaks are given by comparison with NIST library.

**Table 3.2 MS identification of the peaks in the GC-chromatograms of Maize by comparison with NIST library.**

| Rention time (min) | Compound | NIST match |
|---|---|---|
| 4.294 | 2-Furanmethanol (furfuryl alcohol) | 930 |
| 5.758 | 2,2-Diethoxyethanol | 857 |
| 6.938 | Phenol* + unknown | |
| 7.854 | 3-Methyl-1.2-cyclopentanedione/ | 970/ |
| | 2-Hydroxy-3-methyl-2-cyclopenten-1-one | 970 |
| 9.590 | 3-Methoxyphenol (guaiacol)* | 940 |

| | | |
|---|---|---|
| 11.047 | ? | |
| 12.776 | 1,2-Benzenediol (catechol) | 979 |
| 13.208 | ? | |
| 14.189 | ? | |
| 14.825 | 4-Ethyl-2-methoxyphenol (ethyl guaiacol) | 838 |
| 15.646 | 2-Methoxy4-vinylphenol/ | 922/ |
| | 1-(2-Hydroxy-5-methylphenyl)-ethanone | 919 |
| 16.384 | 2,6-Dimethoxyphenol (syringol) | 941 |
| 17.397 | Vanillin | |
| 19.009 | Levoglucosan* | 933 |
| 24.639 | 2,6-Dimethoxy-4-(2-propenyl)-phenol* | 805 |

* Identify also comfirmed in acetylated samples.

The chromatograms all show that the pyrolysis of the maize samples gives rather complex mixtures, where isolating pure compounds would be difficult and most probably not economically feasible. The presence of vanillin is interesting, but we believe that isolation from this mixture would be a bad idea. It would be better if the pyrolysis conditions could be tuned to give more vanillin instead.

### 3.2 Bagasse

Three bagasse samples were analysed, the samples are listed in Table 3.3.

**Table 3.3 Bagasse samples.**

| Sample | Sample Name | Dilution |
|---|---|---|
| SP-2693-4 | Bagasse bigger particles | 92.3 mg/ 10 ml |
| SP-2693-5 | Bagasse mid particles | 102.9 mg/ 10 ml |
| SP-2693-6 | Bagasse smaller particles | 91.0 mg/ 10 ml |

The GC chromatograms of the samples were more or less similar, as seen in figure Figure 3.4 to Figure 3.6. The variation in the chromatograms was only caused by differences in intensities of some peaks.

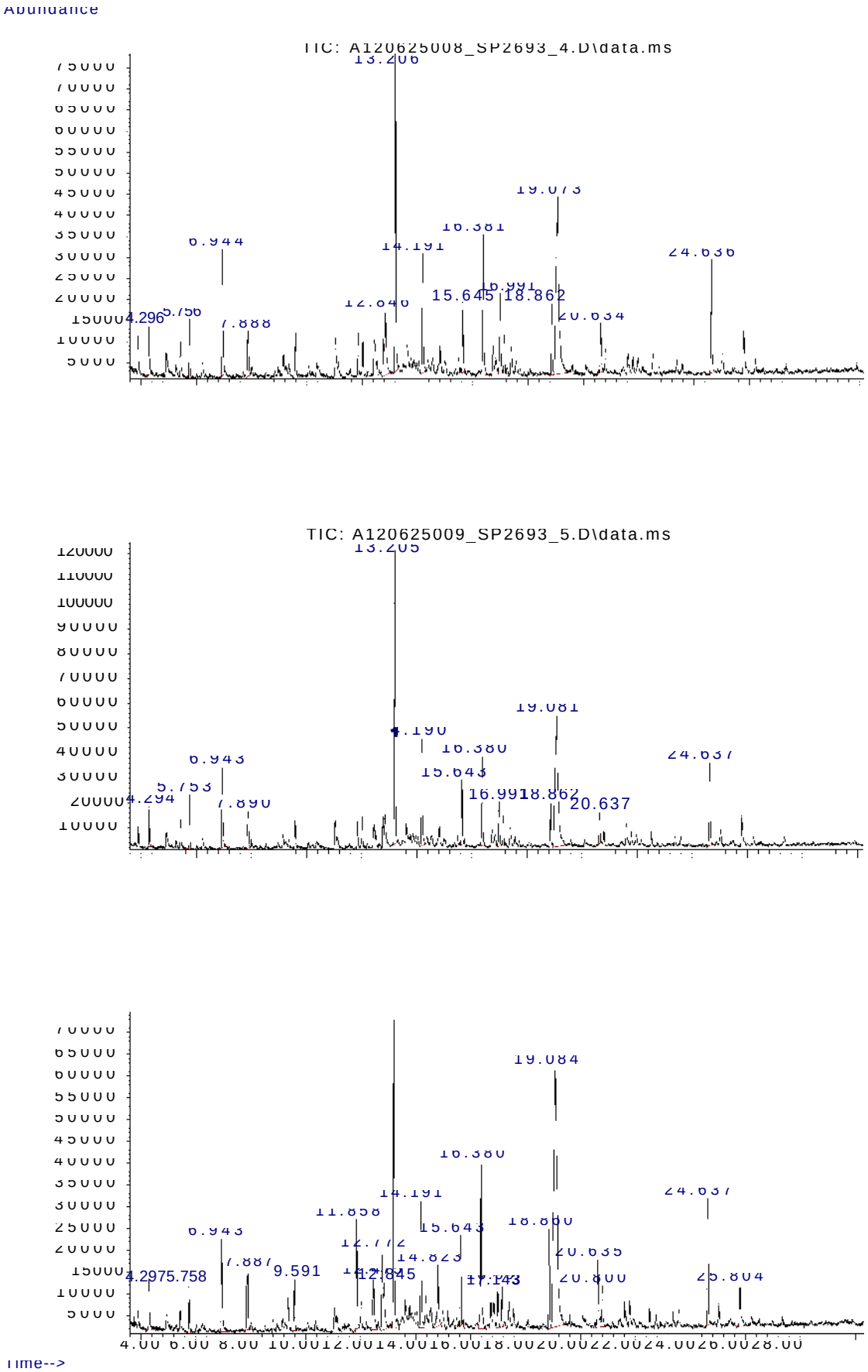



**Figure 3.6 GC chromatogram of Bagasse smaller particles.**

The MS analysis of the chromatographs are given in Table 3.4. The identities of the peaks are given by comparison with NIST library.

**Table 3.4 MS identification of the peaks in the GC-chromatograms of Bagasse by comparison with NIST library.**

| Retention time (min) | Compound | NIST match |
|---|---|---|
| 4.297 | 2-Furanmethanol | 923 |
| 5.758 | 2.2-Diethoxyethanol | 905 |
| 6.943 | ? (some is probably phenol) | |
| 7.887 | ? (some is probably phenol) | |
| 9.590 | Guaiacol | 906 |
| 11.047 | ? | |
| 11.858 | 4-Ethylphenol | 958 |
| 12.475 | 2-Methoxy-3-methylphenol | 873 |
| 12.772 | 1.2-Benzenediol (catechol) | 959 |
| 13.205 | ? | |
| 14.191 | ? | |
| 14.823 | 4-Ethyl-2-methoxyphenol | 873 |
| 15.643 | 2-Methoxy-4-vinylphenol/ | 920/ |
| | 1-(2-Hydroxy-5-methylphenyl)-ethanone | 916 |
| 16.380 | 2.6-Dimethoxyphenol | 975 |
| 18.860 | 1.2.4-Trimethoxybenzene | 892 |
| 19.009 | Levoglucosan | 933 |
| 20.635 | ? | |
| 20.800 | ? | |
| 24.639 | 2.6-Dimethoxy-4-(2-propenyl)-phenol | 902 |

The GC chromatograms shows that also the pyrolysis samples of bagasse were quite complex mixtures where isolation of individual compounds will prove difficult.

