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Conventional and microwave-assisted pyrolysis of biomass under different heating rates

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Abstract: Biomass was subjected to conventional and microwave pyrolysis, to determine the influence of each process on the yield and composition of the derived gas, oil and char products. The influence of pyrolysis temperature and heating rate for the conventional pyrolysis and the microwave power was investigated. Two major stages of gas release were observed during biomass pyrolysis, the first being CO/CO_2 and the second one CH_4/H_2. This two-stage gas release was much more obvious for the conventional pyrolysis. While similar yield of liquid was obtained for both cases of conventional and microwave pyrolysis (~46 wt.%), higher gas yield was produced for the conventional pyrolysis; it is suggested that microwave pyrolysis is much faster. When the heating rate was increased, the peak release of CO and CO_2 was moved to higher reaction temperature for both conventional (500 °C) and microwave pyrolysis (200 °C). The production of CH_4 and H_2 were very low at a conventional pyrolysis temperature of 310 °C and microwave pyrolysis temperature of 200 °C (600 and 900 W). However, at higher heating rate of microwave pyrolysis, clear release of CH_4 was observed. This work tentatively demonstrates possible connections and difference for biomass pyrolysis using two different heating resources (conventional and microwave heating).

Key words: Biomass; Pyrolysis; Conventional; Microwave

1. Introduction

The European Union has introduced several sustainability initiatives to reduce the emission of greenhouse gases, to increase the use of renewable resources, support a more efficient Europe and to create a low carbon economy [1, 2]. Key to this strategy is the need for an increased use of renewable sources such as biomass. Because biomass is carbon neutral, there is increasing interest in the use of biomass to replace fossil fuels and thereby reduce the impacts of climate change. Thermochemical routes to utilise the potential of biomass include pyrolysis which, can generate useful end-products of bio-oil, syngas and bio-char. The bio-oil has a higher energy content per unit mass, than the raw biomass and can be used directly in fuel applications or as refinery feedstock or with upgrading to produce refined fuels and chemicals. The solid char can be used as a solid fuel, alternatively the char can be used as bio-char for soil improvement, upgraded to activated carbon, or gasified to produce syngas. The gases generated have medium to high calorific values and may contain sufficient energy to supply the energy requirements of a pyrolysis plant.

Heating the biomass via an external heating source has been by far the most studied technology. The advantage of pyrolysis is that the yield of end-products can be altered depending on the process conditions; the temperature of pyrolysis and the heating rate are known to have most influence [3-6]. High char yields are obtained from biomass via slow heating rates to moderate temperatures of ~ 400 °C. Moderate heating rates in the range of about 10 °C min^{-1} and maximum temperatures of 600 °C gives an approximate equal distribution of oils, char and gases referred to as conventional pyrolysis [5]. Fast pyrolysis involves high heating rates (~100 °C/s) and pyrolysis temperatures below 650°C and coupled with rapid quenching of the pyrolysis products produces a mainly liquid product [6]. High pyrolysis temperatures result in the thermal cracking of the oil to produce a high gas yield.

In comparison to conventional heating, microwave pyrolysis involves transfer of energy to the biomass through the interaction of the molecules within the biomass [10-12]. Heating of biomass occurs when the microwaves cause dipolar molecules to attempt to rotate in phase with the alternating field of the microwaves [7]. Microwave heating has the advantages that heat is generated within the material rather than from an external source, resulting in of rapid heating and high heating efficiency [3,7]. Microwave pyrolysis has been applied to a range of materials, including biomass [8] and waste tyres and plastics [9]. However, biomass does not readily absorb microwave radiation and initially tyre biomass may be added to a transparent material such as carbon or water. Once pyrolysis progresses, the product carbonaceous char facilitates the microwave pyrolysis.

There are few direct comparisons between conventional and microwave pyrolysis of biomass [13-15]. Obtaining information about connections/differences between these two technologies will facilitate the development of biomass pyrolysis, e.g. using a combination of conventional and microwave heating methods. In this work, the same biomass sample was used in two types of reaction systems (conventional and microwave pyrolysis). In each type of pyrolysis, different heating rates were used; the changing trend of experimental results (product distribution, gas release and char content) derived from different heating rates were explored for both conventional and microwave pyrolysis.

2. Experimental

2.1 Materials

Wood biomass (average size 1.5mm) was used for both conventional and microwave pyrolysis. A Shimadzu TGA-50 thermogravimetric analyser was used to determine the proximate analysis of the biomass samples and elemental analysis was carried out using a Carlo Erba Flash EA 11112 elemental analyser. The biomass moisture, volatile, fixed carbon and ash contents were 6.4, 74.8, 18.3 and 1.2 wt.% respectively. The carbon content was 36.17 wt%, hydrogen 6.30 wt%, nitrogen 1.78 wt% and the oxygen calculated by difference was 36.17 wt%.

2.2 Conventional pyrolysis of biomass

Conventional pyrolysis was carried out using a fixed bed stainless steel reactor of 16 cm length and internal diameter of 2.2 cm, heated externally by an electrical furnace and continually purged with nitrogen. Approximately 3 g of biomass was mixed with water (0.3 g of H_2O was added on the top of the biomass sample) and placed in a sample boat in the reactor and heated to the desired pyrolysis temperatures using different heating rates (5, 20 and 40 °C min^{-1}). Water was added due to the microwave processing of biomass required addition of water to aid microwave absorption. The gaseous pyrolysis products were passed through an air cooled condenser and a dry ice cooled condenser to trap the product liquids. Non-condensed gases were passed into an on-line gas analyser allowing for the determination of CO, CO_2, H_2 and CH_4. N_2 was used as the carrier gas with a flow rate of 80 ml min^{-1}. The conventional reaction system has been reported previously [16].

