



Microwave pyrolysis of distillers dried grain with solubles (DDGS) for biofuel production

Hanwu Lei^{a,*}, Shoujie Ren^a, Lu Wang^a, Quan Bu^a, James Julson^b, John Holladay^c, Roger Ruan^d

^a Bioproducts, Sciences and Engineering Laboratory, Department of Biological Systems Engineering, Washington State University, Richland, WA 99354-1671, USA

^b Department of Agricultural and Biological Engineering, South Dakota State University, Brookings, SD 57006, USA

^c Pacific Northwest National Laboratory, Richland, WA 99354-1671, USA

^d Department of Bioproducts and Biosystems Engineering, University of Minnesota, St. Paul, MN 55108, USA

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ABSTRACT

Microwave pyrolysis of distillers dried grain with solubles (DDGS) was investigated to determine the effects of pyrolytic conditions on the yields of bio-oil, syngas, and biochar. Pyrolysis process variables included reaction temperature, time, and power input. Microwave pyrolysis of DDGS was analyzed using response surface methodology to find out the effect of process variables on the biofuel (bio-oil and syngas) conversion yield and establish prediction models. Bio-oil recovery was in the range of 26.5–50.3 wt.% of the biomass. Biochar yields were 23.5–62.2% depending on the pyrolysis conditions. The energy content of DDGS bio-oils was 28 MJ/kg obtained at the 650 °C and 8 min, which was about 66.7% of the heating value of gasoline. GC/MS analysis indicated that the biooil contained a series of important and useful chemical compounds: aliphatic and aromatic hydrocarbons. At least 13% of DDGS bio-oil was the same hydrocarbon compounds found in regular unleaded gasoline.

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1. Introduction

From mid of 1970s the American government began to encourage the fuel ethanol production and ethanol has become the important renewable energy and one of the most widely used types of biofuels. In the USA about 10% fuel ethanol blended with gasoline has been sold in gas stations and about 3.4 billion gallons of ethanol were consumed in 2004 and the amount was increased to 7 billion gallons in 2006 (Farrell et al., 2006). The demands and production of fuel ethanol are still increasing. The production of distillers dried grains with soluble (DDGS), the co-product from producing fuel ethanol, is markedly increased with the fuel ethanol.

DDGS is mainly composed of protein, fiber and fat (Belyea et al., 2004; Bhadra et al., 2007). Because of its nutrients DDGS is generally used as additives for animal feeds (Senne et al., 1995; Zijlstra and Beltranena, 2007). DDGS can be food additives such as corn oils from DDGS by extraction (Singh and Cheryan, 1998). Following the understanding for its physical and chemical properties (Rosentrater, 2006), DDGS can be used as a bio-filler for biomaterial products such as bio-plastics (Rosentrater and Otieno, 2006; Tatara et al., 2007) and fertilizer source for plant growth (Nelson et al., 2009).

DDGS was also considered to be the potential energy sources and contributed to the energy cost saving by generating electricity (Morey et al., 2006; Tiffany et al., 2007; De Kam et al., 2007). In recent years, some researches were focused on the bio-oil, hydrogen and syngas production using DDGS. Xu et al. investigated the factors that influenced the yields of liquid products when DDGS was liquefied with hot compressed phenol (Xu et al., 2008). Tavasoli et al. using a continuous downflow fixed bed micro reactor gasified corn and wheat DDGS and studied the effect of temperature, reaction time and ratios of oxygen to nitrogen on DDGS gasification (Tavasoli et al., 2009). The reaction temperature was the main factor in the steam–air fluidized bed gasification for DDGS (Kumara et al., 2009). The kinetic analysis of DDGS pyrolysis with the weight lost occurred at three main stages was studied by Giuntoli et al. (2009).

Microwave pyrolysis is an efficient thermochemical process to produce bio-oil, syngas and bio-char. The microwave-assisted pyrolysis method has been successfully applied to plant residues (Yu et al., 2007; Wang et al., 2008; Lei et al., 2009; Huang et al., 2010; Salema and Ani, 2011), wood (Miura et al., 2004) and sewage sludge (Domínguez et al., 2006; Tian et al., 2010). Some advantages were found in microwave pyrolysis, such as high yields of products, harmful chemical reduction in the bio-oil and energy cost saving. But microwave pyrolysis of DDGS has not been found in literature.

