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Assessment of thermal processing behaviour of corn cob, vine rod and sunflower

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Abstract: The efforts to achieve reduction in carbon emission, soaring prices of petroleum and concerns over secured energy supply are the major drivers in the search for alternative renewable energy sources. Typical and abundant biomass residues, such as corn cob, vine rod and sunflower, have potential to produce energy in carbon neutral way when subjected to pyrolysis conversion through biogas, bio-oils and bio-char products. The objective of this work was to assess the thermal behaviour of these three agricultural biomass materials under slow pyrolysis conditions, in order to quantify their energy potential. In that direction, a variety of technological tools were employed in the research. The thermo-gravimetric analyser was used on the raw samples to monitor the mass change and to determine the proximate analysis of the considered biomass samples, which is the basis for estimation of their calorific value. The raw samples and the char products of pyrolysis at 500°C were analysed using Fourier transform-infrared (FT-IR) spectroscopy. The results indicate change in functional groups between raw and solid char products, which refers to the loss of organics after pyrolysis at 500°C. The organic compounds contained in the pyrolysis oils at 500°C were investigated using gas chromatography - mass spectrometry (GC-MS). The obtained data provide important reference information on the opportunities for energy and fuel generation from pyrolysis of the analysed biomass.

Keywords: Renewable energy, biomass, biofuel, slow pyrolysis, corn cob, vine rod, sunflower

1. Introduction

The wider use of renewable energy sources is becoming increasingly necessary, as one of the ways to effectively address the global warming issue. As most scientists acknowledge, global warming is basically an energy crisis, caused by the world's 200-year long dependence on fossil fuels. Therefore, the solution to this issue is nothing less than a global energy revolution that replaces human dependence on petroleum, coal and natural gas with new, non-polluting, renewable energy sources [1]. Biomass is the most common form of renewable energy, widely used in the third world, but until recently, less so in the Western world [2]. With the rapid increase in global energy demand and increasing environmental and sustainability challenges, biomass fuels as renewable energy sources have increasingly been considered as one of the key options to substitute conventional fossil fuels. Currently, biomass and waste contribute to around 10% of the global energy supply [3]. Pyrolysis is one of the thermochemical technologies for converting biomass into energy and chemical products consisting of liquid bio-oil, solid biochar and pyrolytic gas [4]. Bio-oil is a complex mixture, highly oxygenated with a great number of large size molecules, which nearly involve all species of oxygenated organics, such as esters, ethers, aldehydes, ketones, phenols, carboxylic acids and alcohols [5]. L. Van Zwieteren et al., in their work [6] raise the expectation that bio-oil is thought to play a dominant role as a substitute for the crude oil due to its significant potential for the energy production through direct combustion. Bio-oil can be further upgraded for a better quality to make it suitable for engine applications [7]. Bio-char is a porous carbonaceous solid product of pyrolysis or incomplete combustion of organic materials, similar to charcoal but also utilized for agricultural and biological activities and/or environmental applications [8]. Furthermore, bio-char has the potential to be used as a soil amendment due to its very high surface area, which improves the water holding capacity of the soils, has nutrient dynamics and suppresses nutrient leaching [9]. As a mixture of a volatile gases, which consist primarily of CO₂, CO, CH₄ and higher hydrocarbon compounds, pyrolytic biogas can also be used as a combustible fuel [10]. Grapes are one of the world's largest fruit crops and grape stalks are one of the wastes generated by grape juice and wine-making processes. They are currently disposed of by distilleries, land fill or in rural areas [11]. Investigating the

potential of corn residues for energy, fuel, materials and chemicals production according to their thermo-chemical treatment products yields and quality, it can be stated that corn cob could be a solid and gaseous biofuel and they could be a good material for activated carbon production after being activated or gasified with steam [12]. Sunflower is a traditional crop, which can be used for the production of bioenergy and liquid biofuels [13]. The work [14] assesses the pyrolysis behaviour of tobacco waste, by use of methodology similar to the one applied in the present work, and reveals four stage pyrolysis mechanism consisting of dehydration, torrefaction, charring and carbonization.

The present work aims to determine the thermal processing behaviour under slow pyrolysis conditions of three biomass materials, which are typical and abundant agricultural residues in many parts of the world, including the South-Europe: corn cob, vine rod and sunflower. The objective of the research is to quantify their potential to produce energy when subjected to pyrolysis conversion through biogas, bio-oils and bio-char products of pyrolysis. In this work, a variety of technological tools were employed to characterise the pyrolysis of the three samples.