## 3.3 Miscanthus

One of the key issues in pyrolysis is separating and collecting the wide variety of products obtained from the process. A unique feature of microwave decomposition of biomass is a low temperature of pyrolysis, where the observed temperature of the process is always below 200 $^{o}$C. In this temperature range the majority of the organic compounds are stable and could be potentially separated using standard techniques. This observation creates the opportunity for *in-situ* separation of volatiles obtained during microwave pyrolysis. To

increase efficiency of separation, experiments were carried out at relatively large scale (samples mass between 90 and 200g) using a commercially available "Milestone" large cavity multi-mode microwave oven as a pyrolyser.

In the early stage of this project a simple microwave pyrolysis rig was used to develop the system (see Figure 3.6) and allow separation of all volatile components into low and high temperature fractions.

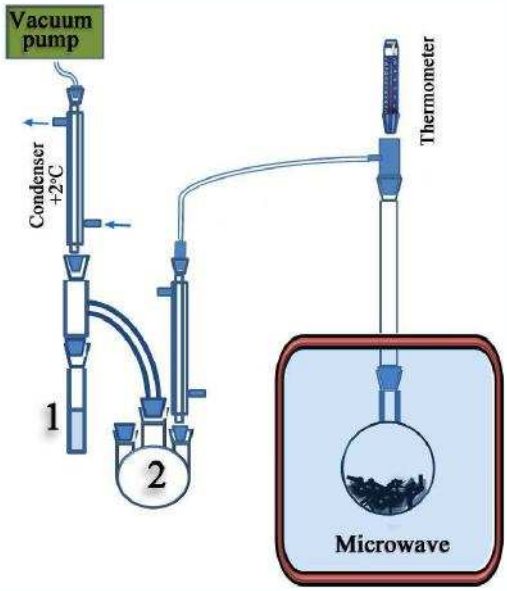



**Figure 3.6. Schematic of apparatus for in-situ bio-oil separation.**

The low temperature fraction of volatiles is condensed using a condenser at 2 $^{o}$C and collected in vessel 1. High temperature fraction is condensed at room temperature into flask 2. Photographs of two sample fractions obtained during microwave pyrolysis of wood are shown in Figure 3.7.

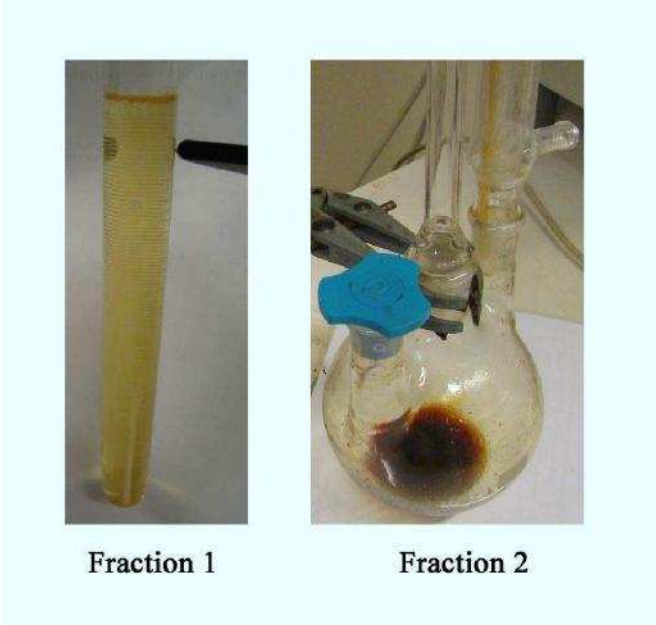



**Figure 3.7. Photograph of aqueous and organic fractions generated from in-situ separation of liquid products from microwave pyrolysis of wood.**

As can be seen in Figure 3.7, there is a clear difference in properties of the fractions: the first fraction is a transparent low viscosity aqueous solution of organic compounds. The other fraction is a higher viscosity dark opaque organic oil containing a small quantity of water.

Using set-up demonstrated on figure 3.6 microwave pyrolysis of Miscanthus have been carried out according the procedure described below: 130 g of Miscanthus dust was microwaved in Millstone MW processor with MW power 1200W during 15 minutes under vacuum 40 mbar. The maximum temperature of pyrolysis 200 $^{o}$C was achieved after 10 minutes of the run and the rest five minutes of the experiment this temperature has being kept constant.

As result of microwave assisted pyrolysis of Miscanthus four type of products were obtained: aqueous fraction (19%) bio-oil (21% of yield), bio-char (36% of yield) and gas fraction (24% of yield).

Obtained bio-oil was further fractionated using the following procedure:

1. 100 ml of ethyl acetate was added to bio-oil
2. 40 ml of 0.1M aqueous solution of NaOH was added to the ethyl acetate- bio-oil mixture.
3. The mixture was shaken and let settle. After separation two fraction were obtained:
    a. The ethyl acetate fraction. Organic solvent was distilled out and the remains kept as "Fraction from EtOAc" (60% of tar or 12% of original mass).
4. The aqueous fraction. The aqueous fraction was acidified and extracted with dichloromethane. and dried. After separation, two fractions were obtained:
    a. The Aqueous fraction-2. It was freeze-dried and kept as "Fraction from water 15/5/2013- SP**-3598"** (3.4% of tar or 12% of original mass).
    b. The DCM solution. DCM was removed under vacuum. Solid residue was washed with ethyl acetate generating two fractions:
        i. Remained solid fraction was kept as "Fraction from organic DCM 14/5/2013- SP**-3599"** (18% of tar or 3.8% of original mass)
        ii. Ethyl acetate solution. ethyl acetate was removed. Remained solid fraction was kept as "Fraction 2 from EtOAc 14/5/2013- SP-3601" (24.2% of tar or 5.1% of original mass).
5. DCM extraction can be repeated to enhance the overall yield

Fractionated samples from pyrolysis of miscanthus were analysed by GC-MS, the samples are listed in Table 3.5.

**Table 3.5 Fractionated samples from pyrolysis of mischantus.**

| Sample | Sample description | Remarks |
|---|---|---|
| SP-3598 | Fraction from water 15/5/2013 | Brown powder |
| SP-3599 | Fraction from organic DCM 14/5/2013 | Semi solid |
| SP-3600 | Fraction from EtOAc | Liquid oil with strong smell |
| SP-3601 | Fraction 2 from EtOAc 14/5/2013 | Liquid oil with strong but different |

Sample SP-3598 was difficult to dissolve in various organic solvents. The GC chromatogram, shown in Figure 3.8, had a low intensity due to the low solubility. MS analysis of the chromatogram proved difficult, but the highest peaks eluting at 16.26 and 18.94 minutes are most likely of a carbohydrate origin.