In this study, DDGS was pyrolyzed to produce bio-oil, syngas and bio-char by microwave pyrolysis. The effects of reaction

* Corresponding author. Tel.: +1 509 372 7628; fax: +1 509 372 7690.

E-mail address: hlei@tricity.wsu.edu (H. Lei).

temperature, reaction time and power input were investigated for DDGS microwave pyrolysis and statistical models were established to predict the yields of products. GC/MS analysis was performed to understand the chemical composition of bio-oils.

2. Methods

2.1. Material

DDGS was provided by Dakota Ethanol LLC (Wentworth, SD). The chemical composition of DDGS was previously reported (Bhadra et al., 2007; Rosentrater et al., 2009) and the same DDGS was used in this study. The chemical compositions included neutral detergent fiber (NDF, with overall mean value of 36.74% db), crude protein (overall mean value 29.93% db), acid detergent fiber (ADF, overall mean value of 16.2% db), total starch (overall mean value 11.07% db), total fat (overall mean value of 10.5% db) and crude fiber (overall mean value of 10.22% db), and crude ash (overall mean value of 12.82% db) (Bhadra et al., 2007; Rosentrater et al., 2009). NDF is the sum of the ADF and hemicellulose content. ADF is composed of cellulose and lignin. Crude fiber is calculated by subtracting ADF value from NDF content. DDGS had a moisture content of about 6% and stored at the room temperature conditions (25 °C) before microwave pyrolysis. Regular unleaded gasoline was purchased from a Shell Gas Station at the state of South Dakota.

2.2. Methods

2.2.1. Microwave pyrolysis apparatus

A Sineo MAS-II batch microwave oven (Shanghai, China) with a rated power of 1000 W was used at the 300–1000 W power setting. The heating rates of DDGS were varied at different power inputs for 100 g of DDGS. The heating rates were 105, 130, 255, 500, and 570 °C/min at the power inputs of 600, 700, 800, 900, and 1000 W, respectively. The 100 g of DDGS was placed in a 500 mL quartz flask inside the microwave oven. Airtight quartz connections, insulated with alumina fibers, lead from the flask to a condenser system with collectors for the liquids. Five parallel bulb condensers, each one half meter long, were used for the condensation. The temperature of the cooling water in the condensers was 0–5 °C. The system was purged with nitrogen for 15 min prior to microwave treatment to create an oxygen-absent gas background. During pyrolysis the heavier volatiles were condensed into liquids as biooils and the lighter volatiles escaped as syngases at the end of the condensers, where they were either burned or collected for analysis. Bio-char was left in the quartz flask. The weight of syngas was calculated using following equation:

$$\text{Weight of syngas} = 100 \text{ g} - \text{weight of bio-oil} - \text{weight of bio-char} \quad (1)$$

2.2.2. Heating value measurement

The heating values of bio-oils were analyzed by the Poultry Laboratory at the University of Arkansas.

2.2.3. GC/MS analysis

Chemical compositions of the bio-oils were determined using a VARIANCE 3400 GC/MS with a DB-5 capillary column. The GC was programmed at 40 °C for 0.5 min and then increased at 10 °C/min to 300 °C and finally held with an isothermal for 10 min. The injector temperature was 300 °C and the injection size was 1 µL. The flow rate of the carrier gas (helium) was 0.6 ml/min. The ion source temperature was 230 °C for the mass selective detector. The com-

pounds were identified by comparing the spectral data with the NIST Mass Spectral library.

2.2.4. Experimental design

Central composite experimental design (CCD) was used in this study (Box and Wilson, 1951). Three factors, reaction temperature (X_1 , °C), reaction time (X_2 , min) and power input (X_3 , w) were selected as independent variables. Reaction time was recorded after the desired temperature was reached. The yields of bio-oil, syngas, bio-char (Y_i ,%) were used as dependent output variables. For statistical calculations, the variables X_i were coded as x_i according to the Eq. (2):

$$x_i = (X_i - X_0) / \Delta X \quad (2)$$

where x_i is dimensionless value of an independent variable, X_i is real value of an independent variable, X_0 is real value of the independent variable at the center point, and ΔX is step change.