2. Materials and methods

For the purpose of this work, agricultural biomass residues, corn cob, vine rod and sunflower, were collected and investigated with several experimental techniques: thermogravimetric analysis (TGA), Fourier spectroscopy, gas chromatography and gas chromatography - mass spectroscopy.

The thermogravimetric analyzer TGA/DSC measures the flow of heat and the changes of the material weight as a function of time and temperature in controlled atmosphere. The analysis is performed in a way that the temperature of the sample increases gradually, resulting in a change in the mass of the sample.

In the present work, the analysis was performed with thermogravimetric analyser TGA/DSC 1 STARe System, Mettler Toledo Ltd. It was measured the mass loss of the biomass samples during heating from 25°C to 1000°C under a heating rate of 10°C/min, with nitrogen as carrier gas at flow rate 20 ml/min.

The analysed samples have the following amount of mass:

- Sunflower: 18.24 mg
- Vine rod: 26.27 mg
- Corn cob: 11.09 mg

The TGA/DSC instrument was also used to determine the proximate analysis of the corn cob, vine rod and sunflower samples. As a result, the content of moisture, volatile matter, ash and fixed carbon in each biomass sample was determined.

The measurement procedure is as follows:

Step 1. Heating the sample from 25 °C to 110 °C with a heating rate of 10 °C/min, and after that the sample is kept at 110 °C for a period of 5 minutes. During the experiment, nitrogen was used as a segment gas with a flow of 50 ml/min.

Step 2. From 110 °C, the temperature is increased up to 900 °C, with a heating rate of 50 °C/min, and the sample is kept at temperature of 900 °C for 15 minutes. In this step, nitrogen is used as a segment gas, with a flow rate of 50 ml/min.

Step 3. The segment gas (nitrogen) is replaced with air at flow rate of 50 ml/min. In this step, the sample with air as a segment gas is kept at 900 °C with duration of 10 minutes.

As for characterisation of pyrolysis products, an infrared furnace with temperature-programming was used to extract gaseous (pyrolytic gas), liquid (mainly pyrolysis oils) and solid matter (chars) by pyrolysis of corn cob, vine rod and sunflower. The fine particles of each rubber type were packed in a quartz tube fixed-bed reactor at a heating rate of 10 °C/min in an inert helium atmosphere.

The char products of pyrolysis at 500°C were analysed using Fourier transform-infrared (FT-IR) spectroscopy. The raw samples of corn cob, vine rod and sunflower were heated to 500°C for char production. Fourier transform infrared (FT-IR) spectrometer (model: Nicolet 6700 FT-IR) with an attenuated total reflectance (ATR) was used to acquire the functional groups of the raw samples and the chars after pyrolysis. The total number of scans was 32, with a spectral resolution of 4 cm⁻¹. The OMNIC software was

used for graphical analysis of results. In the instrument, the sample is placed on a diamond crystal, which by reflection allows to obtain the necessary measurements. The signal appears in a form of an interferogram.

For gas chromatographic (GC) analysis, the samples of sunflower, corn cob and vine rod were heated in infrared furnace at temperature of 800°C. Before the beginning of the experiment, helium was ran through the chromatographic analyser for duration of 20 minutes in order to remove the air in the apparatus channels. During the pyrolysis process, the temperature changes from ambient temperature up to 800 °C, where the gases emitted from the pyrolytic gas of the sample are analyzed at a certain temperature by an online micro gas chromatograph (model: M200, MTI Analytical Instruments) with two thermal conductive detectors. An ultra-high purity helium is used as a carrier gas. The hydrogen and carbon monoxide peaks were determined by molecular sieve 5A column at 60°C with carbon dioxide, methane, ethane and ethylene. The chromatographic spectrum is recorded at time interval of 100 seconds before a new amount of gas from the furnace is insert. With the use of by-pass, gases pass through ice container to the analyzer. The pyrolytic gas is analyzed while the instrument is operating.

Gas chromatography – mass spectrometry (GC-MS) is an analytical method that combines both analyses to determine the bio-oil composition in the considered biomass material. In the present study the pyrolysis oils were continuously swept out of the reactor exit until the temperature reached 500°C. The quartz wool was washed using dichloromethane (DCM) to extract the pyrolysis oil components, which were derivatized by N,O-Bis(trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane (BSTFA+1%TMCS). The model that was used to test the detailed components of pyrolysis oils is Agilent 7890B GC with 5977A MS system equipped with a HP-5MS column (60 m x 0.25 μ m). The oven was initially set to 40 °C for 2 minutes, then heated to 310 °C at 2 °C/min and kept at 310 °C for 30 minutes.