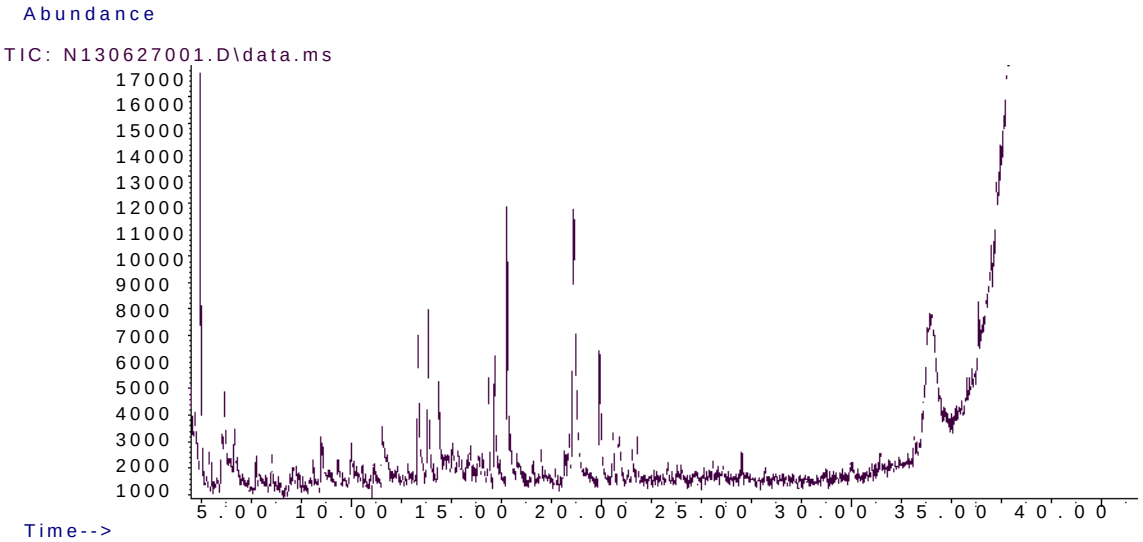
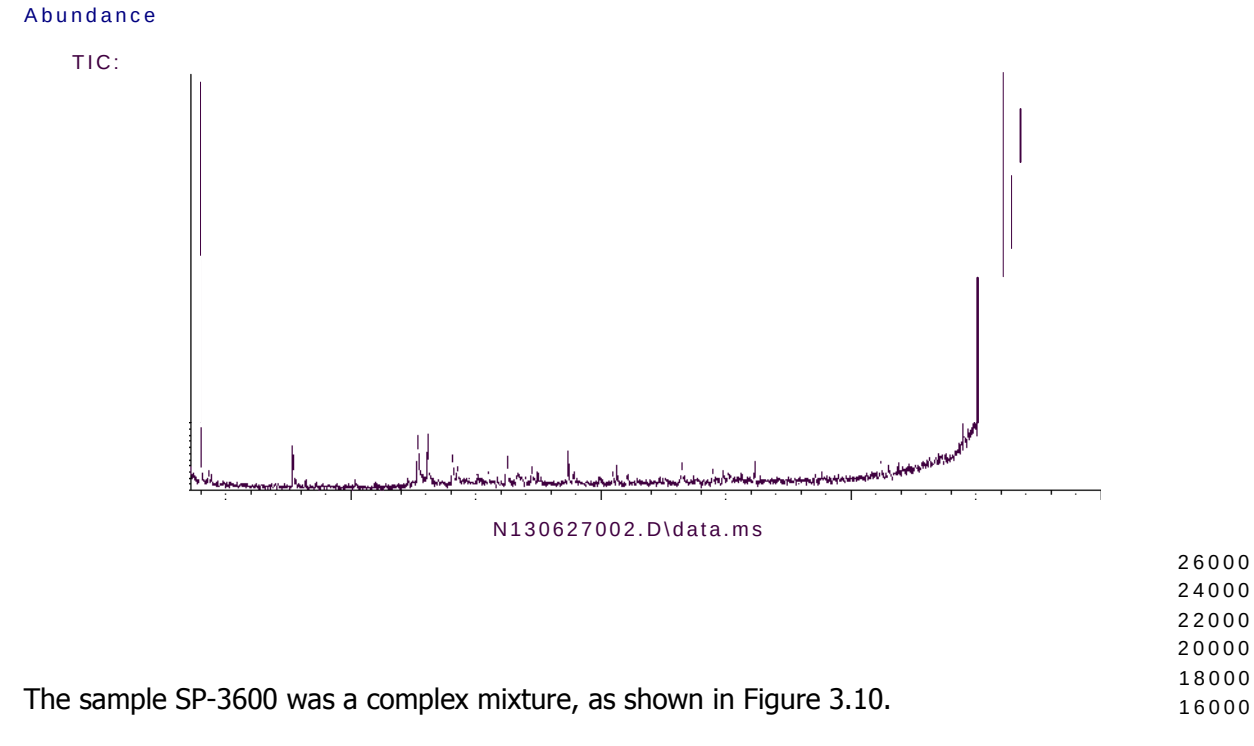



**Figure 3.8 GC chromatogram of sample SP-3598.**

The sample SP-3599 was also difficult to dissolve. The GC chromatogram, shown in Figure 3.9, had an even lower intensity than SP-3598. The MS analysis of this sample showed that the main peaks were lignin derived, catechol and syringol, eluting at 12.65 and 16.28 minutes respectively. The peaks at 7.68 and 18.7 minutes were most likely of carbohydrate origin.




The sample SP-3600 was a complex mixture, as shown in Figure 3.10.

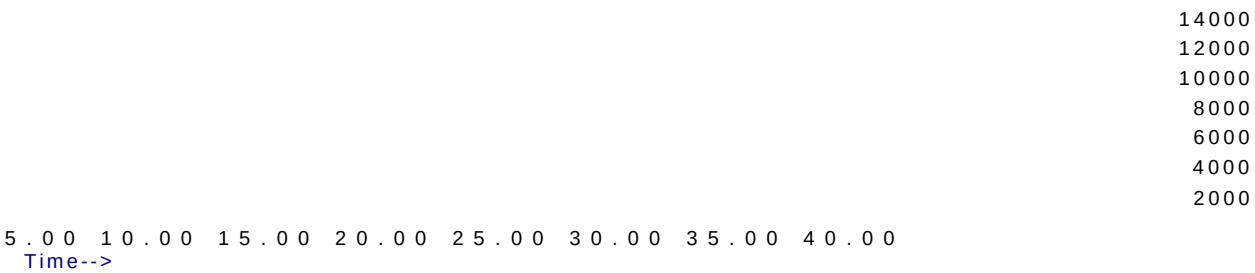



**Figure 3.9 GC chromatogram of sample SP-3599.**

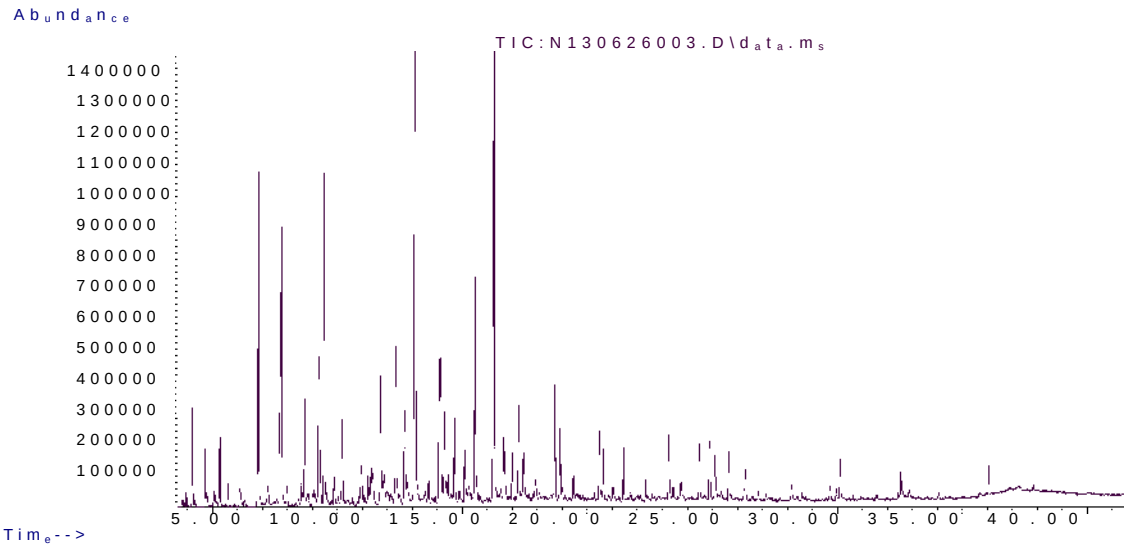



**Figure 3.10 GC chromatogram of sample SP-3600.**

The peaks identified by MS are given in Table 3.6.

