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Study on Characteristics of Oil Palm Empty Fruit Bunch to Predict Condensation Temperature of Tar from its Pyrolysis Gas

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ABSTRACT

Palm oil is one type of plants that has various advantages since almost all parts of the plant, such as trunk, empty fruit bunch (EFB) and the extracted oil, can be utilized as energy. Utilization of EFB for energy, especially the use of combustible gas as direct feed of combustion engines to generate electricity is still rarely used. This is related to the water content and existence of tar in the syngas which is unfavorable. To overcome the problem, information on the characteristics and thermophysical properties, such as dew point, of tar in the pyrolysis of EFB is indispensable. In this study, the kind and quantity of tar was experimentally measured and dew point of the tar was calculated. Three EFB samples were taken from three different environmental conditions, namely samples EFB1, EFB2 and EFB3, to be analyzed. The elemental and proximate analysis as well as GCMS pyrolysis test of the samples were then used to characterize tar in the syngas and to determine its thermo-physical properties in terms of its dew point. The elemental analysis of the EFB samples showed disparity of its content, which was around 52.08±7.59% (C), 7.05±0.53% (H), 2.28±0.43% (N), 0.35±0.36% (S) and 34.84±4.45% (O). Likewise, the proximate analysis showed differences around 4.49 ± 2.60% (MC), 5.80±1.97% fixed carbon (FC) and 73.44±3.78% volatile matter (VM). From the elemental and proximate analysis, it can be predicted that tar compounds produced from pyrolysis of the EFB at a temperature of 400 °C, was in the form of mixed oxidation compounds (mixed oxygenates). The compounds were classified as the first tar compound (class 2) consisting of phenols (ketones, phenol and guaiacol). The dew point temperature and the concentration of the tar in its pyrolysis gas were predicted to be 204.22 °C, tar 1720.79 mg $Nm^{3\ -1}$ (sample EFB1); 256.02 °C, tar 92.97 mg $Nm^{3\ -1}$ (sample EFB2); and 154.85 °C, tar 359.02 mg $Nm^{3\ -1}$ (sample EFB3), respectively. This information can be useful in designing the tar elimination devices from the pyrolysis gas.

Keywords: condensation temperature, oil palm empty fruit bunch, pyrolysis, tar compound, tar concentration

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INTRODUCTION

Empty fruit bunch (EFB) is a by-product of oil palm mill, with great potential to be applied in industrial sectors, including material, chemical and energy sectors (Abdulrazik *et al*. 2017). The EFB contains cellulose, hemicellulose and lignin, with composition range of 23.7-65.0%, 20.6-33.5% and 14.1-30.45%, respectively (Law *et al*. 2007). EFB can be used directly as energy source by direct combustion, or converted into syngas for using in internal combustion engine.

Gasification is one of the available conversion technology to convert biomass into syngas. Gasification is a series of processes, including pyrolysis, oxidation, reduction and drying. Pyrolysis is the main process to convert the biomass structure into simple structure and gases. The gas produces is typically a mixture of hydrogen (H_2), carbon monoxide (CO), methane (CH_4) and carbon dioxide (CO_2) and several other compounds in the form of tar.

Tar is the main contaminant in the syngas, which becomes problem in its uses as engine fuel. Figure 1 shows the diagrammatic process flow of thermochemical reaction in gasification process. It is shown that tar formation cannot be avoided and will be condensed at temperature below 400 °C. If the process temperature drops below the dew point, then tar causes some problems even at low concentrations.