3. Results and discussion

The results of the proximate analysis of biomass waste: corn cob, vine rod and sunflower, obtained with TGA/DSC instrument, are presented in Table 1.

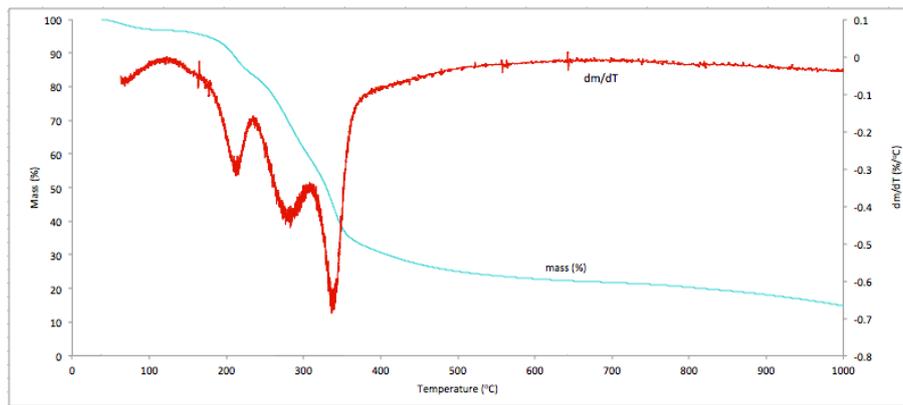
Table 1. Proximate analyses of corn cob, vine rod and sunflower

Biomass sample	Moisture content (%)	Volatile matter content (%)	Ash content (%)	Fixed carbon (%)
Corn cob	2.77	80.18	1.73	15.33
Vine rod	3.90	69.33	4.06	22.71
Sunflower	2.94	72.05	8.30	16.71

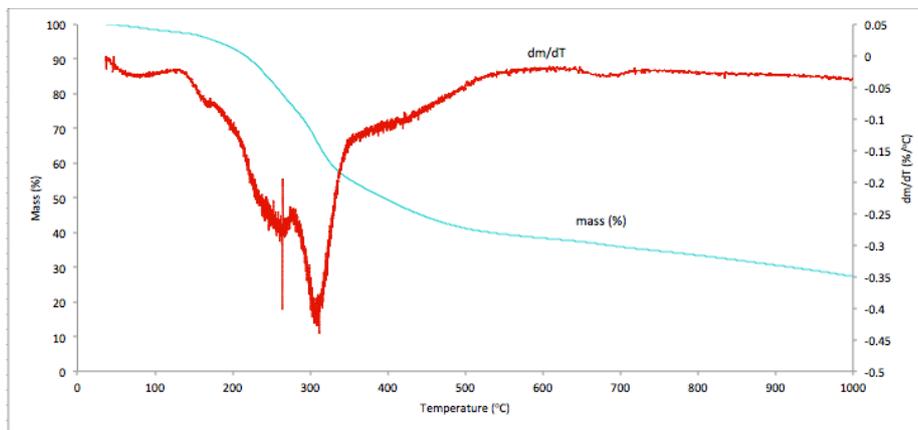
As a result of the analysis of the considered biomass samples, it was found that the corn cob has the highest volatile matter content (80.18%) and the lowest ash content (1.73%). The vine rod was found to have the highest moisture content (3.90%) of all three examined samples, and high content of fixed carbon (22.71%). All the samples were found to have high volatile matter and fixed carbon content, i.e. in total 88 to 95% of all contents.

The mass change of the samples during pyrolysis in function of the temperature, as determined by the Thermogravimetric Analysis method, is shown in Figure 1. The mass loss was measured while increasing the temperature of the samples from room temperature up to 1000 °C with heating rate 10 °C/min. The biggest degradation of mass happens in the temperature range from 200 °C to 450 °C, which indicates degradation of organic matters. At 500°C, the biggest degradation occurs to the corn cob with residual mass content of 24%, then sunflower with 36% and the vine rod with 40% of mass content.