A 2^3 -factorial CCD, with six axial points ($\alpha = 1.68$) and six replications at the center points ($n_0 = 6$) leading to a total number of 20 experiments was employed (Tables 1 and 2) for the optimization of the conditions of pyrolysis process. The power input was increased by 100 W per step as set by the manufacturer. Six hundred watts was used to replace 631 W at $\alpha = -1.68$ and 1000 W was used to replace 968 W at $\alpha = 1.68$. The second degree polynomials (Eq. (3)) were calculated with the statistical package (SAS Institute Inc., USA) to estimate the response of the dependent variable.

Table 1
Codes levels of independent variables in the experiment plan.

Levels	Reaction temperature (°C)	Reaction time (min)	Power input (W)
$-\alpha = -1.68$	516	4.6	600
-1	550	8	700
0	600	13	800
1	650	18	900
$+\alpha = 1.68$	684	21.4	1000
Δj	50	5	100

Table 2
Central composite design matrix and experimental results of DDGS.

Run	x_1	x_2	x_3	Bio-oil yield (%)	Syngas yield (%)	Bio-char yield (%)
1	-1	-1	-1	26.53	11.30	62.17
2	1	-1	-1	47.31	25.71	26.98
3	-1	1	-1	49.49	23.36	27.15
4	1	1	-1	45.98	30.54	23.48
5	-1	-1	1	29.85	14.67	55.48
6	1	-1	1	41.64	27.91	30.45
7	-1	1	1	45.80	26.87	27.33
8	1	1	1	45.86	31.52	22.62
9	-1.68	0	0	39.20	15.69	45.11
10	1.68	0	0	50.30	26.08	23.62
11	0	-1.68	0	24.46	9.78	65.76
12	0	1.68	0	45.46	31.01	23.53
13	0	0	-1.68	47.68	23.06	29.26
14	0	0	1.68	40.96	21.64	37.40
15	0	0	0	40.79	25.21	34.00
16	0	0	0	43.80	21.44	34.76
17	0	0	0	42.33	24.53	33.14
18	0	0	0	43.27	22.37	34.36
19	0	0	0	43.43	23.60	32.97
20	0	0	0	42.03	24.20	33.77

Note: X_1 is reaction temperature; X_2 is reaction time; X_3 is power input.

$$Y_i = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x_1^2 + b_{21}x_2x_1 + b_{22}x_2^2 + b_{31}x_3x_1 + b_{32}x_3x_2 + b_{33}x_3^2 \quad (3)$$

where Y_i is predicted response, X_1 , X_2 , and X_3 are independent variables, b_0 is offset term, b_1 , b_2 , and b_3 are linear effects, b_{11} , b_{22} , and b_{33} are squared effects, and b_{21} , b_{31} , and b_{32} are interaction terms.

3. Result and discussion

3.1. Responses surface analysis

The most important physical factors which affected production were the reaction time and reaction temperature. The suitable levels for these parameters were determined using statistical CCD. The central composite design matrix and experimental results of DDGS microwave pyrolysis were shown in Table 2. Twenty

experiments were performed using different combinations of the variables as per the CCD. The Eq. (3) was reduced by using backward statistical analysis, and insignificant parameters (P -value > 0.05) were removed from the full model Eq. (3). Using the results of the experiments, second order polynomial regression equations (Eqs. (4) and (5)) were obtained to model the yields of bio-oil, and bio-char; and the first order regression equation (Eq. (6)) was obtained to model the yield of syngas. The Eqs. (4)–(6) were obtained with its significant terms (P -value < 0.05).

