Thermal, chemical and morphological properties of carbon fibres derived from chemically pre-treated wool fibres

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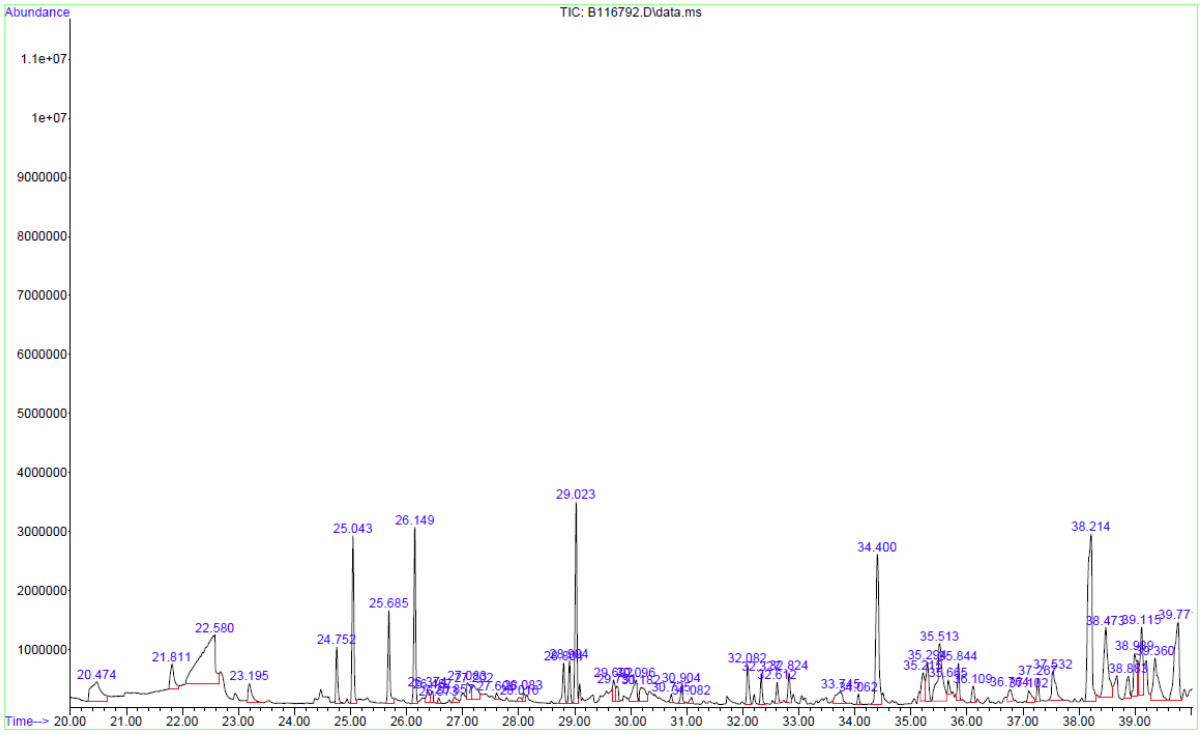
Py-GC-MS analysis of volatiles evolved during pyrolysis of wool fibre at 300 to 400 °C and 400 to 600 °C.

Py-GC-MS Procedure

Approximately 1 mg of wool fibre was placed with clean tweezers into a quartz pyrolysis tube (CDS Analytical, pre-cleaned by heating at 800 °C) plugged with deactivated quartz wool. Pyrolysis was performed using a CDS Analytical Pyroprobe 5250. The sample was pyrolysed in a helium atmosphere with a temperature programme increasing from 300 to 400 °C at 10 °C/min. Products flowed through a transfer line (with He as the carrier gas) into an Agilent HP 6890 GC coupled to an Agilent HP 5973 MSD. The GC column was an Agilent DB5-MSI (60m long, 0.25 mm I.D., 0.25 μm film thickness). The GC oven was held at -40 °C for 12 minutes after the pyrolysis temperature programme started to cryogenically focus the products onto the column. GC oven temperature was then increased to 40 °C at 8 °C/min, then to 310 °C at 4 °C/min, then held at 310 °C for 5 minutes. Helium was used as the carrier gas at a constant flow rate of 1.2 mL/min. The split ratio was 30:1. Mass spectra were acquired in full scan mode (*m/z* 15 to 600) with ionisation energy of 70 eV. Once the pyrolysis temperature programme had completed, the sample was ejected from the pyrolysis unit and immediately sealed in a glass vial to limit exposure to atmosphere. The procedure above was repeated on the sample, with a pyrolysis temperature program increased from 400 to 600 °C at 10 °C/min. The GC oven was held at -40 °C for 22 minutes to cryogenically focus products, and held at the final temperature of 310 °C for 25 minutes. All other experimental parameters were identical.