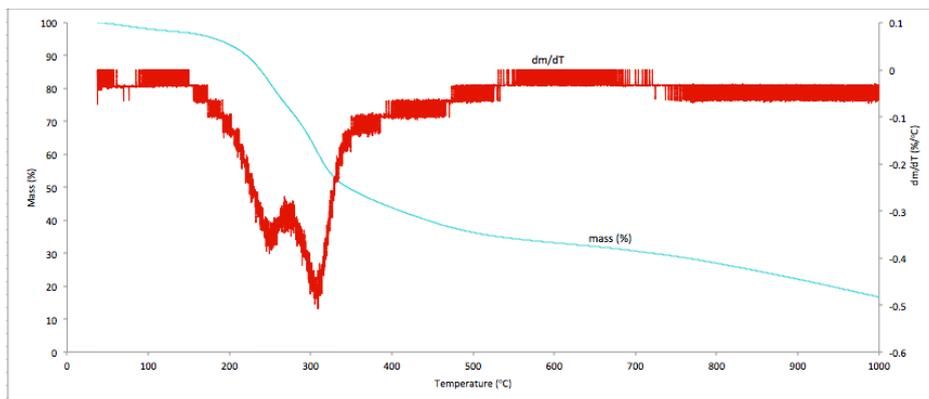
There can be observed three regions on differential thermo-gravimetric curve, suggesting that characteristic processes are taking place in these regions. The first one is a result of evaporation of the absorbed water in the sample. The second region is a result of the main complex reactions taking place from 200°C to 400°C, where the highest mass loss is observed and most of the volatiles are released. The peaks in this temperature range show the degradation of hemicellulose on the first peak and the decomposition of cellulose and lignin on the second peak. The third region that starts at 400°C is characterised with a continuous degradation with very low mass loss rate.



a)



b)



c)

Figure 1. Thermogravimetric analysis of (a) corn cob, (b) vine rod and (c) sunflower

Figure 2 shows the Fourier Transform Infrared (FT-IR) spectroscopic data of the biomass waste and its char products. The solid products of pyrolysis, were produced at temperature 500 °C. The FTIR spectra of the corn cob on figure 2(a) shows that the greatest changes happen from 500 cm^{-1} to 1750 cm^{-1} , which are expressed with much lower intensity at the bio-char. After 1750 cm^{-1} , the change of the bio-char is unremarkable, which shows that it is fully decomposed. Compared to raw corn cob, bio-char loses the functional groups O-H and $>\text{CH}_2$. From Figure 2(b), it is found that the greatest changes happen from 500 cm^{-1} to 1800 cm^{-1} wavelength. Compared to raw vine rod, bio-char loses the functional groups O-H and $>\text{CH}_2$. From Figure 2(c), it is found that the bio-char from the sunflower has less functional groups and

changes because of the loss of organic compounds. In comparison with the raw sunflower, bio-char is fully decomposed.