2.3 Microwave pyrolysis

Microwave pyrolysis was conducted using samples of 140 g of biomass mixed with about 14 g water. The samples were heated to 200 °C with different fixed microwave powers (600, 900 and 1200 W). It is noted that the heating rate is around 8, 15 and 20 °C min^{-1}, for microwave power of 600, 900 and 1200 W, respectively. For this purpose a Milestone ROTO Synth Rotative Solid Phase Microwave Reactor (Milestone Srl.) was used with an operating MW frequency of 2.45 GHz.

A schematic diagram of the microwave pyrolysis reactor system can be found in previous work [14]. Samples of the biomass were placed in 2 L glass flask within the cavity of the microwave. The temperature of the solid biomass was measured using an infra-red detector within the reactor cavity. In addition the temperature of the evolved volatile fractions was measured using a thermocouple on the exit tube. Both temperature measurements were within 15 °C of each other [14]. The generated liquid products were condensed in a water cooled vacuum trap using several consecutive flasks

The non-condensable gases from the microwave pyrolysis system were collected using a 5 L Tedlar^{TM} gas bag, which was analysed by two separate gas chromatographs. H_2, CO, O_2, N_2 and CO_2 were analysed by a Varian 3380 GC with two packed columns and with two thermal conductivity detectors (GC/TCD). Hydrogen, oxygen and carbon monoxide, methane and nitrogen were analysed on a 2m length by 2mm diameter column, packed with 60-80 mesh molecular sieve. Argon was used as the carrier gas. Carbon dioxide was analysed on a separate 2m length by 2mm diameter column with Haysep 80-100 mesh packing material. C_1 to C_4 hydrocarbons were analysed using a second Varian 3380 gas chromatograph with a flame ionisation detector, with a 80-100 mesh Hysep column and nitrogen carrier gas.

2.4. Oil analysis

Prior to analysis the liquid pyrolysis products from both the conventional and microwave experiments were dried using anhydrous sodium sulphate and diluted with dichloromethane (DCM). They were analysed by coupled gas chromatography (GC) – mass spectrometry (GC-MS) using a Varian CP-3800 GC coupled to a Varian Saturn 2200 mass spectrometer (MS). Detailed conditions for the GC-MS analysis of the oil fraction can be obtained from a previous report [17].

2.5. TGA-FTIR analysis

In addition to the conventional and microwave pyrolysis experiments, a Stanton-Redcroft thermogravimetric analyser (TGA) coupled to an FTIR analyser (Thermo Scientific iS10), was used to examine the release of gas products. Around 25 mg of biomass sample was pyrolysed in the TGA under nitrogen conditions.

3. Results and discussion

3.1 Effect of the heating rate on the product distribution

Table 1, shows the mass balances of the pyrolysis of biomass carried out using the conventional and microwave pyrolysis units. Direct comparison of the product yield between the two systems is difficult because of the nature of the size and configuration of the reactors in addition to the different heating systems. However, general trends in regard to heating rate for the conventional pyrolysis or microwave power which corresponds to heating rate can be drawn. From Table 1, the liquid (bio-oil) yield shows a small increase with the applied heating rate or microwave power. It can be seen from the data that with conventional pyrolysis an increase of the amount of liquids from 53.94 to 58.18 wt. % is observed with increasing heating rate, while in the case of microwave pyrolysis liquid yield increases from 43.23 to 47.10 wt. % with increasing microwave power. This is in agreement with other reports by Isahak et al. [18] and Mohan et al. [19]; they proposed the high yield of liquid is due to cracking reactions which are promoted at a higher heating rate.

Table 1 also shows that markedly lower char production is obtained when conventional pyrolysis (~23 wt.%) is used to pyrolyse biomass compared to microwave pyrolysis (~ 45 wt.%). It should be noted that the microwave pyrolysis was conducted at 200 °C. The conventional pyrolysis carried out at the lower temperature of 310 °C, showed that the liquid and gas yields were decreased appreciably in favour of char formation (up to 59.79 wt.%). It was found that conventional pyrolysis conducted at 350 °C under a heating rate of 20 °C min^{-1} gave a similar liquid yield (~46 wt.%) as the microwave-assisted pyrolysis at 900W. Interestingly, under these conditions the char and gas yields obtained with microwave pyrolysis were respectively significantly higher (~40 wt.%) and lower (~10 wt.%) compared to the conventionally performed experiment. This relates directly to the different mechanism of microwave heating as opposed to conventional heating. Indeed, while conventional heating occurs through convection [20,21], microwave heating is more volumetric, activating the biomass instantly as a whole [22]. This combined with the overall lower reaction temperature (200 - 350 °C) limits the probability of secondary cracking reactions responsible for creating gaseous products.

3.2 Effect of heating rate on gas release and concentration

The profile of gas release during the conventional pyrolysis and microwave pyrolysis are shown in Fig. 1 and Fig. 2, respectively. For conventional pyrolysis at a heating rate of 5 °C min^{-1}, the biomass starts to decompose at around 200 °C. The starting point of the decomposition was found to increase with the heating rate. At a heating rate of 40 °C min^{-1}, the decomposition temperature was around 350°C. Two stages of gas evolution can be discerned from Fig 1; the first being CO_2/CO formation and the second CH_4/H_2. With increased heating rate the maximum CO_2/CO evolution is found to shift towards higher temperatures. Indeed, in increasing the heating rate from 5 to 40 °C min^{-1}, the maximum CO_2/CO production is shifted from 330 to 480 °C. Interestingly, the occurrence of the CH_4/H_2 peak becomes quasi simultaneous with the CO_2/CO evolution at higher heating rates.