Results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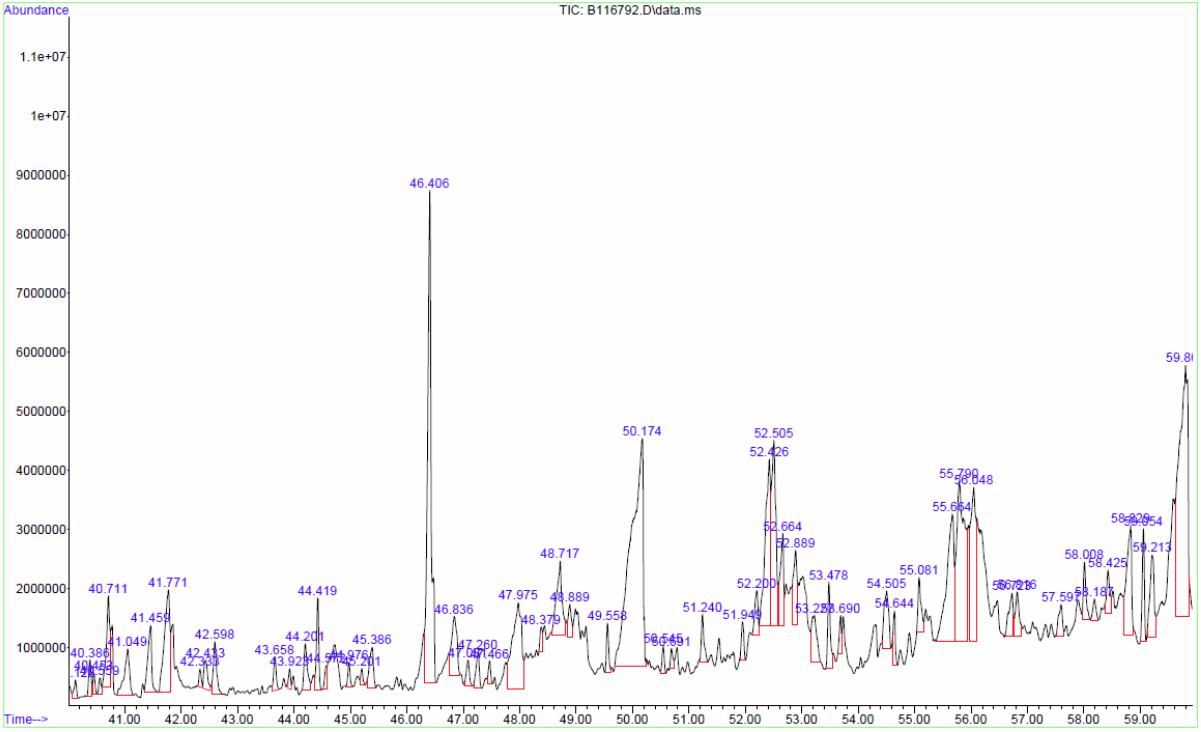
26.149
29.023
32.082
34.400
35.513
38.214
39.115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47.975
48.379
48.717
48.889
49.558
50.174
51.240