**Table 3.6 MS identification of the peaks in the GC chromatogram of sample SP-3600.**

| Retention time (min) | Compound |
|---|---|
| 6.8 | Phenol |
| 8.7 | *o*-Cresol |
| 9.3 | *m*-Cresol |
| 9.5 | Guaiacol |
| 11.7 | 4-Ethylphenol |
| 12.3 | Creosol |
| 12.7 | Cathecol |
| 13.1 | 2,3-Dihydrobenzofuran |
| 15.5 | Vinylguaiacol |
| 16.3 | Syringol |
| 17.3 | Vanillin |
| 17.4 | Ethylcatechol |
| 18.7 | Trimethoxybenzene |
| 18.9 | Isoeugenol |
| 19.5 | Acetovanillone |

Syringol and 2,3-dihydrobenzofuran were the the highest intensity peaks in the sample. Most of the peaks appear to be lignin derived, although some carbohydrate derived peaks were also seen (trimethoxybenzene). Both isoeugenol and vanillin are interesting commercially, but they are present in too low concentration for it to be practically possible to isolate them in pure enough quality.

The sample SP-3601 was also a complex mixture, Figure 3.71. Comparing this chromatogram of sample SP-3600 we see that this sample appears to be somewhat more purified as the total amount of peaks have decreased.

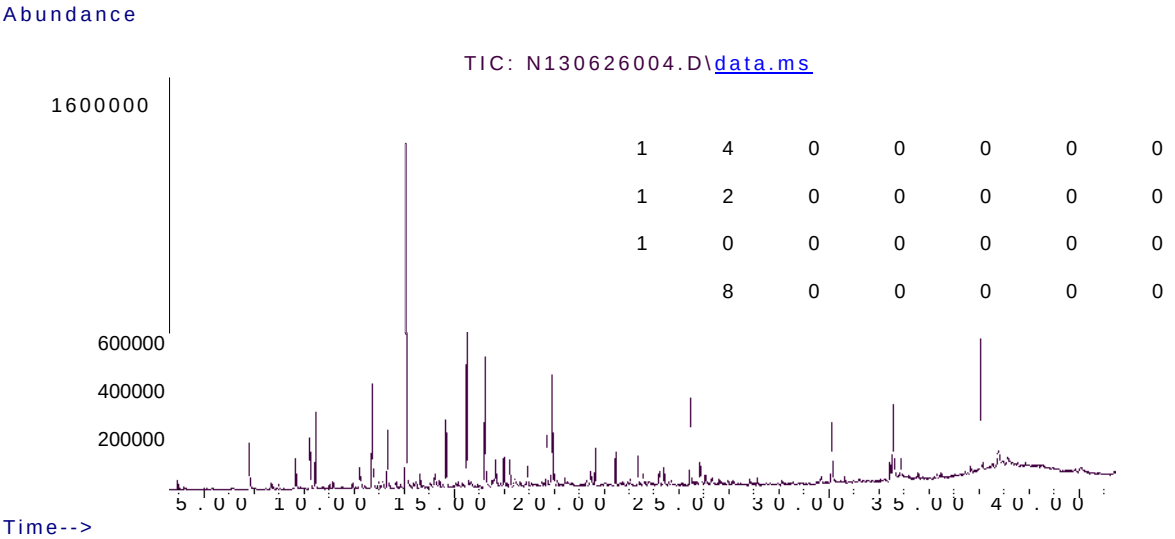



**Figure 3.71 GC chromatogram of sample SP-3601.**

The main peak in this sample was 2,3-dihydrobenzofuran, other peaks are given in Table 3.7. This product could be of interest and represent a high value product but further work would be required to develop applications for this molecule.

**Table 3.7 MS identification of the peaks in sample SP-3601**

| Retention time (min) | Compound |
|---|---|
| 7.9 | Phenol |
| 8.7 | *o*-Cresol |
| 13.1 | 2,3-Dihydrobenzofuran |
| 15.5 | Vinylguaiacol |
| 16.2 | Syringol |
| 18.7 | 1,2,4-Trimethoxybenzene |
| 22.3 | 2,6-Dimethoxy-4-allylphenol |

In addition to the compounds listed in the table above also palmitic an oleic oil were found in the samples, as well as the softening agent diisooctyl phthalate.

**In-situ fractionation**

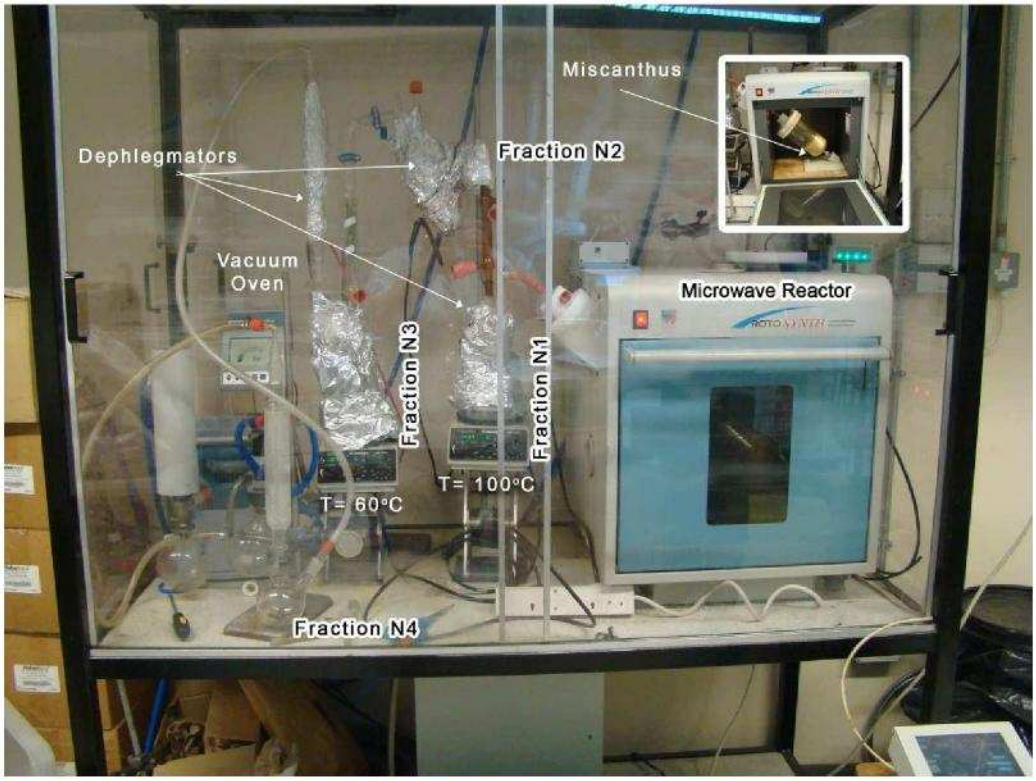



**Figure 3.12. Second generation microwave pyrolysis system with improved in-situ separation of liquid components (Vacuum ca. 20 mbar)**

For further development of MW technology aimed to separate the various fractions and sub-fractionate *in-situ (see Figure 3.12).* The benefits of this are multi-fold: post process separation is extremely energy and time consuming in comparison with the process itself. As described above and in Figures 3.6 and 3.7 microwave pyrolysis lends itself to condensation fractionation of the products generated as volatile liquids. Fraction 4, the aqueous fraction, represents the low temperature boiling compounds consists from physi- and cheniadsorbed water; acids and aldehydes. This fraction emerges with a vapour temperature of 40-50 $^{o}$C (under vacuum). Following this, at the pyrolysis temperature (as defined by the generation of oil and colour change), a continuous vapour stream is observed. Using the various condensers as described earlier the *in-situ* separation of oil fraction and water can be achieved. In total 4 fractions of oil have been obtained: Fraction 1 (12F056-T1-05)- 9.04%, Fraction 2 (12F056-T1-04)- 5.54%, Fraction 3 (12F056-T1-03)- 7.3%; Fraction 4 (aqueous fraction) (12F056-T1-02)- 17%.

In Table 3.8 the samples are listed and the fractionation method given.