The regression equations of bio-oil (4), bio-char (5), and syngas (6) were as follows:

$$Y_{\text{bio-oil}} = 43.71 + 3.5x_1 + 5.65x_2 - 1.28x_3 - 2.78x_2^2 - 4.5x_1x_2 \quad (4)$$

$$Y_{\text{bio-char}} = 32.94 - 7.67x_1 - 10.66x_2 + 3.27x_2^2 + 6.48x_1x_2 \quad (5)$$

$$Y_{\text{syngas}} = 23.02 + 4.17x_1 + 5.01x_2 \quad (6)$$

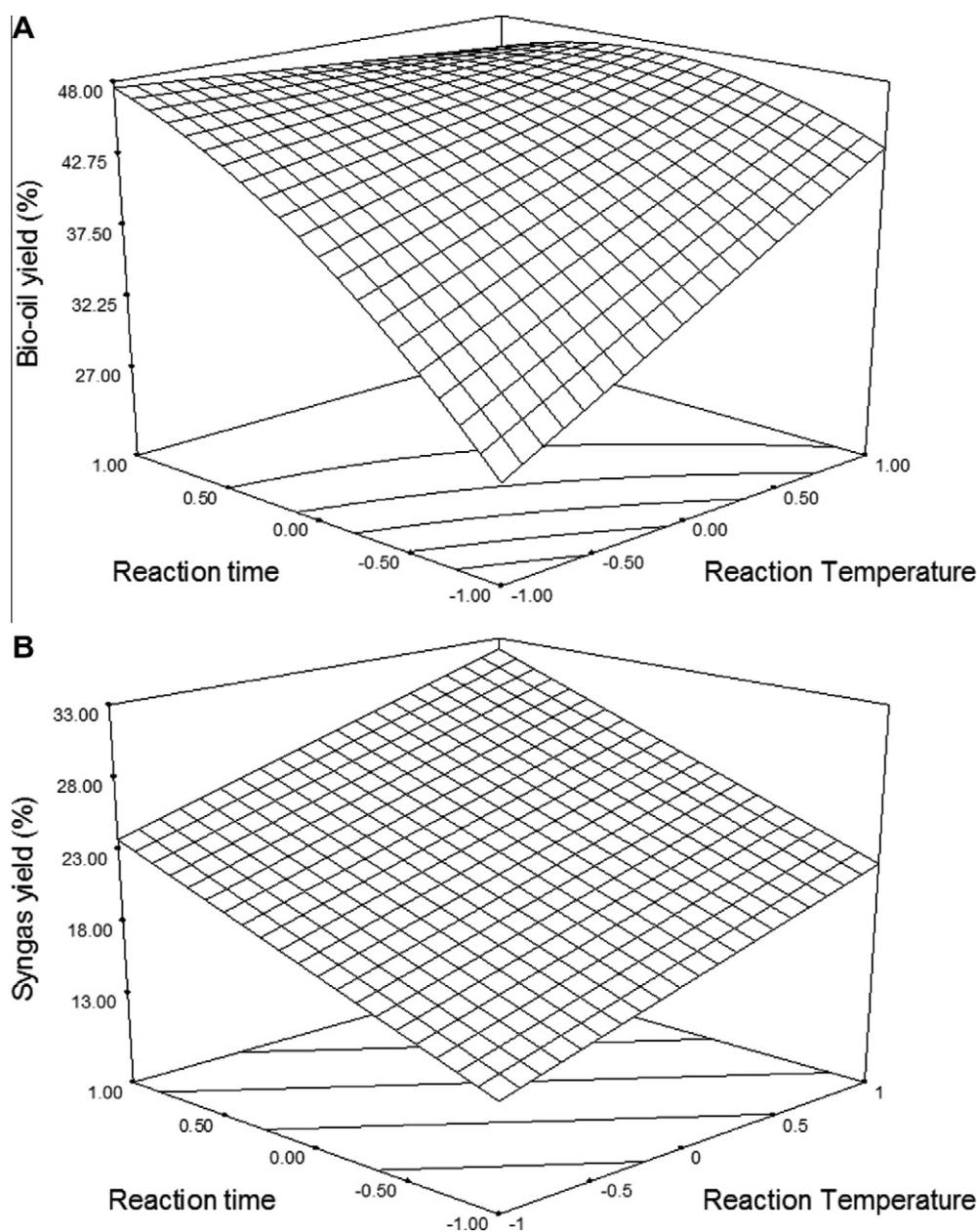


Fig. 1. The response surface and contour line of bio-oil yield (A), and syngas yield (B) as a function of reaction temperature and reaction time at the power input of 800 W.