Tar is generally in the form of aerosols and polymerization with more complex structures. It is described as a substance with dark colour, oily and thick. Generally, tar is defined as a mixture of condensed hydrocarbon complexes, which consists of a single ring (aromatic compound), 5-ring together with other oxygen-containing hydrocarbons (aromatic hydrocarbons) and polyclic complex aromatic hydrocarbons (PAHs). Tar comes from the organic part of biomass through a series of complex thermochemical reactions, such as chemolysis, oxidation, depolymerization and polymerization, which

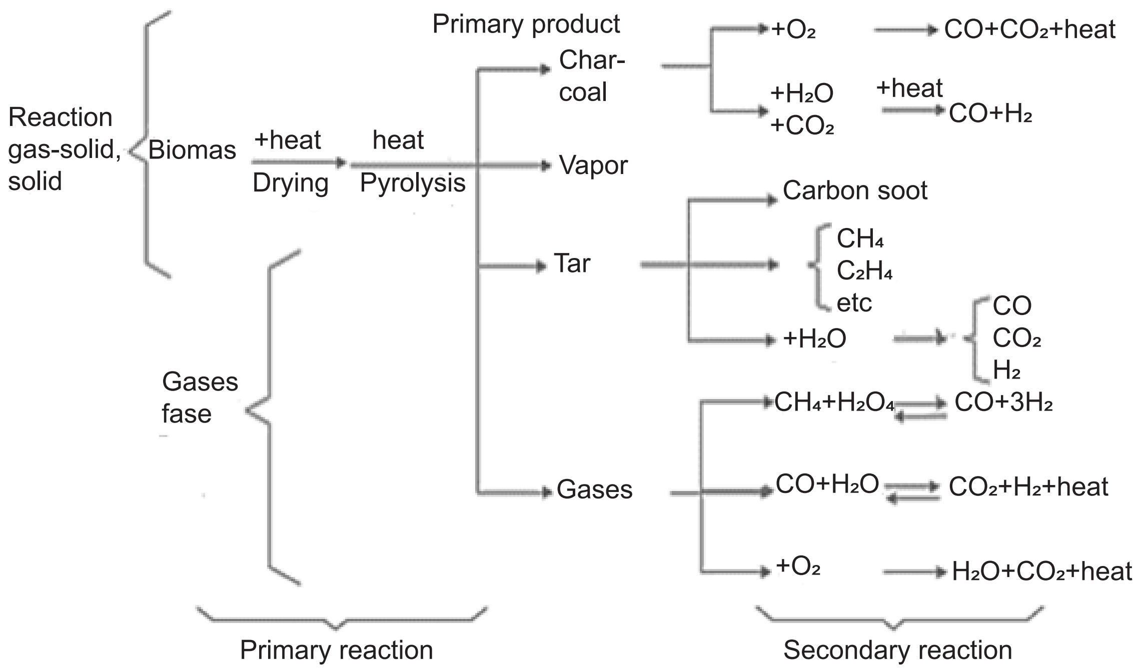

Figure 1 Biomass thermochemical reaction (Richardson *et al*. 2015).

occurs during gasification of cellulose, hemicellulose and lignin in the reactor. Lignin decomposition yields a much higher tar than cellulose and hemicellulose decomposition.

In addition, at a gasification temperature of 800 °C an extraordinary difference was observed between tar compositions. The main components, besides PAH, after decomposition of lignin are phenols and their derivatives. Benzene, toluene, ethylbenzene, xylene isomer (BTEX) and other hydrocarbons, consisting mainly of ethers, esters and furans, are the main products of cellulose and hemicellulose gasification. The quality and quantity of tar depends, in addition to the nature of the fuel, on the gasification process parameters (temperature, pressure, oxidizing media, type of gasifier, etc.).

Tar is grouped according to 5 rings classes, namely: heavy (not detected by GC); heterocyclic aromatics; mild aromatics (1 ring); lightweight PAH (2-3 rings); and heavy PAH (4-7 rings). The group depends on process temperature, where at 400 °C there exists a mixture of oxidation compounds; at 500 °C exist phenol and esters; at 600 °C exists alkyl and phenolic; at 700 °C exists heterocyclic and esters; at 800 °C exists mild PAH; and at 900 °C exists severe PHA. Besides, type of biomass (depend on its physical and chemical properties) also affect the type of tar in the produced gas (Morf *et al*. 2002).

This objective of this study is to identify and classify tar compounds exists in the pyrolysis gas of oil palm empty fruit bunch, and predict its condensation temperature. The result of this study is required for the development of tar compound cleaning tools for further applications.