**Table 3.8 Samples analysed**

| Sample ID | Sample Name | Fractionation method | Remarks |
|---|---|---|---|
| 12F056-T1-01 | Bio oil f2N2 | In-situ | Solid |
| 12F056-T1-02 | Miscanthus fraction N1 | ? | Solid |
| 12F056-T1-03 | Miscanthus fraction N2 | ? | Solid |
| 12F056-T1-04 | Miscanthus fraction N3 | ? | Solid |
| 12F056-T1-05 | Miscanthus fraction N4 | ? | Solid |

All the samples were dissolved in acetonitrile and filtered through a 0.45 µm filter before the GC-MS analysis. Some fractions were very inhomogenous, which made it difficult to take out a representative sample. The solid residue after acetonitrile treatment was not analysed.

All the samples gave GC chromatograms showing complex mixtures, like previous samples we have analysed. Below the chromatograms are given together with the identity of the main peaks.

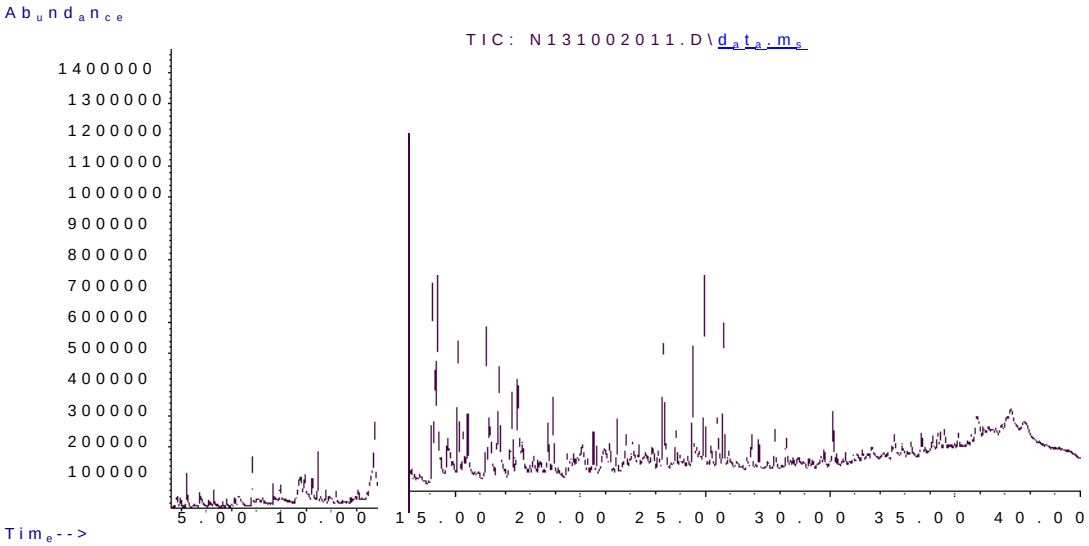



**Figure 3.13 GC-chromatogram of sample 12F056-T1-01.**

The main peaks in sample 12F056-T1-01 were: 2,3-Dihydrobenzofuran (RT 13.1), catechol (RT 12.7), guaiacol (RT 9.5), 4-hydroxybenzaldehyde (16.8) and methoxy eugenol (RT 24.5).

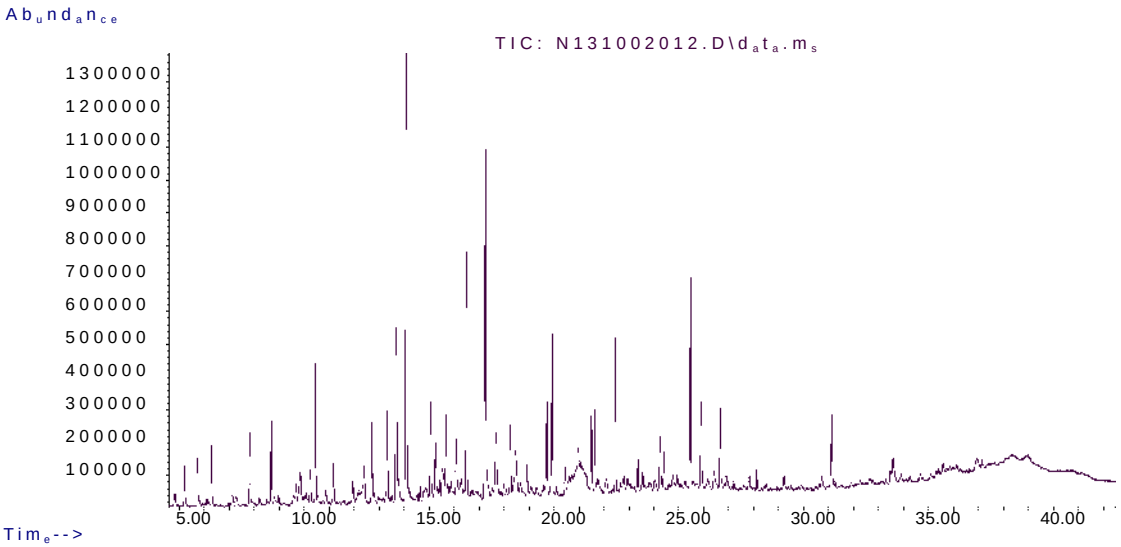



**Figure 3.14 GC-chromatogram of sample 12F056-T1-02.**

**The main peaks in sample 12F056-T1-02 were: 2,3-Dihydrobenzofuran (RT 13.1), syringol (RT 16.2), methoxy eugenol (RT 24.5) and vinylguaiacol (RT 15.5).**

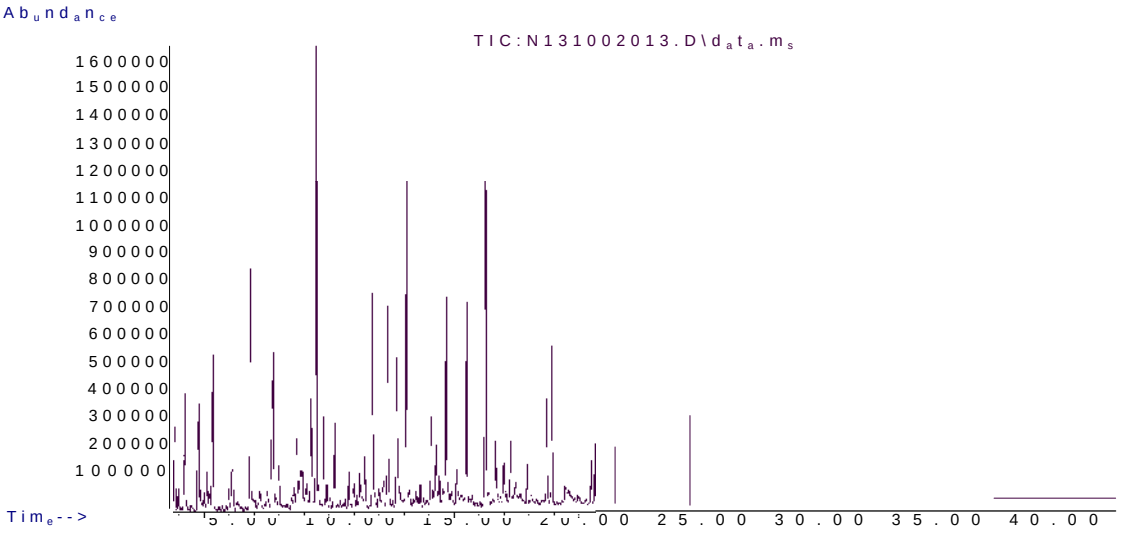



**Figure 3.15 GC-chromatogram of sample 12F056-T1-03.**

**The main peaks in sample 12F056-T1-03 were: guaiacol (RT 9.5), syringol (RT 16.2), 2,3-Dihydrobenzofuran (RT 13.1) and phenol (RT 6.8).**