where Y represents the yield of bio-oil, bio-char, and syngas and x_1 , x_2 , and x_3 are the code values of reaction temperature, reaction time and power input respectively. The corresponding analysis of variance (ANOVA) indicated less deviation between observed and fitted values with P -values for the F test statistic less than 0.0001. ANOVA provided strong evidence for the relationship between product yields and the process conditions.

The correlation coefficients of determination, R^2 were 0.94, 0.95, and 0.83, respectively for bio-oil, bio-char, and syngas, implies that the reduced regression models can be used to explain the pyrolysis reaction, and the variations of product yields were attributed to the independent variables of reaction time, reaction temperature, and their interaction. The power input was found significant for the yield of bio-oils.

Fig. 1 represents the isoresponse contour and surface plots for the pyrolysis conditions of DDGS for the yields of bio-oil (A) and syngas (B). The yields of bio-oil and syngas ranged from 24.46% to 50.30% and 9.78% to 31.52%, respectively, and increased with the increase of reaction temperature and time. The highest yields of syngas (31.52%) were observed at 650 °C, 18 min and 900 W, and the highest yield of bio-oil (50.30%) was observed at 684 °C, 13 min and 800 W. The yield of bio-char decreased with the increase of reaction temperature and time. The lowest yield of bio-char (22.62%) was observed at the conditions of 650 °C, 18 min, and 900 W.

The reaction temperature and time were the important factors in the biomass microwave pyrolysis in previous report (Lei et al., 2009). In this study, the results also showed that the reaction temperature and time and their interactions had significant effects on the DDGS microwave pyrolysis. The power input was not significant in the yields of volatile and bio-char, but the power input had significant effect on the yield of bio-oils according to the Eq. (4). At the same reaction time and lower temperature, higher heating rates and power inputs (e.g. 900 W of 500 °C/min) resulted in increased yields of bio-oil and syngas, for example runs 1 and 5 at the same

reaction temperature of 550 °C and time of 8 min. But at the same reaction time and higher temperature, higher heating rates and power inputs (e.g. 900 W of 500 °C/min) resulted in decreased yields of bio-oil and increased yields of syngas, for example runs 4 and 8 at the same reaction temperature of 650 °C and time of 18 min. Higher power with higher pyrolysis temperature favored gasification reactions which decreased the yield of bio-oils. These results are similar to those reported in the literature (Şensöz et al., 2006; Pan et al., 2010; Islam et al., 2010; Du et al., 2011).

3.2. The heating value of bio-oils

The bio-oils were phase separated into two levels at room temperature. The lower level was aqueous phase, and the upper level was oil phase. The heating value of the aqueous phase was about 2–5 MJ/kg, which was lower than the heating value of other biomass bio-oils (~18 MJ/kg; Bridgwater and Peacocke, 2000). The heating value of the oil phase was about 20–28 MJ/kg, which was much higher than the heating value of other biomass bio-oils (Bridgwater and Peacocke, 2000; Huang et al., 2008). This was confirmed by the high hydrocarbon contents (C_xH_y) in organic bio-oil from DDGS which resulted in a much higher energy density for this bio-oil. The average heating value of the oil phase was about 23 MJ/kg, which was about 54.8% of the heating value of gasoline (42 MJ/kg). The highest heating value of DDGS bio-oil was obtained at the 650 °C and 8 min, which was about 66.7% of the heating value of gasoline.