For a heating rate of 20 °C min^{-1}, a decrease of the final pyrolysis temperature from 520 to 310 °C, seems to spread the CO_2/CO evolution out in time. At these lower pyrolysis temperatures (310 °C) no evolution of CH_4 and/or H_2 can be detected. The evolution of the CO_2 and CO gas can be directly linked to the C-O and C=O functional groups, which are abundantly present in biomass [23]. Given the lower temperature at which these gases appear, they are likely to derive from the hemicellulose fraction, which is normally decomposed at lower temperature compared with other biomass components [24-26]. The formation of CH_4 and H_2 is linked to higher pyrolysis temperatures, exceeding markedly those needed for CO_2/CO production. This is consistent with the observations by Yang et al. [27]; they suggested that the CH_4/H_2 formation relates to secondary or primary reactions with a high activation energy.

Microwave pyrolysis follows the trend observed for conventional pyrolysis in that CO_2 and CO are the main gaseous products (Fig. 2). Increasing the power from 600 to 1200 W, thereby essentially increasing the heating rate, shifts the appearance of CO_2 and CO from below 60 °C to around 175 °C. As discussed above, the release of CH_4 and H_2 mainly occurs at higher reaction temperature (e.g. conventional pyrolysis at 310 °C shows negligible production of CH_4 and H_2, Figure 1). Herein, for the microwave pyrolysis, production of CH_4 can be clearly observed when the microwave power was increased to 900 or 1200 W. Therefore, release of CH_4 gas can be significantly promoted with higher microwave power due to promoted interactions of biomass molecules, while the reaction temperature was kept constant. There might be a potential interest in further exploring the production of CH_4 from microwave pyrolysis with high microwave power and at low reaction temperature.

In addition to the experimental conventional and microwave pyrolysis, the pyrolysis of smaller biomass samples (~15 mg) was also investigated using TGA-FTIR to determine the production of gas in relation to increasing temperature and also to determine the influence of heating rate (Fig. 3) . Using this technique, the convection of heat into the biomass particles is not a limiting step. While CO_2 and CO were again the main gaseous products, their evolution was found independent of the heating rate, typically occurring around 320 °C (Fig. 3). In addition, a second release of CO_2 is observed at ~600 °C. This release of CO_2 is not accompanied with CO gas, as was the case at lower temperatures. With reference to the work of Biagini et al. [28] suggested that this second CO_2 release is likely to be related to the pyrolysis of lignin, typically occurring with a low CO formation.

3.3 Effect of heating rate on char residue

The elemental analysis of the char residue derived from both conventional and microwave pyrolysis is shown in Table 2. Focussing on the data for which a similar liquid yield was obtained (i.e. conventional pyrolysis at 20 °C min^{-1} to 350 °C and microwave pyrolysis at 900W to 200 °C), it is found that the oxygen and carbon content of the chars are respectively lower and higher when the pyrolysis is performed conventionally as compared to microwave-pyrolysis This is in agreement with the higher gas yield, mainly CO_2 and CO thus high in oxygen content, when conventional pyrolysis is applied. Additionally, the C/H ratio of char is markedly lower in the case of microwave pyrolysis compared to conventional. As raw biomass has the lowest C/H ratio, this is consistent with a lower degree of biomass conversion when microwaves are applied.

Under conventional pyrolysis, and with the heating rate maintained at 20 °C min^{-1}, increasing the pyrolysis temperature from 350 to 520 °C, the C/O ratio increases from 3.18 to 4.9. Conversely decreasing the pyrolysis temperature from 350 to 310 °C, shows a decrease of the C/O ratio from 3.18 to 1.56. In addition, with the increase of pyrolysis temperature from 310 to 520 °C, the C/H ratio of the char residue increased significantly from 9.45 to 22.47, and slightly increased further at pyrolysis temperature of 520 °C for the conventional pyrolysis. Varying the heating rate in conventional pyrolysis to 520 °C shows no real trend with only minor differences in the C/O and C/H ratios.

Under microwave conditions an increase in the applied power from 600 to 1200 W, shows a decreasing C/O value with increasing power from 2.28 to 1.85. As an increase in applied microwave power is essentially equal to an increase in the heating rate, this features a obviously difference between microwave and conventionally operated pyrolysis.

3.4 Effect of heating rate on oil product composition

The liquid fractions obtained from the conventional and microwave pyrolysis of biomass were analyzed using GC-MS (Table 3). Furfuraldehyde and methoxyphenol (guaiacol) are the two main components accounting for more than 80 area% (percentage of total ion chromatogram peak areas) for conventional pyrolysis oil and more than 90% for microwave derived oil. This is consistent with previously published work on bio-oil [29,30]. While in the case of conventional pyrolysis, the methoxyphenol content decreases with increasing heating rate, the reverse is shown for microwave-facilitated pyrolysis in that the methoxyphenol content increases with increasing power. Irrespective of the operational mode of the pyrolysis process, the content of furfuraldehyde decreases with increasing heating rate or microwave power. Interestingly the GC-MS fingerprint of conventional and microwave pyrolysis are very similar (Figs. 4 and 5) and this in spite of the significantly lower biomass conversion in the case of microwave-assisted pyrolysis (Table 1).

In view of the above results and discussions, it is proposed that conventional pyrolysis is essentially a two-stage process in which the (hemi)-celluloses are firstly converted into CO_2 and CO and secondly CH_4 and H_2 are generated from lignin decomposition at higher temperatures. Intriguingly methane was found to evolve at lower temperatures during microwave pyrolysis with high microwave power.