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52.200
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52.505
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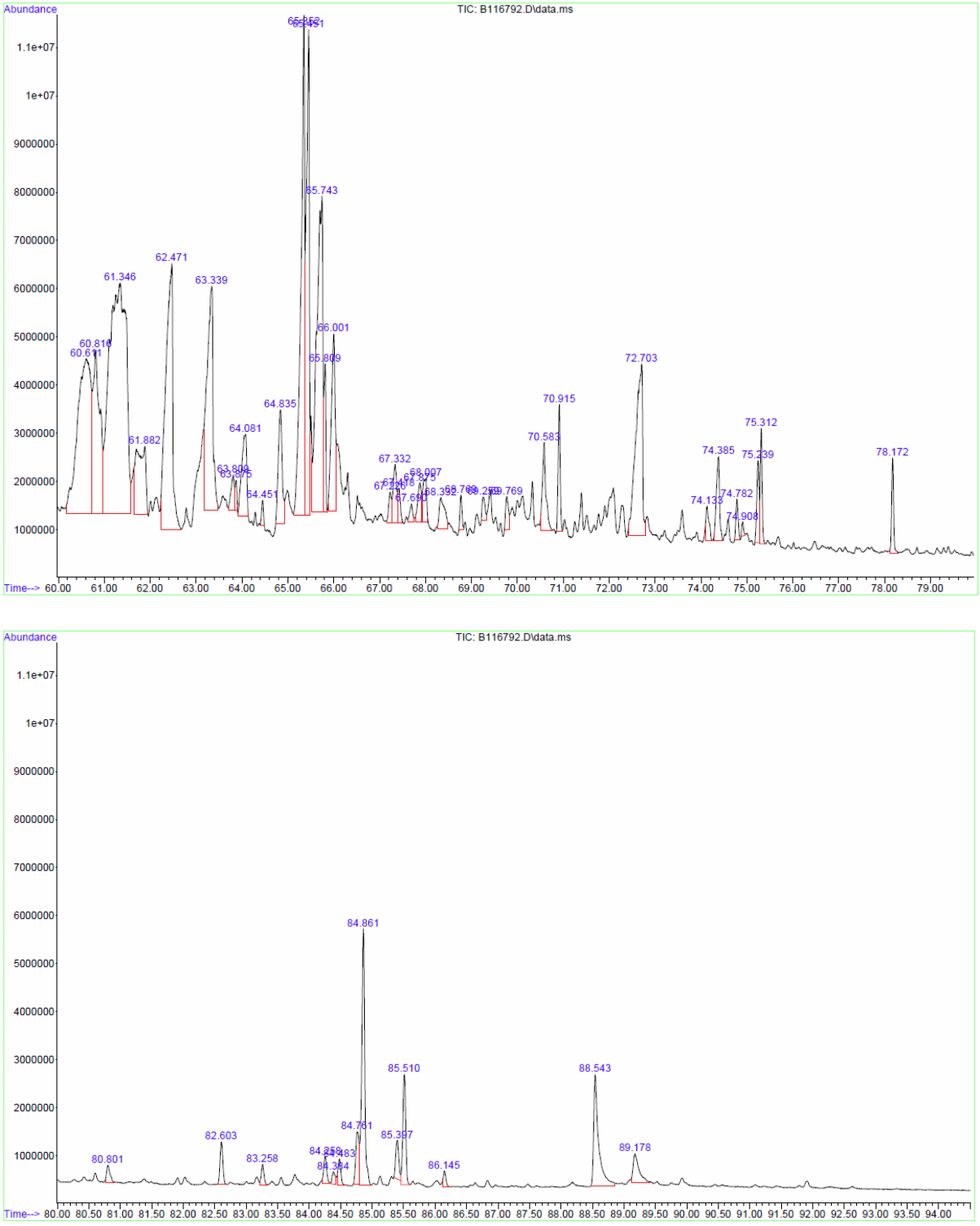
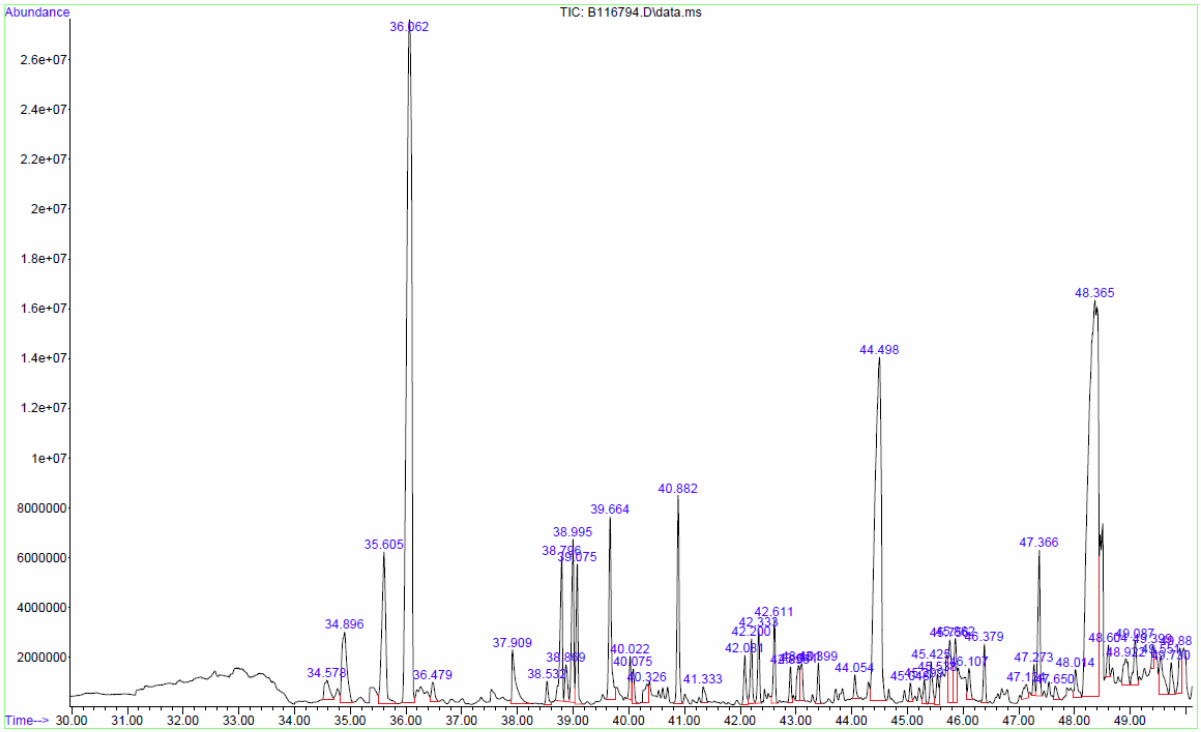