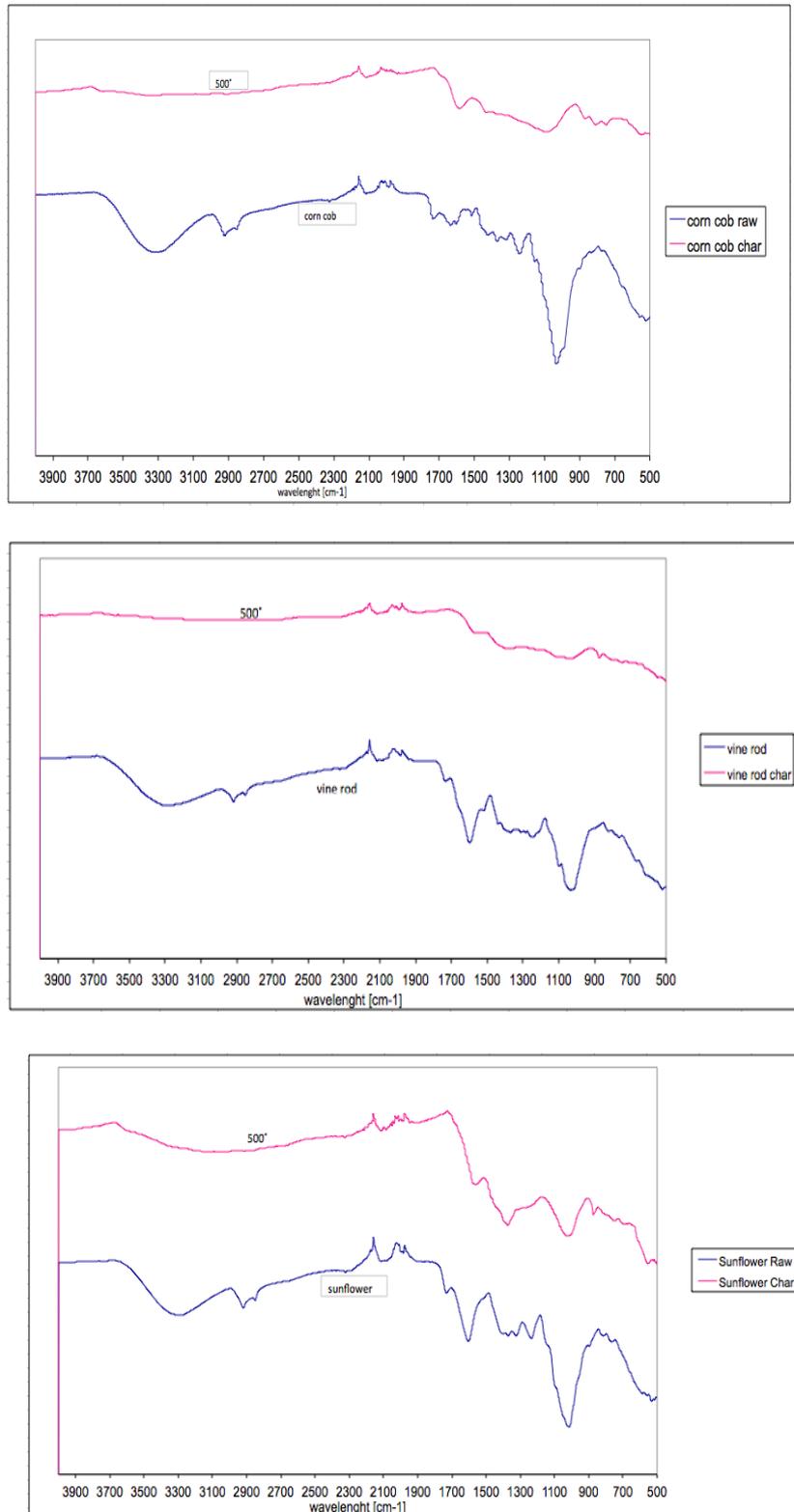


Figure 2. FTIR spectra of biomass waste pyrolysis products; (a) corn cob; (b) vine rod; (c) sunflower

In Figure 3, are shown the gas analysis results from the three examined samples: corn cob, vine rod and sunflower. It can be noticed that the largest quantity of gasses are released from vine rode, with amount of 21.18%, slightly less from sunflower with 18.82% and the smallest amount of gasses release corn cob with 12.29%. Carbon dioxide and carbon monoxide mainly originate from the degradation and transformation of

carbonyl (C=O) and carboxyl (COO) functional groups. Methanol is mainly derived from methoxyl (-O-CH₃) and methylene (-CH₂-) functional groups. The hydrogen origin is from the C=C and C-H functional groups. The pyrolysis gases have multiple potential application: direct use for production of heat or electricity and production of individual gas components (CH₄, H₂, etc.).

In Figure 3(c) is shown the amount of gases from sunflower released during the heating. At 500°C, of the total amount of biomass from sunflower, 18.82 % are gases, of which 15.7% is CO₂, 1.83% is CO, 0.34% CH₄, 0.61% H₂ and the other gases with smaller amount.