4. Conclusions

In this work, two heating methods (conventional and microwave) were applied for biomass pyrolysis. It should be noted that direct comparison of data between the two systems is very difficult since the temperatures of pyrolysis of the two systems are different, the reactor size and configuration are also different. However, with these reservations in mind, general comparisons of the trends by changing heating rates for each scenario, the two systems can be tentatively made;

Two stages of gas release (CO/CO_2 at low temperature and CH_4/H_2 at high temperature) was clearly observed for the conventional pyrolysis; the peak release for CO/CO_2 and CH_4/H_2 were moved closer when the heating rate was increased from 5 to 40 °C min^{-1}. It was shown that microwave pyrolysis tends to produce less gas compared to conventional pyrolysis, for a similar liquid yield properly due to the fast heat transfer of microwave pyrolysis. Also, the release of methane and hydrogen was not observed at a conventional pyrolysis temperature of 310 °C; while release of CH_4 was increased for the microwave pyrolysis with the increase of heating power._While the C/O and C/H ratios in the conventionally obtained char residue are largely unaffected by the heating rate, the C/O ratio determined for the microwave char residue shows a clear decrease in yield with increasing power.

Acknowledgement

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Table 1

Mass balance of conventional and microwave pyrolysis of wood sawdust

	Conventional					Microwave[b]		
Pyrolysis temperature	520	520	520	350	310	200	200	200
Heating rate (°C min^{-1})/ microwave power (W)	5	20	40	20	20	600	900	1200
Product yield (wt.%)								
Char	25.45	22.12	21.21	38.48	59.70	48.39	45.16	43.23
Liquid*	53.94	59.09	58.18	46.67	31.52	43.23	45.81	47.10
Gas[a]	20.61	18.79	20.61	14.85	8.79	8.39	9.03	9.68

[a] Includes water; [δ] Gas yield was obtained from normalization of all products to 100%; [b] Microwave power of 600, 900 and 1200 W is related to heating rate around 8, 15 and 20 °C min^{-1}, respectively

Table 2

Element analysis of solid char obtained from various pyrolysis of biomass

	Conventional					Microwave			Biomass
Pyrolysis temperature	520	520	520	350	310	200	200	200	-
Heating rate (°C min^{-1})/ microwave power (W)	5	20	40	20	20	600	900	1200	-
Element yield of solid char (wt.%)									
N	2.05	2.48	2.53	2.51	2.06	2.31	2.39	2.35	1.78
C	79.21	78.29	78.19	69.55	56.06	63.91	61.27	59.29	36.17
H	3.98	3.28	3.48	6.08	5.93	5.76	7.17	6.33	6.30
O*	14.76	15.95	15.80	21.85	35.95	28.02	29.18	32.03	55.75
C/H	19.90	23.87	22.47	11.44	9.45	11.10	8.55	9.37	5.74
C/O	5.37	4.91	4.95	3.18	1.56	2.28	2.10	1.85	0.65

*Obtained from the mass balance

Table 3

GC-MS analysis of oil compounds (Area %)

RT (min)	Compound	Conventional pyrolysis heating rate (°C min^{-1})			Microwave pyrolysis power (W)		
		5	20	40	600	900	1200
5.393	Furfuraldehyde	31.32	22.37	25.59	40.78	27.79	22.61
9.244	Phenol	4.53	5.58	10.24	-	-	1.42
11.378	Methylphenol	1.67	0.86	5.43	0.37	0.29	0.71
12.049	Methylphenol	8.64	15.12	18.93	2.62	3.18	4.76
12.307	Methoxyphenol	51.36	49.15	28.05	56.22	68.52	69.35
12.864	Dimethylanisole	-	0.40	0.42	-	-	-
13.675	Ethylphenol	0.15	0.26	0.53	-	-	-
14.1	Dimethylphenol	1.05	4.54	7.30	-	0.23	0.56
14.636	Ethylphenol	0.80	0.39	1.01	-	-	0.59
15.497	Isopropylphenol	-	-	0.74	-	-	-
15.704	Trimethylphenol	0.25	0.76	0.91	-	-	-
16.266	Isopropylphenol	0.23	0.46	0.87	-	-	-
25.184	Dibenzofuran	-	0.10	-	-	-	-

Figure Captions

Fig. 1. Intensity of gas concentration (CO, CO_2, H_2 and CH_4) at different reaction time/temperature for conventional pyrolysis

Fig. 2. Intensity of gas concentration (CO, CO_2, H_2 and CH_4) at various reaction temperatures for microwave pyrolysis; Power: 600, 900 and 1200 W

Fig. 3. TGA-FTIR results for wood sawdust at different heating rates: 5, 20 and 40 °C min^{-1}

Fig. 4. GC-MS chromatograph of bio-oil from conventional pyrolysis under different heating rate: 5, 20 and 40 °C min^{-1}

Fig. 5. GC-MS chromatograph of bio-oil from microwave pyrolysis under different powers: 600, 900 and 1200 W