Figure S1. Pyrochromatogram of wool fibre pyrolysed at 300-400 °C at 10 °C/min under helium atmosphere.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42.611
42.333
42.200
40.022
40.075
34.578
36.479
38.533
40.326
41.333
44.054
46.379
47.273
48.014
Time-->
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31.00
32.00
33.00
34.00
35.00
36.00
37.00
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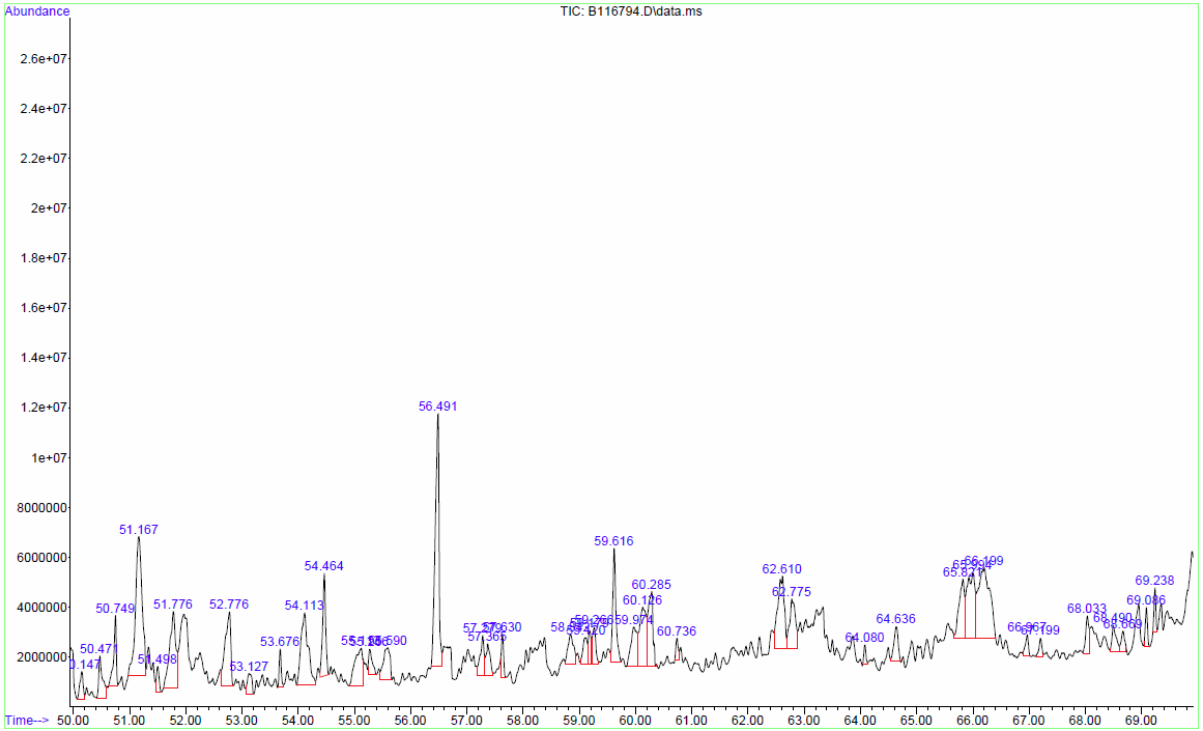
49.00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52.776
54.113
53.676
64.636
68.033
69.238
50.471
51.498
53.127
60.736
64.080
Time-->
50.00
51.00
52.00
53.00
54.00
55.00
56.00
57.00
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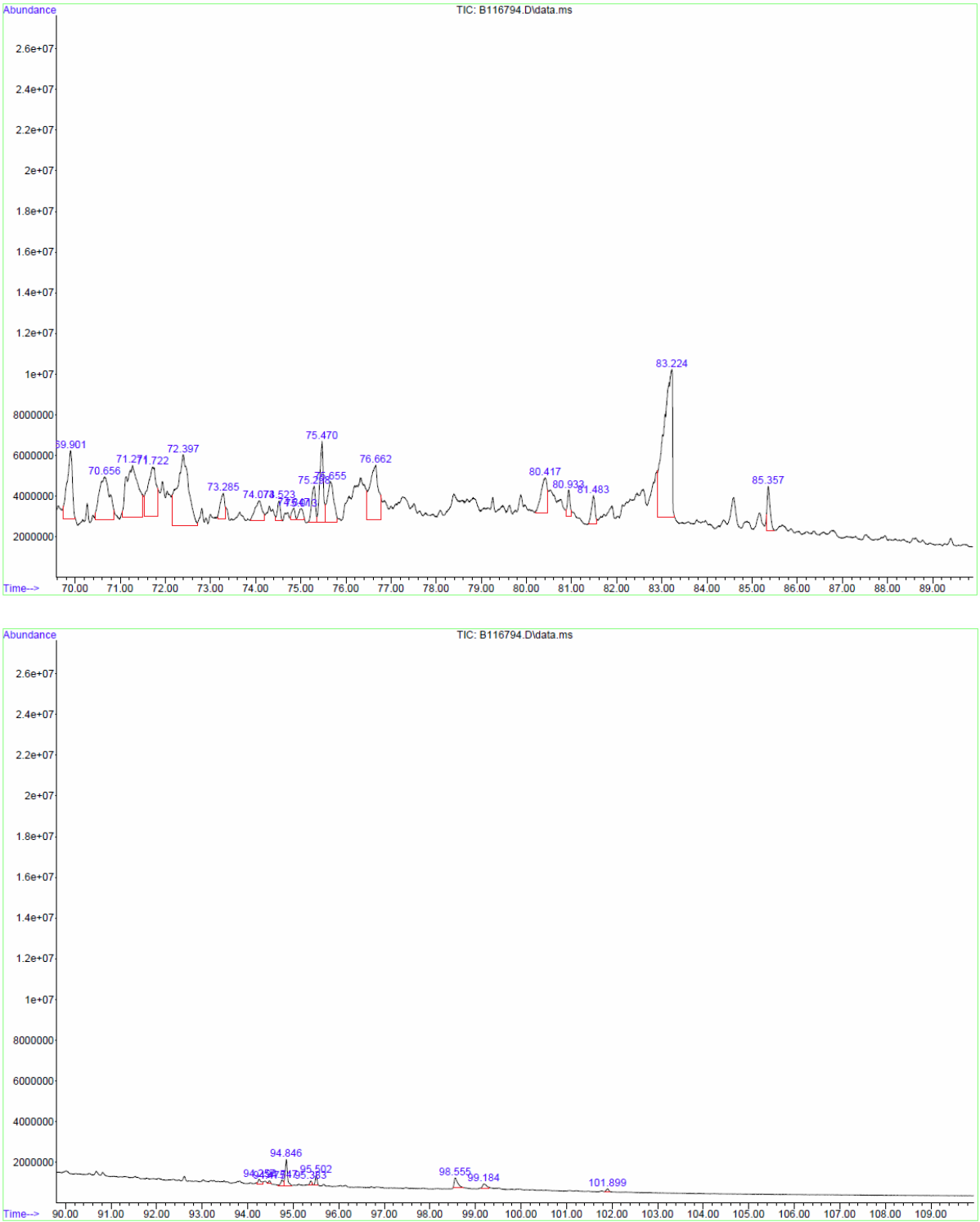