3.3. The analysis of bio-oils by GC/MS

To further understand the chemical reactions from microwave pyrolysis, GC/MS analysis was carried out to determine the composition of bio-oils. The bio-oils were heterogeneous and contained two phases at room temperature. At higher temperature of 50 °C, phase separation was not obvious. The DDGS bio-oils were prepared in the incubator shaker at 50 °C for 30 min and shaken vigorously using Vortex Mixer for 1 min before uniformly sampling for GC/MS analysis. The major composition of bio-oil products are shown in Table 3. The product peaks were assigned by automatic library search NIST98. More than 300 chemicals were detected in bio-oils from DDGS. Bio-oil was mainly composed of aliphatic hydrocarbons and aromatic hydrocarbons. Aliphatic hydrocarbons present in the bio-oil were about 48.9–66.7% of the bio-oil and mainly composed of cycloalkene, hexane, cyclohexane, 6,8-dodecadien-1-ol (6Z,8Z), cyclododecanemethanol, and nonene. Aromatic hydrocarbons detected in the bio-oil were benzenes (4.8–15.8%), phenols (1.3–9.2%), and naphthalene (6–10%). The acetic acids were considered a main component in the liquid bio-oils from lignocelluloses biomass pyrolysis. In this study acetic acid was not detected, but other organic acids, such as benzoic acid and butenoic acid detected were about 1% of the bio-oil. However, several basic organo-nitrogen compounds that are the result of protein pyrolysis were observed in the GC/MS chromatogram of the DDGS bio-oil. Nitrogen bases identified in the DDGS bio-oil included benzonitrile, 1-isocyanato-3-methyl-benzene, 3-phenyl-(E)-2-propenenitrile, and indole among several others. The large amounts of water insolubles in the bio-oil from DDGS are more likely derived from protein than from lignin because DDGS does not contain large amounts of lignin.

Fig. 2 shows the carbon number distributions for bio-oil samples prepared from the pyrolysis of DDGS by microwave heating at 550 and 650 °C. The analysis of chromatograms and carbon number distribution confirmed that the chemical composition of bio-oil was temperature and time dependent. It was noted that the range of carbon sizes run from C5 to C17 with the most prevalent size, C6–14. C6–14 chemical compounds that found in DDGS

Table 3
Major identified molecular products from microwave pyrolysis of DDGS.

Number	Retention time	Area percentage/ %	ID
1	3.842/3.991	2.002	C ₈ H ₁₀
2	4.028/8.02/10.984/12.208/13.908	5.059	C ₁₀ H ₂₀
3	4.369	6.104	C ₁₁ H ₂₄ O
4	4.701/6.348	0.745	C ₁₀ H ₁₈
5	5.384/5.439/5.627/5.641/5.887/ 6.401	3.07	C ₉ H ₁₂
6	5.759	1.459	C ₇ H ₅ N
7	6.715	0.737	C ₁₀ H ₁₄
8	6.781/6.914	1.740	C ₉ H ₁₀
9	7.095	4.131	C ₉ H ₈
10	7.503/7.600/8.444/11.167	3.826	C ₈ H ₇ N
11	7.996/8.134/8.671	2.504	C ₁₀ H ₁₀
12	8.444	0.773	C ₁₀ H ₁₂
13	8.955/16.285	1.875	C ₁₁ H ₁₆
14	9.515	8.046	C ₁₀ H ₈
15	10.415	0.583	C ₉ H ₇ N
16	10.583	0.741	C ₁₂ H ₁₈
17	10.726/20.321	1.877	C ₁₀ H ₁₆ O
18	11.268/11.500	3.640	C ₁₁ H ₁₀
19	11.826	0.741	C ₁₀ H ₉ N
20	12.794/12.809/12.939	1.774	C ₁₂ H ₁₂
21	13.153	1.848	C ₁₃ H ₁₈ O ₃
22	13.268	1.617	C ₁₂ H ₁₀
23	15.567	1.372	C ₁₃ H ₁₀
24	16.161/16.210	2.623	C ₁₃ H ₂₆ O
25	17.049	1.023	C ₁₄ H ₂₂ O
26	20.704	0.802	C ₁₁ H ₁₈ O
27	21.309	4.111	C ₁₂ H ₂₂ O
28	21.889	1.192	C ₁₆ H ₂₈ O