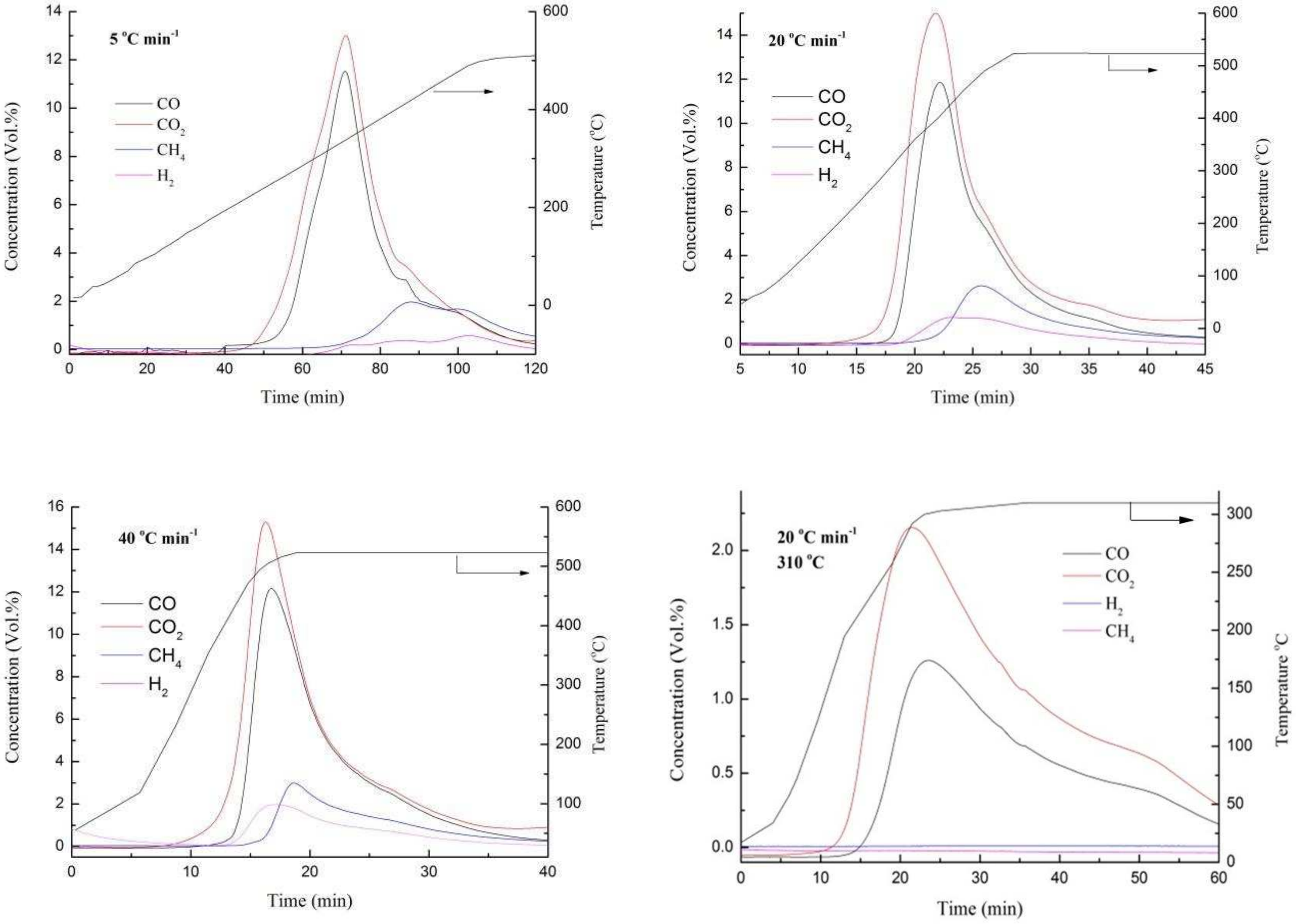


Fig. 1 Intensity of gas concentration (CO, CO_2, H_2 and CH_4) at different reaction time/temperature for conventional pyrolysis