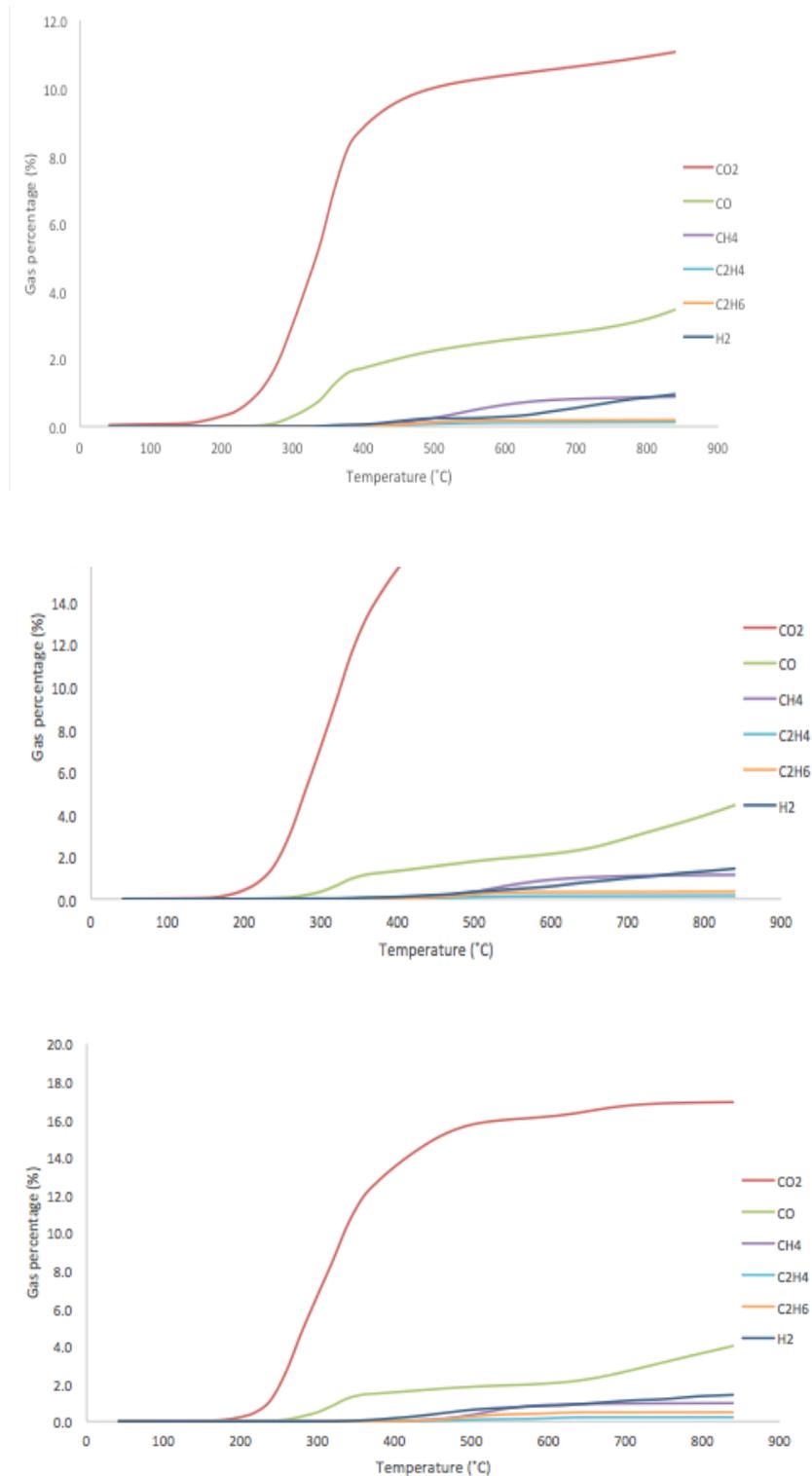


Figure 3. Gas concentration of biomass waste pyrolysis (a) corn cob; (b) vine rod; (c) sunflower

The organic compounds contained in the pyrolysis oils at 500°C were investigated using gas chromatography - mass spectrometry (GC-MS). The results of the conducted analysis are given in Tables 2, 3 and 4, showing that the most commonly present are the compounds that contain oxygen. A general consideration is that the bio-oils derived from corn cob, vine rod and sunflower have potential for further use. Because of the high quantity of acids, it is recommended that hydro-thermal process should be implemented to reduce the amount of oxygen.

Table 2. Results of gas chromatography - mass spectrometry (GC-MS) analysis of bio-oils of corn cob