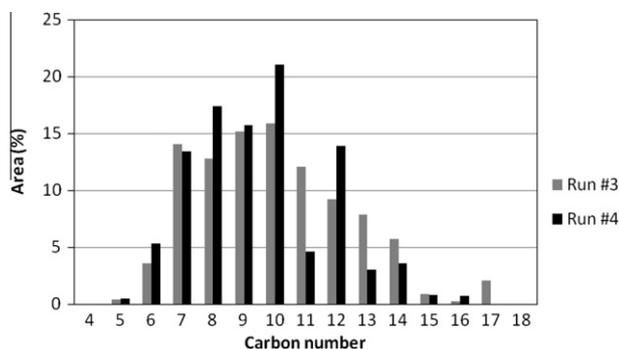


Fig. 2. Carbon number distribution of bio-oils: run 3 (550 °C and 18 min), and run 4 (650 °C and 18 min).

bio-oils were up to 95% of bio-oils. Fig. 2 also indicated that increasing pyrolysis temperature decreased large sized carbons (C13–17) and increased small sized carbons (C8–10).

The bio-oil from microwave pyrolysis of DDGS was separated into two phases at room temperature. The presence of less oxygenated compounds (C_xH_y) in bio-oil was found in the upper level of oil phase which will increase the stability of oil and energy density of the final hydrocarbon fuel. This was confirmed by about 40–55% of hydrocarbons (C_xH_y) produced without the co-feeding of hydrogen (Table 3) by GC/MS. It was found that this hydrocarbon oil was miscible with gasoline up to 1:1 (v/v of bio-oil:gas) without phase separation and without adding any additives and emulsifiers. Carbon number distributions of the gasoline and blend were determined using GC/MS (Fig. 3). The carbon number distribution of the blend did not change too much as compared with that of gasoline. The amount of C8 was decreased along with the increase of C9 and C10. The important hydrocarbons were found in both gasoline and DDGS bio-oil (Table 4). At least 13% of DDGS bio-oil was the same hydrocarbon compounds found in gasoline, which indicated that oil phase of DDGS bio-oil from microwave pyrolysis is good hydrocarbon sources to partially replace gasoline.

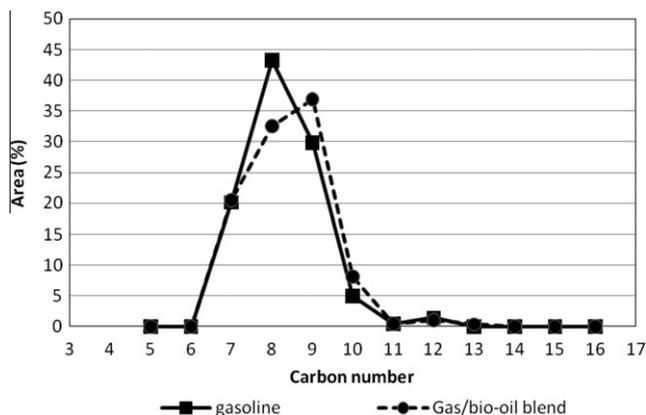


Fig. 3. Carbon number distribution of gasoline/DDGS bio-oil blend at 1:3 (v/v of bio-oil:gas).

Table 4
Identified hydrocarbon products for gasoline and DDGS bio-oil.

Name	ID	Gasoline	Bio-oil
Ethylbenzene	C_8H_{10}	6.669	1.836
p-Xylene	C_8H_{10}	23.807	1.449
Benzene, cyclopropyl	C_9H_{10}	0.247	1.740
Benzene, 1,2,3-trimethyl-, and others	C_9H_{12}	25.848	3.07
1-Octene, 3-ethyl-, and others	$C_{10}H_{20}$	2.575	5.059

4. Conclusions

Microwave pyrolysis of DDGS was investigated to determine the effects of pyrolytic conditions on the yields of bio-oil, syngas, and biochar. It was found that reaction temperature and time were the main factors affecting the DDGS biofuel yields. The highest heating value of DDGS bio-oil was obtained at the 650 °C and 8 min, which was about 66.7% of the heating value of gasoline. DDGS bio-oils contained a series of important and useful chemical compounds which were related to the pyrolysis conditions and can be blended with gasoline without phase separation.

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