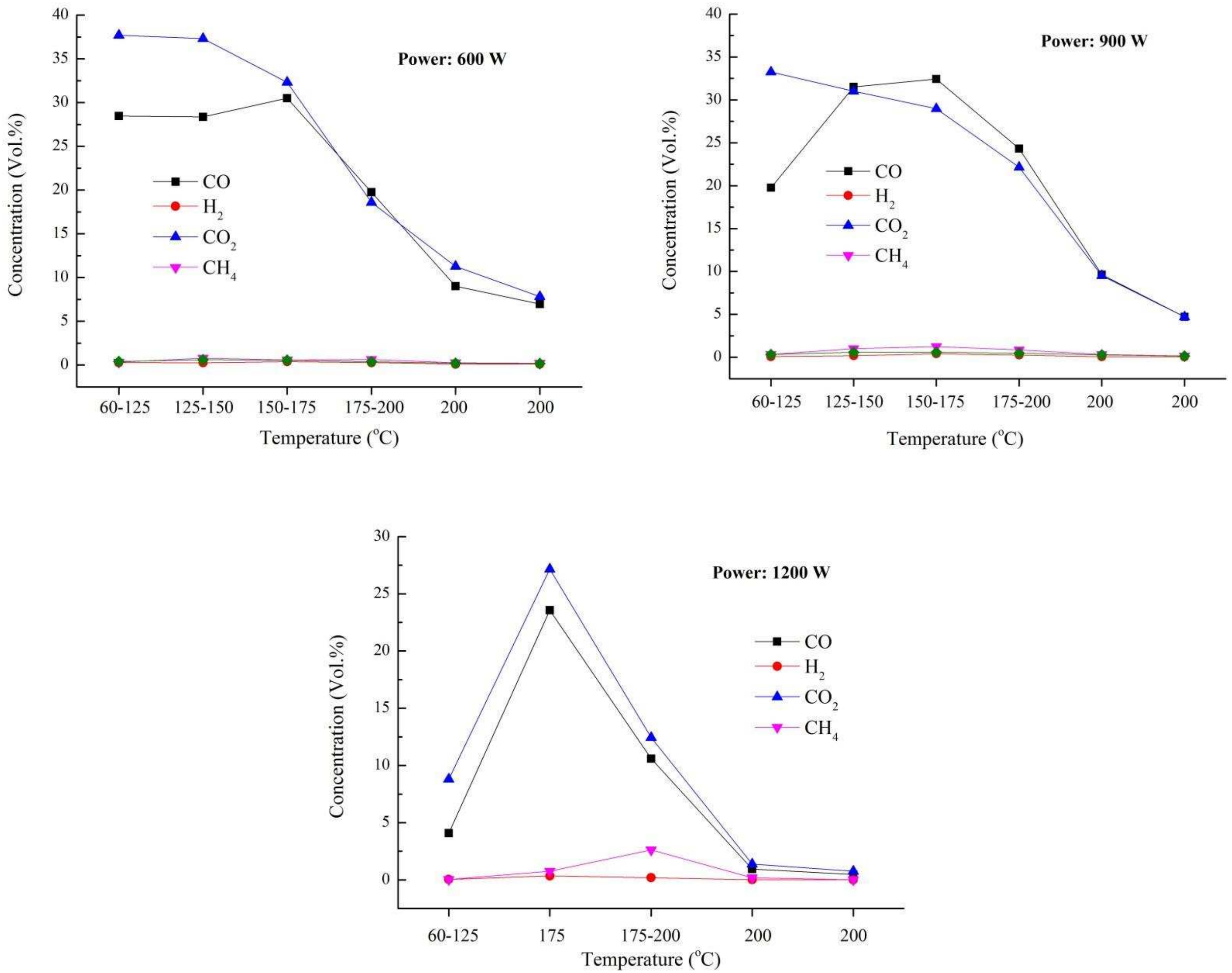
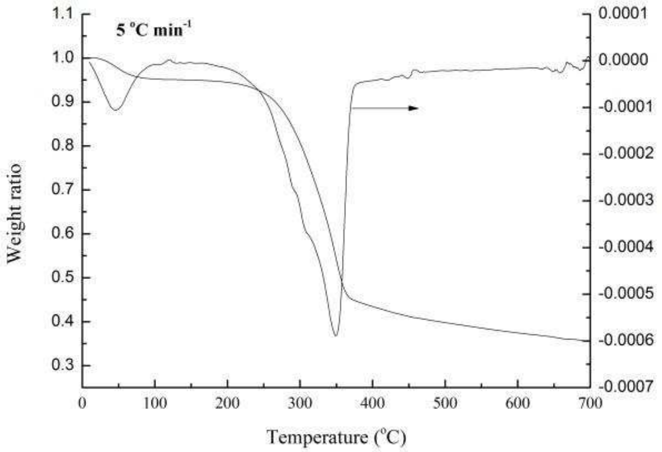
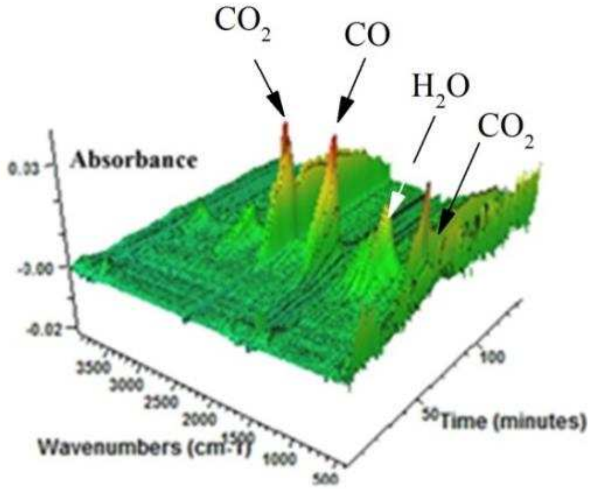


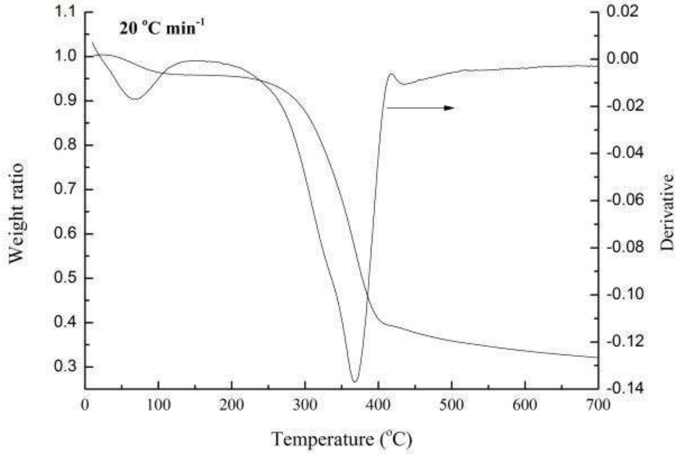
Fig. 2. Intensity of gas concentration (CO, CO_2, H_2 and CH_4) at various reaction temperatures for microwave pyrolysis; Power: 600, 900 and 1200 W



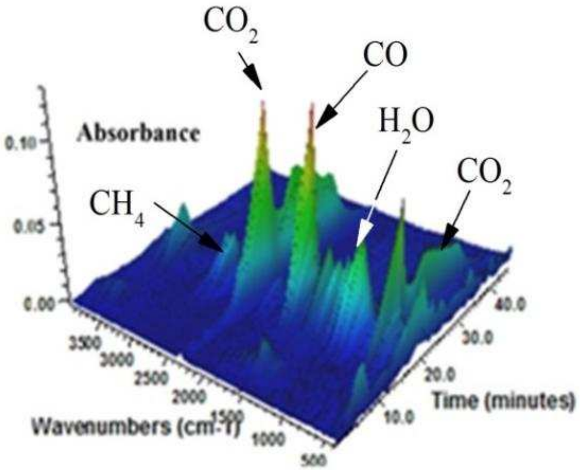
5 °C min-1
Weight ratio
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
Derivative
0.0001
0.0000
-0.0001
-0.0002
-0.0003
-0.0004
-0.0005
-0.0006
-0.0007
0
100
200
300
400
500
600
700
Temperature (°C)