No	Name of compound	Chemical formula	Content, %
1	isobutanol	C ₄ H ₁₀ O	2.63
2	neopentyl alcohol	C ₅ H ₁₂ O	1.05
3	furfuryl alcohol	C ₅ H ₆ O ₂	2.59
4	phenol	C ₆ H ₆ O	1.75
5	D-(-)-lactic acid	C ₃ H ₆ O ₃	0.72
6	pimelic acid	C ₇ H ₁₂ O ₄	2.72
7	cyclopentene-3-carboxylic acid, 1-hydroxyl-	C ₇ H ₁₀ O ₃	1.02
8	p-cresol	C ₇ H ₈ O	0.73
9	tetrahydro-2-furanylmethanol	C ₅ H ₁₀ O ₂	0.71
10	sorbic acid	C ₆ H ₈ O ₂	1.21
11	2-hexenoic acid, (E)-	C ₆ H ₁₀ O ₂	2.65
12	guaiacol	C ₇ H ₈ O ₂	0.93
13	3-ethylphenol	C ₈ H ₁₀ O	0.67
14	m-cresol	C ₇ H ₈ O	2.89
15	cis-4-hydroxycyclohexanecarboxylic acid	C ₇ H ₁₂ O ₃	3.36
16	glycerol	C ₃ H ₈ O ₃	1.66
17	catechol	C ₆ H ₆ O ₂	4.80
18	hydroquinone	C ₆ H ₆ O ₂	0.97
19	2-oxooctanoic acid	C ₈ H ₁₄ O ₃	1.29
20	4-methylcatechol	C ₇ H ₈ O ₂	0.93
21	syringol	C ₈ H ₁₀ O ₃	1.50
22	hydroquinone	C ₆ H ₆ O ₂	1.22
23	1,2,3,4-tetrahydroisoquinolin-6-ol-1-carboxylic acid, 7-methoxy-1-methyl-	C ₁₂ H ₁₅ NO ₄	3.08
24	2-hydroxycyclohexane-1-carboxylic acid	C ₇ H ₁₂ O ₃	3.75
25	2-{3,4-bis[hydroxy]phenyl}ethanamine	C ₈ H ₁₁ NO ₂	0.81
26	bicyclo[3.3.0]octan-2-one, [7-(1-phenyl)methylene]	C ₁₅ H ₁₆ O	0.91
27	2,2-dimethyl-5-[2-(ethoxymethoxy)-propyl]-[1,3]dioxolane-4-carboxaldehyde	C ₁₂ H ₂₂ O ₅	1.71
28	triethylene glycol	C ₆ H ₁₄ O ₄	2.03
29	D-(+)-ribono-1,4-lactone	C ₅ H ₈ O ₅	2.55
30	levoglucosan	C ₆ H ₁₀ O ₅	2.59
31	palmitic acid	C ₁₆ H ₃₂ O ₂	4.51
32	9,12-octadecadienoic acid (Z,Z)-	C ₁₈ H ₃₂ O ₂	3.54
33	9-octadecenoic acid, (E)-	C ₁₈ H ₃₄ O ₂	4.60
34	oleic acid, (Z)-	C ₁₈ H ₃₄ O ₂	1.22
35	stearic acid	C ₁₈ H ₃₆ O ₂	1.39

Table 3. Results of gas chromatography - mass spectrometry (GC-MS) analysis of bio-oils of vine rod

No	Name of compound	Chemical formula	Content, %
1	acetamide	C ₂ H ₅ NOi	2.15
2	ethylene glycol	C ₂ H ₆ O ₂	0.82
3	furfuryl alcohol	C ₅ H ₆ O ₂	0.63
4	phenol	C ₆ H ₆ O	3.63
5	o-cresol	C ₇ H ₈ O ₈	1.13
6	3-pyridinol	C ₅ H ₈ NO ₈	4.13
7	m-cresol	C ₇ H ₈ O ₈	3.16
8	2-pyrrolidinone	C ₄ H ₇ NO	3.04
9	p-cresol	C ₇ H ₈ O	1.33
10	hymexazole	C ₄ H ₅ NO ₂	1.01
11	3,5-dimethylphenol	C ₈ H ₁₀ O	1.55
12	guaiaicol	C ₇ H ₈ O ₂	0.56
13	3-ethylphenol	C ₈ H ₁₀ O	0.57
14	hexanoic acid	C ₆ H ₁₂ O ₂	1.68
15	maltol	C ₆ H ₆ O ₃	0.59
16	glycerol	C ₃ H ₈ O ₃	0.80
17	catechol	C ₆ H ₆ O ₂	9.15
18	acetin	C ₅ H ₁₀ O ₄	0.54
19	4-methylcatechol	C ₇ H ₈ O ₂	5.74
20	syringol	C ₈ H ₁₀ O ₃	2.40
21	hydroquinone	C ₆ H ₆ O ₂	2.71
22	ritalinic acid	C ₁₃ H ₁₇ NO ₂	0.66
23	2,3-2h-quinolin-2-one, 3,3,4,7-tetramethyl-	C ₁₂ H ₁₅ NO	0.58
24	(+)-salsolidine	C ₁₂ H ₁₇ NO ₂	0.57
25	2-(3,4-hydroxyl)ethanamine	C ₈ H ₁₁ NO ₂	1.88
26	bicyclo[3.3.0]octan-2-one, [7-(1-phenyl)methylene]	C ₁₅ H ₁₆ O	0.97
27	3-(2-hydroxyethyl)phenol	C ₈ H ₁₀ O ₂	0.64
28	triethylene glycol	C ₆ H ₁₄ O ₄	2.39
29	3,9-dioxa-2,10-disilaundecane, 2,2,10,10-tetramethyl-5-[(trimethylsilyl)oxy]-	C ₅ H ₁₂ O ₃	0.87
30	dodecanoic acid	C ₁₂ H ₂₄ O ₂	0.91
31	palmitic acid	C ₁₆ H ₃₂ O ₂	3.93
32	9,12-octadecadienoic acid (Z,Z)-	C ₁₈ H ₃₂ O ₂	2.33
33	.alpha.-linolenic acid	C ₁₈ H ₃₀ O ₂	1.81
34	stearic acid	C ₁₈ H ₃₆ O ₂	1.87
35	stigmast-5-ene, 3.beta.-(hydroxyl)-, (24S)-	C ₂₉ H ₅₀ O	1.06