MATERIAL AND METHODS

Tar Identification

The material used in this research is oil palm empty fruit bunches (EFB) obtained from a palm oil processing factory in Cikasungka, West Java, Indonesia. Three samples, namely EFB1, EFB2 and EFB3, were analysed. EFB1 and EFB2 were obtained directly from the factory with new conditions, while EFB3 was obtained from the waste pile and has been wasted for 2 months around the factory. EFB1 was dried naturally and EFB2 was oven dried at temperature 105 °C in 24 hours.

Ultimate and proximate analysis were performed to the samples, with compliance to ASTM standards. The standard include ASTM D5373 for carbon, hydrogen, nitrogen test; ASTM D3176 for oxygen test; ASTM D4239 for sulfur test; ASTM D3173 for moisture content test in dry air; ASTM D3174 for ash content test; ASTM D3175 for volatile test; ASTM D3172 for fixed carbon test; and ASTM D5865 for heating value.

Produced gas and tar identification was performed using pyrolysis-GCMS, as shown in Figure 2. It consists of pyrolysis reactor and GCMS, with their respective types are PY-2020iS and GCMS-QP2010 Shimadzu. EFB were sample (0.1 μg) was placed in a container of the reactor and processed at a temperature of 400 °C without catalyst. Helium gas (He) carrier was used to channel the pyrolysis gas. The resulting decomposition product was transferred to a separate separation column for identification by pyrogram. The individual gas components in the pyrogram were identified continuously using mass spectra. The test results were obtained in the form of diagrams and concentration data and types of compounds.

Condensation Temperature Prediction

Condensation of temperature can be calculated from the vapor pressure of the gas, which is burnable when it is in a single compound or consists of several components in the compound using equation 1. Here, the behaviour of the tar vapor is assumed as an ideal gas (Rabou *et al*. 2009).

$$22400 \times \frac{C_{tar} \times T_{dp}}{273 \times MW \times P_{dp}(T)} = 1 \quad (1)$$

where, C_{tar} is compound concentration of tar (g $Nm^{3\ -1}$); T_{dp} is dew point temperature (K); MW is molecular weight of compounds (g mol^{-1}); $P_{dp}(T)$ is dew point pressure at designated temperature (atm); ideal molar volume of gas is 22.400 m^3 mol^{-1}.

The saturated vapor pressure of a gas containing tar can be calculated using equation 2, and dew point pressure (P_{dp}) is expressed as in equation 3.

$$P_{sw} = 10^{\left(A - \frac{B}{T+C}\right)} \quad (2)$$

$$P_{dp} = \frac{1}{\sum_{i-1}^{k} \left(\frac{y_i}{P_{sw}} \right)} \quad (3)$$

where, A, B, C are Antoine constant; T is pyrolysis temperature (K); yi is steam volume fraction (%); P_{sw} is saturated vapour pressure (mmHg).

Relationship between liquid volume fraction (x_i) and steam volume fraction (y_i) of the tar is shown in equation 4.

$$x_i = \frac{y_i}{P_{sw}} \times P_{dp} \quad (4)$$

RESULTS AND DISCUSSION

Ultimate and Proximate Analysis

Table 1 shows the comparison of ultimate and proximate analysis for EFB1, EFB2 and EFB3 samples. The proximate analysis is displayed in wet bases (wb), while the ultimate analysis in dry bases (db). The composition of the EFB was found to be 52±8% (C), 7.1±0.5% (H), 2.3±0.4% (N), 0.5±0.2% (S) and 35±4 (O); while the ash was 6±2%, fixed carbon (FC) 17±1% and volatile matter (VM) 73±4%. Variation of the data was high due to many factors, including the age of the plant, harvest time, etc. The same data variation also found in other