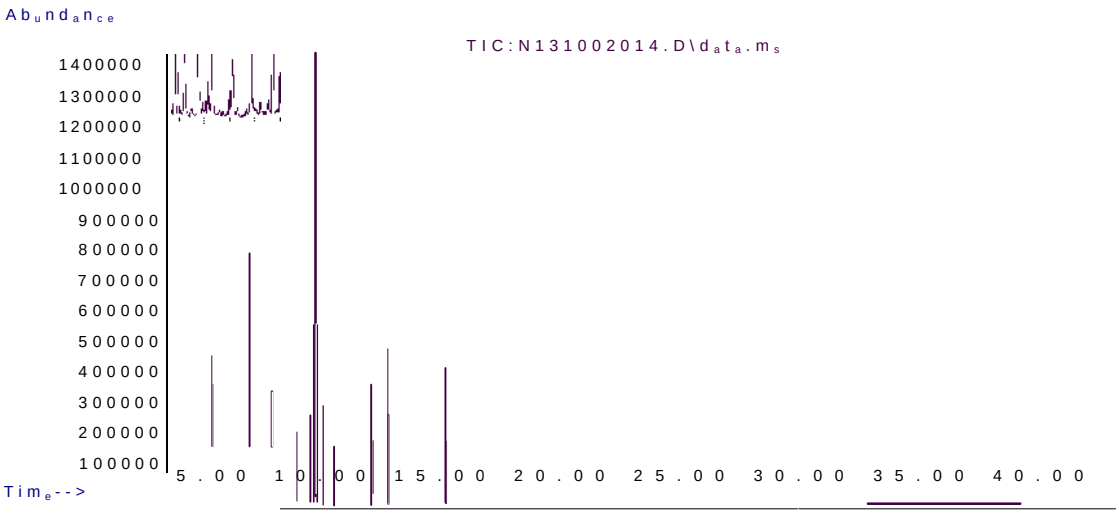



**Figure 3.16 GC-chromatogram of sample 12F056-T1-04.**

The main peaks in sample 12F056-T1-04 were: guaiacol (RT 9.5), phenol (RT 6.8) and syringol (RT 16.2).

### Supercritical CO2 fractionation

Supercritical CO2 fractionation was carried out using a Thar SFE 500 system (Thar technologies). Figure 3.17 shows the system that was used for the carbon dioxide fractionation study.

A mixture of 10 g bio-oil and 70 g of celite was loaded into the 500 mL extractor. The CO2 was supplied from a cylinder as a liquid and was maintained in this state through cooling unit (273 K) to avoid cavitation in the high pressure pump. The liquefied CO2 was then sent to the pre-heater and converted into a supercritical fluid prior to entering the extractor. Temperature (323 K), pressure (75 - 350 bar) and flow rate of 40 mL.min$^{-1}$ were selected according to the experiment.

The fractions were all collected at 308 K and at atmospheric pressure in separator 1.

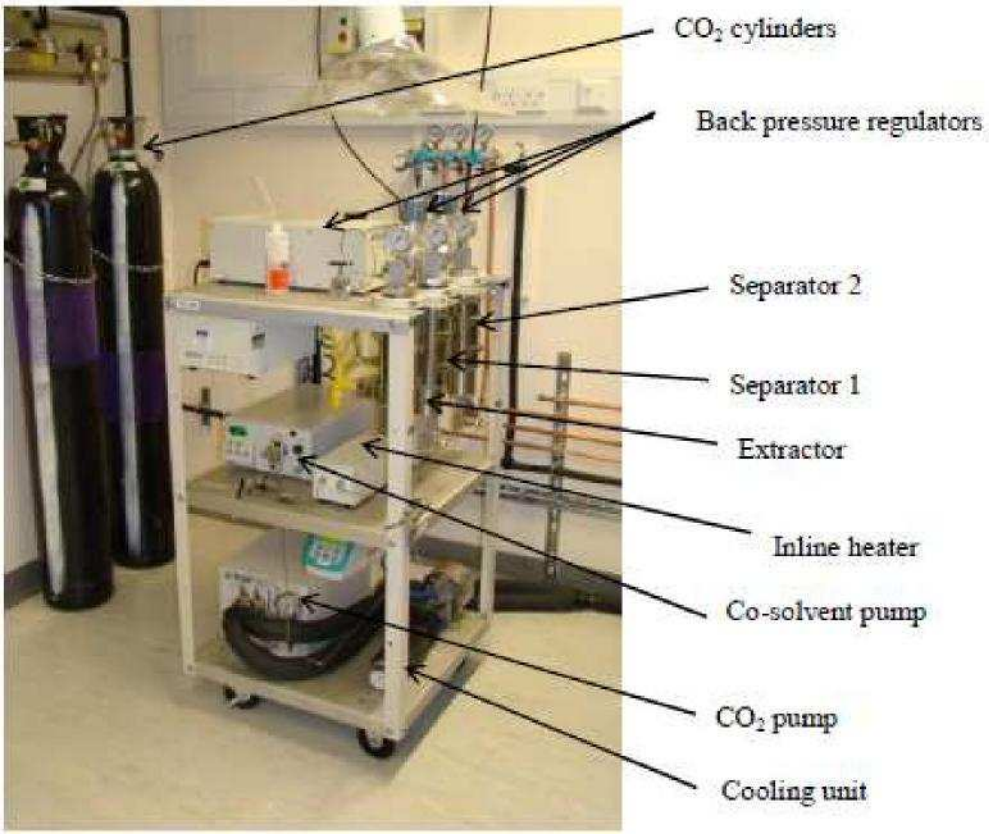
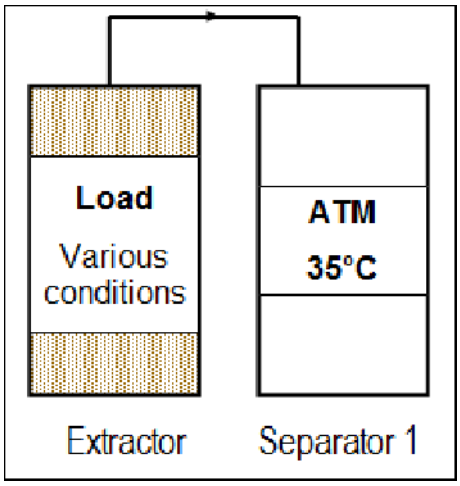



**Figure 3.17 - Thar SFE 500 system used for carbone dioxide extractions Step by step fractionation**

Each fractionation lasted one hour. The extractor compartment was set under different pressures, in the following order: 75 bar, 150 bar, 250 bar and 350 bar as detailed on Figure.

Once completed, the system was depressurised and the collecting compartment was washed with a minimal amount of dichloromethane before launching the experiment with the following conditions of work.

The dichloromethane was removed *in vacuo* and extracts were air-dried at room temperature until constant weight was achieved.




**Run 1 (N1)(75 bar): 22% of the total bio-oil collected.**

**Run 2 (N3)(150 bar): 23% of the total bio-oil collected.**

**Run 3 (N4)(250 bar): 24% of the total bio-oil collected.**

**Run 4 (N5)(350 bar): 18% of the total bio-oil collected.**

**Amount remaining on solid: 13% of the total bio-oil collected Figure 3.18 - Step by step fractionation**

**Table 3.9 Samples analysed**

| Sample ID | Sample Name | Fractionation method | Remarks |
|---|---|---|---|
| 12F056-T1-06 | Fraction N1 Andy $ScCO_2$ | Supercritical $CO_2$ | Solid |
| 12F056-T1-07 | Fraction N3 Andy $ScCO_2$ | Supercritical $CO_2$ | Solid |
| 12F056-T1-08 | Fraction N4 Andy $ScCO_2$ | Supercritical $CO_2$ | Solid |
| 12F056-T1-09 | Fraction N5 Andy $ScCO_2$ | Supercritical $CO_2$ | Solid |