CO2
CO
H2O
CO2
Absorbance
0.03
-3.00
-0.02
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
100
50
Time (minutes)



20 °C min-1
Weight ratio
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
Derivative
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
-0.12
-0.14
0
100
200
300
400
500
600
700
Temperature (°C)



CO2
CO
H2O
CO2
CH4
Absorbance
0.10
0.05
0.00
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
40.0
30.0
20.0
10.0
Time (minutes)

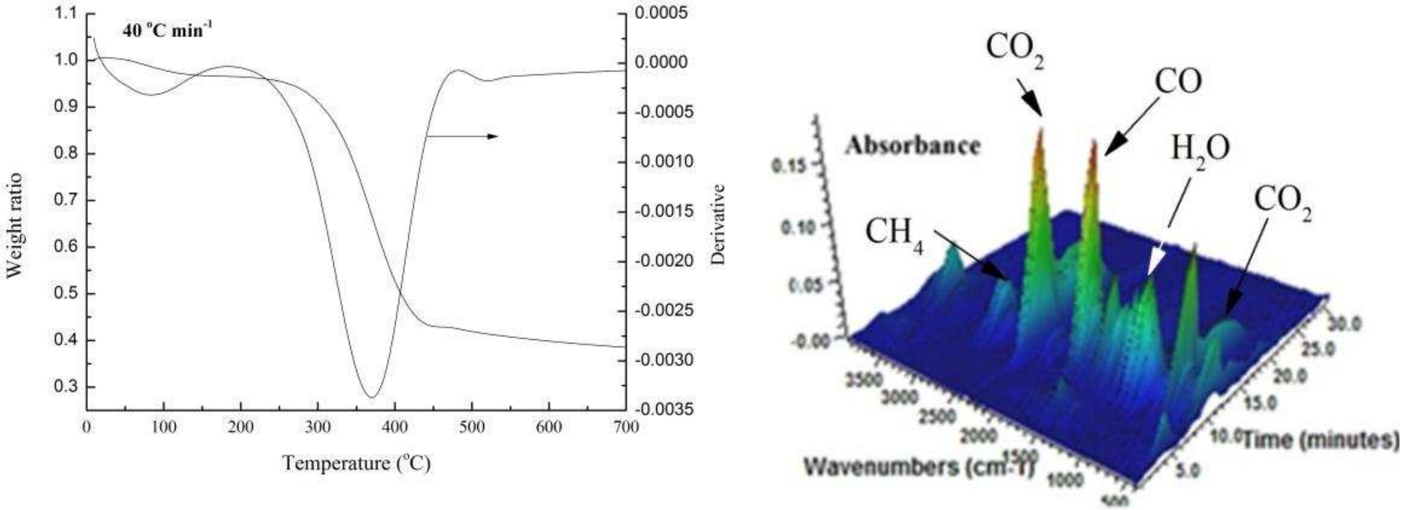


Fig. 3. TGA-FTIR results for wood sawdust at different heating rates: 5, 20 and 40 °C min^{-1}