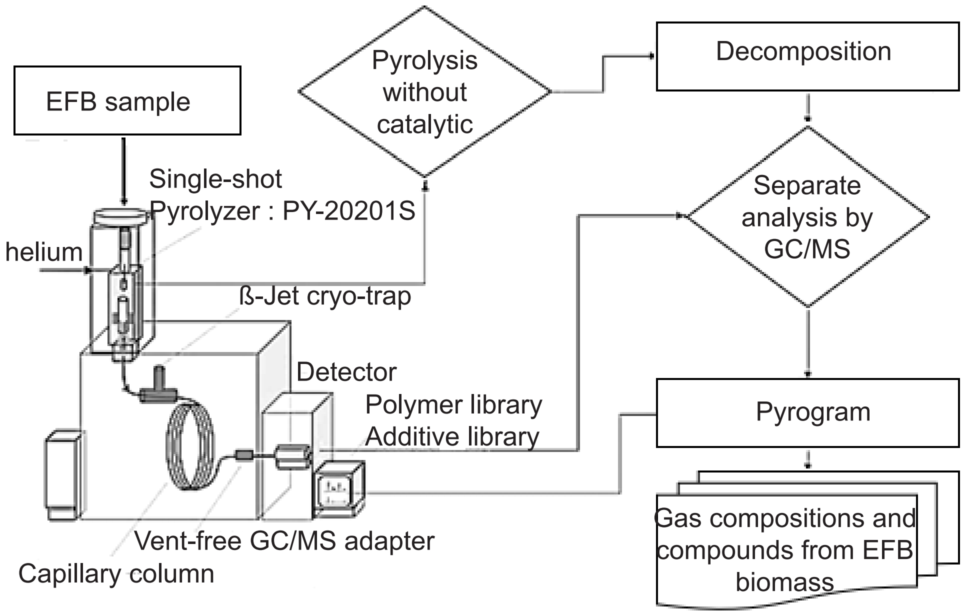

Figure 2 Flow chart of biomass gas characterization using Pyrolysis-GCMS (Shin *et al.* 2011).

literatures, which is summarized in Table 2 (C 48±5%, H 6.4±1%, N 0.8±0.6%, S 0.3±0.3% and O 44±6; while the ash was 5±3%, FC 14±4% and VM 85±6%). The value of higher heating value (HHV) of the sample was also measured and shown in the. The HHV was in the range 20±2 MJ kg^{-1}. Normally, higher content of carbon,

Table 1 Main analysis and results of direct EFB tests

Sample	Proximate nalysis (% wb)			Ultimate analysis (% db)					HHV (MJ kg^{-1})
	Ash	FC	VM	C	H	N	S	O	
EFB1	7.40	18.25	89.20	60.40	7.60	2.20	na	29.80	18.74
EFB2	3.74	16.23	89.93	50.32	7.02	1.89	0.72	40.05	21.54
EFB3	7.05	18.73	83.73	45.53	6.54	2.74	0.34	44.81	18.72
Mean	6.06	17.73	87.62	52.08	7.05	2.28	0.35	34.84	19.67
St.D	2.02	1.33	3.39	7.59	0.53	0.43	0.27	4.45	1.62

Tabel 2 The results of test are based on ultimate and proximate analysis data in dry basis (db) of EFB