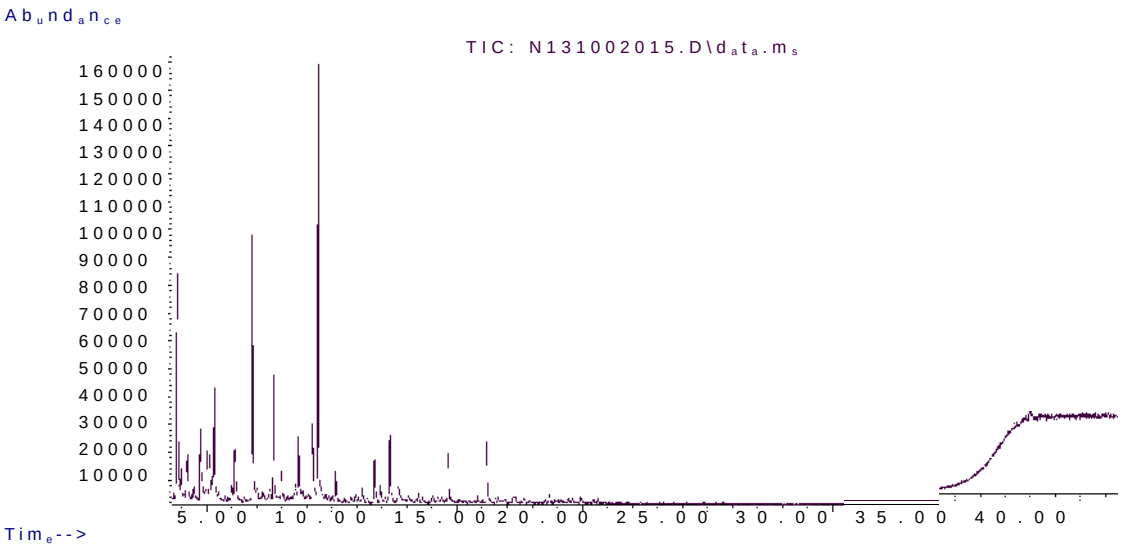



**Figure 3.8 GC-chromatogram of sample 12F056-T1-05.**

**Sample 12F056-T1-05 had a similar composition as sample 12F05-T1-04.**

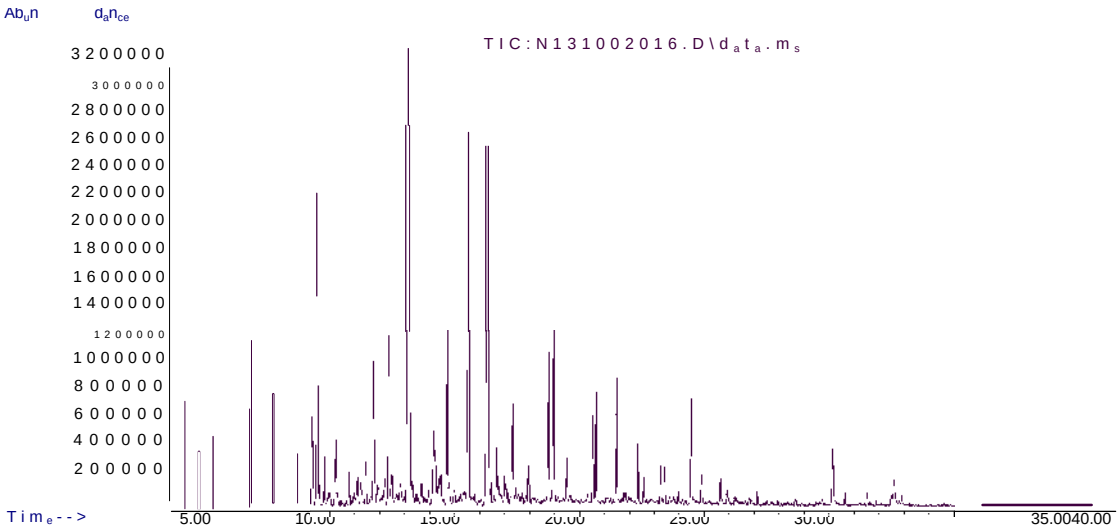



**Figure 3.9 GC-chromatogram of sample 12F056-T1-06.**

The main peaks in sample 12F056-T1-06 were: 2,3-Dihydrobenzofuran (RT 13.1), syringol (RT 16.2), vinylguaiacol (RT 15.5) and guaiacol (RT 9.5).

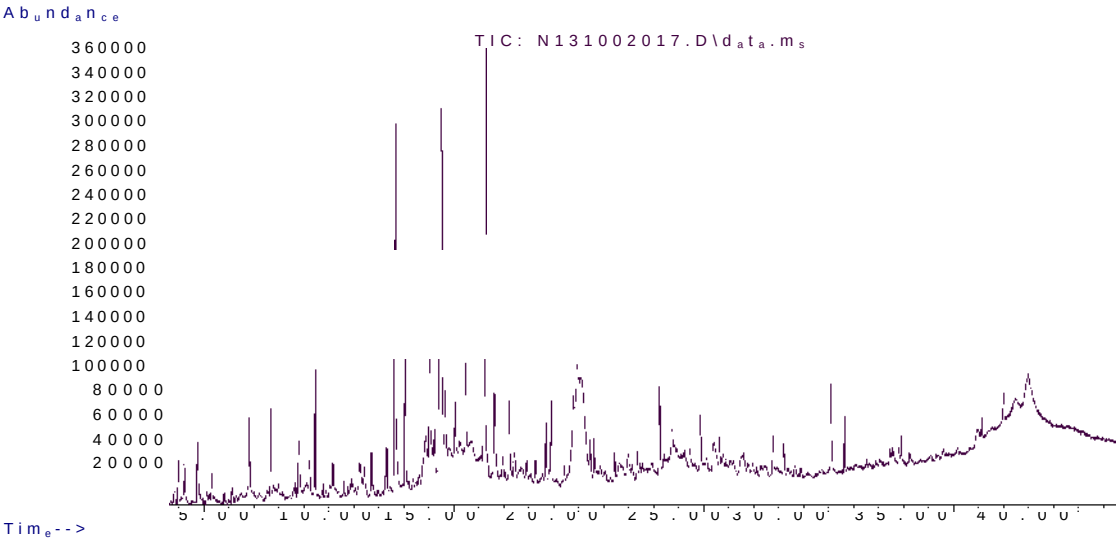



**Figure 3.10 GC-chromatogram of sample 12F056-T1-07.**

The main peaks in sample 12F056-T1-07 were: syringol (RT 16.2), 1,4-benzene diol (RT 14.5), catechol (RT 12.7) and 2,3-Dihydrobenzofuran (RT 13.1). In addition the broad peak at around RT 20 minutes is levoglucosan, a pyrolysis product from cellulose.

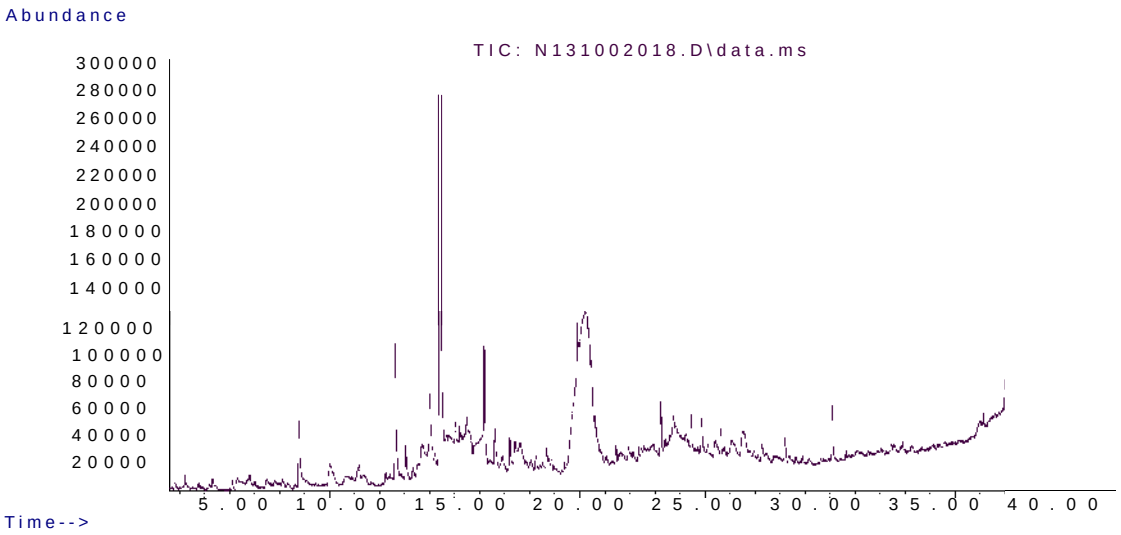



**Figure 3.11 GC-chromatogram of sample 12F056-T1-08.**

Sample 12F056-T1-08 contained mostly levoglucosan and 1,4-benzene diol (RT 14.5).