Figure S2. Pyrochromatogram of wool fibre pyrolysed at 400-600 °C at 10 °C/min under helium atmosphere.

Table S1. Retention time and the percentage of peak areas of the pyrolysis products obtained from wool fibre at 300 to 400 °C.

Retention time (min)	Compounds	% of peak area
20.474	Propanenitrile	0.32
21.811	Isobutyronitrile	0.14
22.58	Acetic acid	1.18
23.195	Butanenitrile	0.12
24.752	2-methyl butanenitrile	0.17
25.043	3-methyl butanenitrile	0.48
25.685	Pyrrole	0.28
26.149	Toluene	0.49
27.083	Acetamide	0.10
28.804	2-methyl 1H-pyrrole	0.13
29.023	4-methyl pentanenitrile	0.56
30.096	2-methyl butanoic acid	0.14
30.182	Propanamide	0.13
32.082	Dimethyl 1H-pyrrole	0.14
32.824	3-buten-2-ol	0.10
33.745	4-methyl pentanoic acid	0.13
34.4	Phenol	0.72
35.215	Methyl butanamide	0.14
35.294	Methyl butanamide	0.15
35.513	Aminopyridine	0.47
35.844	Trimethylpyrrole	0.1
37.532	Amino methylpyridine	0.2
38.214	4-methyl phenol	1.12
38.473	Imidazole-4-carboxamide	0.48
38.989	3-methyl 2-pyridinamine	0.21
39.115	Unknown	0.32
39.36	3-methyl butanamide	0.34
39.771	Pentanamide	0.58
40.387	Unknown	0.14
40.711	Benzyl nitrile	0.48
41.459	2,6-piperidinedione	0.41
41.771	Dimethyl pyrrole dione	0.92
42.433	2-Piperidinone	0.17
42.598	2,6-piperidinedione	0.28
44.201	Unknown	0.25
44.419	Benzenepropanenitrile	0.33
45.386	Unknown (possibly dimethyl pyrrolinone)	0.25
46.406	Indole	2.86
46.836	Pyrrole-2-carboxamide	0.5
47.26	Unknown	0.19
47.975	Unknown	1.26
48.717	Unknown	0.64
50.174	Unknown	4.33

Table S1. Continued

Retention time (min)	Compounds	% of peak area
51.24	Unknown	0.22
52.426	Unknown	1.33
52.505	Unknown	1.27
52.664	Unknown	0.46
52.889	Unknown	0.43
53.227	Unknown	0.39
53.478	Unknown	0.35
53.69	N-Methyl-O-methyltyrosine methyl ester	0.13
54.505	Unknown	0.42
54.644	Unknown	0.17
55.081	Unknown	0.27
55.664	Unknown	1.48
55.79	Unknown	2.06
56.048	Unknown	1.21
56.723	1-Methyl-3-(N-methylethanamine)-indole	0.28
56.816	Unknown	0.26
58.829	Unknown	0.85
59.213	Unknown	0.56
59.803	Unknown	3.42
60.611	Unknown	4.92
60.816	Tetraethyl pyrazine	2.62
61.346	Unknown	9.45
61.882	Unknown	1.49
62.471	Unknown	4.42
63.339	3,6-Diisobutylpiperazine-2,5-dione	3.29
64.835	Unknown	1.04
65.352	Dimer of pyrrolidinedione	4.29
65.451	Unknown	3.56
65.743	Unknown	4.54
65.809	N-Methyl-N-methyltryptophan methyl ester	0.65
66.001	Hexadecanoic acid	1.65
67.332	Unknown	0.48
67.875	Unknown	0.32
68.007	Unknown	0.39
68.332	Unknown	0.40
69.769	Unknown	0.23
70.583	Octadecanoic acid	0.72
70.915	Hexadecanamide	0.63
72.703	Unknown	2.84
74.385	Unknown	0.64
75.312	Octadecanamide	0.50
78.172	1,2-benzenedicarboxylic acid ester	0.46
84.761	Sterene	0.29
84.861	Sterene	1.36
85.51	Sterene	0.55