Proximate analysis (db %)			Ultimate analysis (db %)					HHV (MJ kg^{-1})	Reference
Ash	FC	VM	C	H	N	S	O		
3.31	15.44	86.16	48.79	7.33	0.00	0.68	43.20	18.96	Yang *et al.* 2004
3.29	8.87	87.14	48.79	7.33	0.00	0.68	43.18	18.96	Hamzah 2008
4.29	12.84	85.66	43.21	7.42	0.86	0.75	47.76	18.66	Khor *et al.* 2009
4.98	16.13	83.33	40.93	5.42	1.56	0.31	51.78	16.80	Idris *et al.* 2010
5.82	9.52	85.37	49.07	6.48	0.70	0.10	43.65	19.35	Abdullah *et al.* 2011
8.92	11.69	78.78	50.00	6.50	0.62	0.12	34.82	19.74	Erlich *et al.* 2011
6.87	13.02	83.33	44.30	6.20	0.44	0.09	48.94	19.24	Kerdsuwan *et al.* 2011
4.88	8.75	86.58	43.52	5.72	1.20	0.66	48.90	15.22	Lahijani *et al.* 2011
7.77	19.79	87.15	45.00	6.40	0.25	1.06	47.30	18.10	Omar *et al.* 2011
3.18	13.00	93.71	53.78	4.37	0.35	0.00	41.50	17.08	Sukiran *et al.* 2011
5.26	16.65	90.38	47.65	5.20	1.82	0.36	44.97	16.80	Idris *et al.* 2012
3.64	13.32	90.10	46.62	6.45	1.21	0.04	45.66	17.02	Mohammed *et al.* 2012
14.50	12.14	78.23	53.22	6.25	0.97	0.48	39.08	17.61	Ruengvilairat 2012
5.82	9.52	85.37	49.07	6.48	0.70	0.10	43.65	19.35	Geng 2013
4.97	16.18	84.95	43.15	5.73	1.20	0.04	49.88	17.57	Nyakumaa *et al.* 2013
4.12	13.22	92.65	44.71	6.76	0.21	0.41	47.91	21.77	Alias *et al.* 2014
3.21	22.05	93.08	45.36	6.43	0.32	0.41	47.48	18.51	Alias *et al.* 2014
4.97	16.18	84.95	43.14	5.73	1.20	0.05	49.88	17.57	Nyakumaa *et al.* 2015
7.50	18.59	82.93	44.80	7.30	0.65	0.47	46.78	17.94	Chew *et al.* 2016
4.11	14.74	88.65	66.17	9.54	1.51	0.06	22.72	18.72	Raju *et al.* 2016
2.66	5.90	87.89	45.44	6.22	1.58	0.36	46.40	17.00	Tang *et al.* 2017
3.39	12.03	68.05	46.71	5.97	0.07	0.03	47.22	16.00	Lisandy *et al.* 2017
3.50	10.08	74.72	45.90	6.10	0.43	0.13	47.44	15.50	Han & Kim 2018
4.13	15.40	91.79	55.59	6.30	1.82	0.07	36.23	20.38	Lee *et al.* 2018
8.61	19.36	86.20	52.74	5.73	1.43	0.12	39.98	17.62	Kim *et al.* 2019
5.35	13.78	85.49	47.91	6.37	0.84	0.30	44.25	18.06	**Mean**
2.60	3.90	5.84	5.34	0.96	0.58	0.29	6.17	1.52	**St.D**

hydrogen and sulphur will lead to higher value of HHV, while in contrary, oxygen content will lower the value.

Fixed carbon (FC) is an indicator of the carbon content contained in biomass. Its value is influenced by the age of the biomass itself. Biomass that is harvested too fast has a low fixed carbon content, whereas lately harvested will have higher content of fixed carbon. Consequently, this also affects concentration of other components such as ash content and volatile matter.

Pyrolysis of EFB

Based on the pyrolysis-GCMS measurements, there was 32-40 compound species produced after 30-45 minutes of the process. The compound was dominated by fatty acids and some of them were phenol compounds. These compounds can be grouped into class 2 (Paul *et al*. 1994; Milne *et al*. 1998; Morf *et al*. 2002) and belongs to the category of oxygenate mixtures which are commonly found in gasification or pyrolysis upper layers at 400 °C. The results of this grouping are shown in Table 3, Table 4 and Table 5, for each sample of EFB1, EFB2 and EFB3, respectively.

If the compound, which is still in the form of an oxygenate mixture, is recirculated in a reactor or combustion chamber with temperatures above 600 °C, a number of aromatic hydrocarbons with 1 and 2 rings could be obtained in the form of poly-aromatic hydrocarbon (PAH). The greater the number of carbon chain bonds in a hydrocarbon compound, the more energy is required to break this chain into more simple compounds.