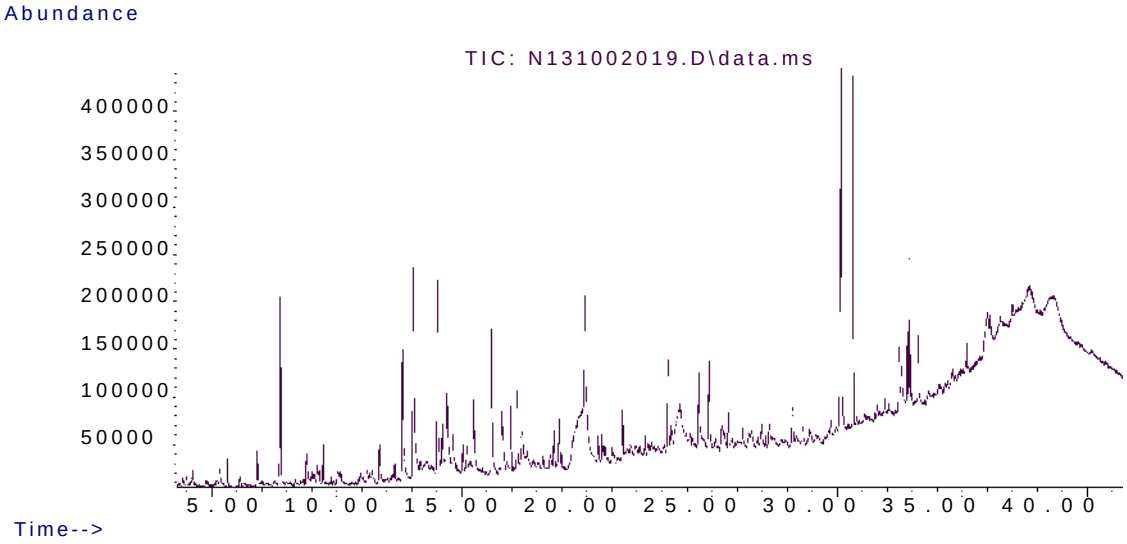



**Figure 3.12 GC-chromatogram of sample 12F056-T1-09.**

Sample 12F056-T1-09 contained levoglucosan and the highest peak was probably palmitic acid followed by a hydrocarbon (RT 30.1).

### 3.4 Further processing of the samples.

None of the samples were tested any further for reactivity in BAS oxidative decomposition process. There would be little gain in further trying to decompose these samples as they were already quite decomposed. Further fractionation in BAS process was not tested as the samples were too inhomogenous; the end result would still only be a complex mixture of compounds.

# 4 Experimental details

| Parameter | GCMS method |
|---|---|
| Column | DB-1MS (30 m, 0.25 mm ID, 0.25 µm film, column no |
| Temperature program | 417) 70°C, hold for 3 min, 5°C/min to 200°C, hold for 10 min, 50°C/min to 250°C, hold for 4 min |
| Injector temperature | 250°C |
| Injection volume | 1 µl |
| Split | 1:200 |
| Carrier gas | Helium, 1 ml/min |
| GCMS interface | 150°C |
| Detector | MS, EI 70 eV, scan range m/z 45-400 |

# 5 Mass and Energy balance of the Miscanthus MW processing and economic considerations

Aqueous fraction (19%) bio-oil (21% of yield), bio-char (36% of yield) and 24% of gas. Pyrolysis temperature 130-180 $^{o}$C. Water contents = 7%

For 1 ton of Miscanthus

| **Drying Phase (20 to 130 C); 1000kg; AT= 110$^{o}$C** | | | | |
|---|---|---|---|---|
| ***Components*** | ***Mass (kg)*** | ***Cp (kJ/kg.K)*** | ***Evaporation Energy kJ/kg*** | **Energy Required mJ** |
| ***Heat water*** | ***70*** | ***4.184*** | ***0*** | ***32.2*** |
| ***Heat Wood*** | ***930*** | ***1.59*** | ***0*** | ***162.7*** |
| ***Evaporate water*** | ***70*** | ***0*** | ***2261*** | ***158.3*** |
| | | | ***ΣE=*** | ***353.2*** |

| **Low Temp Pyrolysis Phase (130 - 200 C); 930kg; AT= 70$^{o}$C** | | | | |
|---|---|---|---|---|
| ***Components*** | ***Mass (kg)*** | ***Cp (kJ/kg.K)*** | ***Evaporation Energy kJ/kg*** | **Energy Required mJ** |
| ***Heat char*** | ***360*** | ***1.62*** | ***0*** | ***40.8*** |
| ***Evaporate produced water*** | ***120*** | ***0*** | ***2261*** | ***271.3*** |
| ***Heat produced organics*** | ***210*** | ***1.38*** | ***0*** | ***60.9*** |
| ***Evaporate produced organic*** | ***210*** | ***0*** | ***371*** | ***77.9*** |

| | |
|---|---|
| ***ΣE=*** | ***450.9*** |

***Total Energy consumption: 804 mJ/ton***

***Energy Required: 450.9 mJ***

- ***Energy which could be producedfrom 1ton of Miscanthus: 18000mJ(18kj/g-calorific value of Miscanthus)***
- ***Balance: 18000-450mJ=1350mJ***
- ***We can check energy cost and it give an estimation about our profit (without labour). Based on a figure of 3.6mJ=14.5p, 1000 Kg of Miscanthus could bring 14.5*1350/3.6=£5.4 profit if we will used products as a fuel.***

***Cost: European Market Study for BioOil (Pyrolysis Oil) Dec 15, 2006:***

- ***Fig 3.7 Oil price- £451/ton; 21% oil yield = 0.21*451=$94/per 1 ton of Miscanthus***
  - ***Coal price= $70/ton; 36% yield: 0.36*70=$25.2/per 1 ton of Miscanthus***
  - ***Total: $119.2 (£80) per 1000 Kg of Miscanthus***
- ***Energy Required: 450.9 mJ, 14.5*450.9/3.6= £18 per 1000 Kg of Miscanthus.***
- ***Total profit: £80-18 = £62 per 1000 Kg of Miscanthus***

Current market price of Miscanthus is estimated to be £30-40 per 1000 Kg.
This could increase the profitability by 100% for growers.

# 6 Conclusions

The samples mainly contained different phenols of lignin origin and carbohydrate derived pyrolysis products. Also polyaromatic compounds and fatty acids could be found. No component was found to be of unique enough character and high enough concentration to justify any further separation.

The analysis shows that the complex starting materials give mainly complex product mixtures. The samples analysed here are not significantly different from other pyrolysis samples we have analysed.

The use of microwave pyrolysis is an effective tool for generating higher value products from lignin, however further technologies need to be developed to effectively separate molecules into commercially important fractions. The pyrolysis and fractionation techniques used for these samples were not able to fine tune the decomposition of the biomass into a product mixture which is commercially interesting. Both the pyrolysis and fractionation techniques need to be altered to provide fractions which are more homogenous in composition. Inhomogenous samples, such as these, will not give any more information than that the sample is a complex mixture and thus not commercially interesting.

# 7 Abbreviations

| | |
|---|---|
| RT. | Retention time in minutes. |
| BAS | Borregaard AS |