Table 4. Results of gas chromatography - mass spectrometry (GC-MS) analysis of bio-oils of sunflower

No	Name of compound	Chemical formula	Content, %
1	isobutanol	C ₄ H ₁₀ O	0.58
2	acetamide	C ₂ H ₅ NO	1.18
3	3,3-dimethylacrylic acid	C ₅ H ₈ O ₂	0.69
4	ethylene glycol	C ₂ H ₆ O ₂	1.51
5	furfuryl alcohol	C ₅ H ₆ O ₂	1.03
6	tiglic acid	C ₅ H ₈ O ₂	0.66
7	phenol	C ₆ H ₆ O	3.70
8	propene	C ₃ H ₆	0.70
9	o-cresol	C ₇ H ₈ O	0.71
10	3-pyridinol	C ₅ H ₅ NO	1.64
11	m-cresol	C ₇ H ₈ O	1.15
12	p-cresol	C ₇ H ₈ O	0.90
13	hexanoic acid	C ₆ H ₁₂ O ₂	1.53
14	glycerol	C ₃ H ₈ O ₃	1.31
15	catechol	C ₆ H ₆ O ₂	3.65
16	acetin	C ₅ H ₁₀ O ₄	0.99
17	hexanoic acid, 3-hydroxyl-	C ₆ H ₁₂ O ₃	4.21
18	4-methylcatechol	C ₇ H ₈ O ₂	0.61
19	syringol	C ₈ H ₁₀ O ₃	0.92
20	hydroquinone	C ₆ H ₆ O ₂	4.83
21	pyrimidine, 5-methyl-2,4-hydroxy-	C ₅ H ₆ N ₂ O ₂	0.59
22	2,3-2h-quinolin-2-one, 3,3,4,7-tetramethyl-	C ₁₂ H ₁₅ NO	0.80
23	2-methylresorcinol	C ₇ H ₈ O ₂	0.94
24	triethylene glycol	C ₆ H ₁₄ O ₄	2.02
25	4-hydroxybutanoic acid	C ₄ H ₈ O ₃	0.88
26	1,5 pentanediol, 2-hydroxyl-	C ₅ H ₁₂ O ₃	0.83
27	dodecanoic acid	C ₁₂ H ₂₄ O ₂	0.68
28	myristic acid	C ₁₄ H ₂₈ O ₂	0.58
29	palmitic acid	C ₁₃ H ₃₂ O ₂	4.86
30	9,12-octadecadienoic acid (Z,Z)-	C ₁₈ H ₃₂ O ₂	3.89
31	.alpha.-linolenic acid	C ₁₈ H ₃₀ O ₂	2.10
32	stearic acid	C ₁₈ H ₃₆ O ₂	1.10
33	pimaric acid	C ₂₀ H ₂₀ O ₂	9.23
34	isopimaric acid	C ₂₀ H ₃₀ O ₂	2.02
35	abietic acid	C ₂₀ H ₃₀ O ₂	4.93

4. Conclusions

Agricultural biomass residues are potential source of energy locally. The process of pyrolysis is one of the options that offer conversion of the biomass waste to higher value products, such as bio-char, bio-oils and bio-gas. In this study, three different biomass types, including corn cob, vine rod and sunflower, were pyrolysed at 10°C/min to generate fuel of pyrolytic gas, oil and char. From the proximate analysis of the considered biomass types, it can be concluded that they are the best to use for production of gaseous bio-fuel. The fixed carbon as the second most abundant content, can be used to improve the properties of soil. The oxides of carbon, CO₂ and CO, as well as H₂ and CH₄ were basically the primary gas species in the gas product, indicating the potential of using the pyrolytic gas. In terms of bio-oil analysis, chemical compounds with their relevant contents were identified. Palmitic acids and catechol were the dominant compounds in the bio-oils, indicating its large potential for further biodiesel production. Generally, biomass pyrolysis is currently at the mature stage of development, with several technologies already achieving full commercialization stage. Their input into the global energy production is expected to achieve the full potential in the near future.

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