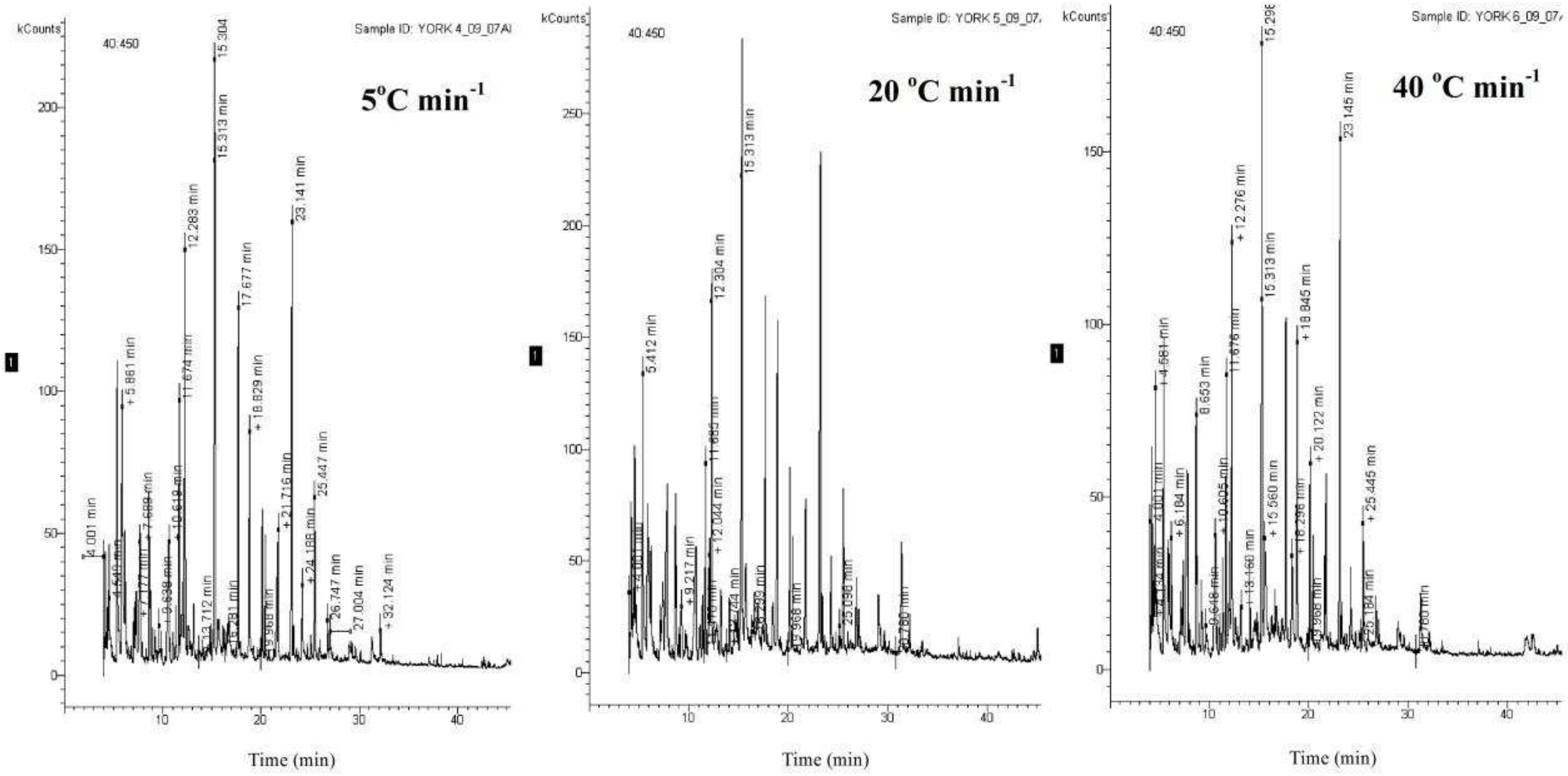


Fig. 4. GC-MS chromatograph of bio-oil from conventional pyrolysis under different heating rate: 5, 20 and 40 °C min^{-1}

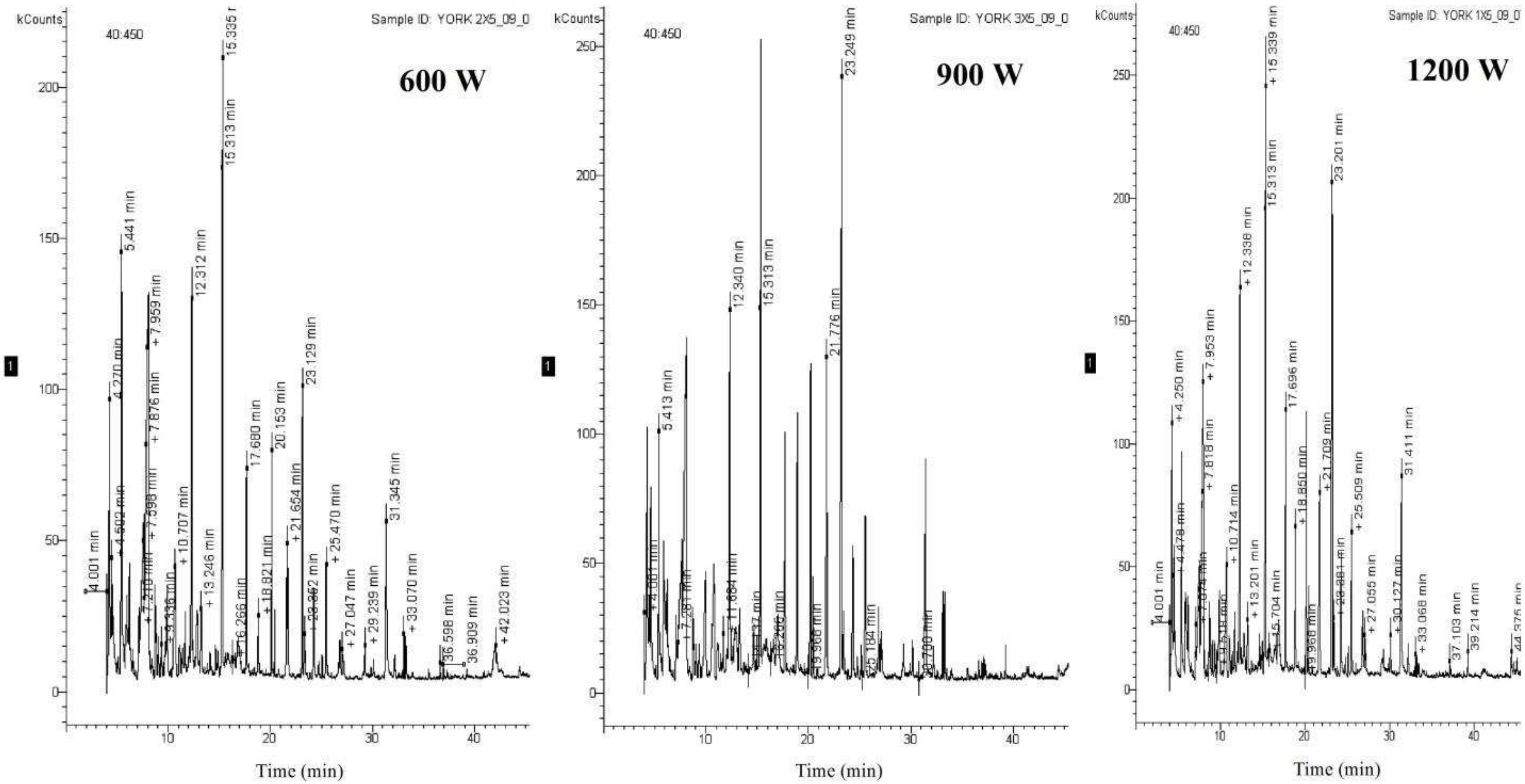


Fig. 5. GC-MS chromatograph of bio-oil from microwave pyrolysis under different powers: 600, 900 and 1200 W