Table S1. Continued

Retention time (min)	Compounds	% of peak area
88.543	Sterol	0.95
89.178	Sterol	0.29

Table S2. Retention time and the percentage of peak areas of the pyrolysis products obtained from wool fibre at 400 to 600 °C.

Retention time (min)	Compounds	% of peak area
34.578	2-methyl butanenitrile	0.30
34.896	3-methyl butanenitrile	0.99
35.605	Pyrrole	1.65
36.062	Toluene	9.79
36.479	Unknown	0.18
37.909	Methyl pyridine	0.59
38.796	2-methyl 1H-pyrrole	0.96
38.869	Unknown	0.27
38.995	4-methyl pentanenitrile	1.00
39.075	3-methyl 1H-pyrrole	0.74
39.664	Ethylbenzene	1.25
40.022	m-xylene	0.24
40.075	p-xylene	0.19
40.326	Dimethyl thiophene	0.21
40.882	Styrene	1.20
41.333	Ethyl pyridine	0.13
42.081	Dimethyl 1H-pyrrole	0.31
42.2	Ethyl 1H-pyrrole	0.38
42.333	Dimethyl 1H-pyrrole	0.40
42.611	Phenylalanine methyl ester	0.44
42.896	Ethyl 1H-pyrrole	0.18
43.041	3-Methyl-4(-N-methylethanimine)-imidazole	0.28
43.101	Dimethyl 1H-pyrrole	0.24
44.498	Phenol	5.97
45.048	Trimethylbenzene	0.10
45.425	Ethyl methyl 1H-pyrrole	0.30
45.756	Unknown	0.52
45.862	Trimethyl 1H-pyrrole	0.51
46.107	Ethyl methyl 1H-pyrrole	0.20
47.134	Unknown	0.17
47.366	2-methylphenol	0.89
48.014	2-Amino-4-methylpyrimidine	0.21
48.365	4-methylphenol	10.83
49.087	2-Pyridinamine, 3-methyl-	0.38
49.551	Unknown (possibly imethylphenol)	0.43
49.73	2-Pyridinamine, 5-methyl-	0.23

Table S2. Continued.

Retention time (min)	Compounds	% of peak area
49.889	2-Pyridinamine, 6-methyl-	0.38
50.147	Unknown	0.25
50.471	2-pyrimidinamine, 4,6-dimethyl-	0.43
50.749	Benzyl nitrile	0.52
51.167	Dimethylphenol	2.53
51.776	2,6-Piperidinedione	1.32
52.776	Unknown (possibly 2-Piperidinone)	1.13
54.113	Unknown	1.42
54.464	Benzenepropanenitrile	0.85
55.113	Picolinamide	0.77
55.266	4-Propylphenol	0.25
55.59	Unknown	0.58
56.491	Indole	2.68
57.279	Unknown	0.41
58.842	Unknown	0.48
59.616	Methyl indole	1.01
59.974	Unknown	0.68
60.126	Unknown	1.06
60.285	Unknown	1.00
62.61	3,6-Diisobutyl-1-methylpiperazine-2,5-dione	1.31
62.775	Dimer of pyrrolidinedione	0.81
65.821	Unknown	0.89
65.994	Unknown	1.48
66.199	N-Methyltryptophan methyl ester	2.27
69.901	Unknown	1.52
70.656	Unknown	1.91
71.271	Unknown	2.65
71.722	Unknown	1.77
72.397	Unknown	3.45
73.285	Unknown	0.47
75.298	Unknown	0.56
75.47	Unknown	1.22
75.655	Unknown	1.06
76.662	Unknown	1.93
80.417	Unknown	0.89
83.224	Unknown	5.61
85.357	Unknown	0.53
94.846	Sterene	0.24
98.555	Sterol	0.15