Decomposition of cellulose and hemicellulose by pyrolysis at temperature range of 329-350 °C results in depolymerization and dehydration process to form furfuran compounds and 2-furancarboxaldehyde. While, at higher temperature range of 400-471 °C, the C=O bond breaks and

Table 3 Group of individual organic compounds in tar compounds produced from pyrolysis of EFB1 sample

Tar classes	Compounds	Conc. (%)	Name	Chemical formula	MW (g mol^{-1})	CAS
The primary group of tar compounds (Class 2)	Furan	0.01	2-Furanmethanol (CAS) Furfuryl alcohol	$C_5H_6O_2$	98	98-00-0
	Guaiacol	0.06	Phenol, 2-methoxy-(CAS) Guaiacol	$C_7H_8O_2$	124	90-05-1
	Phenols	0.07	Phenol (CAS) Izal	C_6H_6O	94	108-95-2
	syringol	0.10	Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol	$C_8H_{10}O_3$	154	91-10-1
	4-Ethylsyringol	0.02	Phenol, 4-ethyl-2-methoxy- (CAS) 2,6-Dimethoxyphenol	$C_{10}H_{14}O_3$	182	14059-92-8
	Mixed oxygenates	0.07	2-Propanone, 1-hydroxy-(CAS) Acetol	$C_3H_6O_2$	74	116-09-6

Table 4 Group of individual organic compounds in tar compounds produced from pyrolysis of EFB2 sample

Tar classes	Compounds	Conc. (%)	Name	Chemical formula	MW (g mol^{-1})	CAS
The primary group of tar compounds (Class 2)	Acid	0.01	Hepatonic acid (CAS) Heptoic acid	$C_7H_{14}O_2$	130	111-14-8
	Guaiacol	0.01	2-Methoxy-4-methylphenol	$C_8H_{10}O_2$	138	93-51-6
	Guaiacol	0.01	Phenol, 4-ethyl-2-methoxy- (CAS) p-Ethylguaiacol	$C_9H_{12}O_2$	152	2785-89-9

forms a meth-methoxy (S)-2-furanethanol and tetrahydro-2,5-dimethoxy-furan compound. Whereas, lignin decomposition consists of relatively complex depolymerization, dehydration, cracking and hydrogenation process. Lignin is depolymerized and dehydrated to propenyl-guaiacol at low temperature range of 329-350 °C. Propenyl-guaiacol can then be hydrogenated to propyl-guaiacol at 350-400 °C. Lignin and guaiacol cracks occur at 350-471°C and the position of the damaged C-C bond is strongly related to temperature. In addition, the breaking of the C_β-C_γ bond occurs at 350 °C, followed by the breaking of the C_α-C_β bond at 400 °C compared to the C_4-C_α bond at 450 °C. Finally, cleavage of C-O-CH3 occurs at 471 °C (Zhang *et al.* 2017).

Condensation Temperature

Condensation temperature is one of the physical properties possessed by a mixture of two or more types of compounds in a gas. This temperature indicator is used to predict when the gas mixture will condense under predetermined conditions. Thus, it can be assumed that tar will first condense at this temperature. From the identification data of pyrolysis gas composition categorized as tar compounds, as shown in Table 3, Table 4 and Table 5, the condensation temperature can be predicted using equations 1 to 4. The constant values of Antoine (A, B and C) is determined based on the type of the compound, as in Table 6. The results of the analysis to predict saturated vapor pressure, condensation pressure and condensation temperature of compounds can be seen also in the table.

Concentration of the compound at the calculated condensation temperature were 1720.79 mg $Nm^{3\ -1}$, 92.97 mg $Nm^{3\ -1}$ and 359.02 mg $Nm^{3\ -1}$, for each sample respectively. Relationship of the concentration and condensation temperature of tar compounds in each of EFB samples can be seen in Figure 3. The difference in the condensation temperature and tar concentration in the EFB sample is influenced by the physical properties of the EFB. This study found that condensation temperature of sample EFB1 was 204.22 °C with tar concentration of 1720.79 mg $Nm^{3\ -1}$, sample EFB2 was 256.02 °C with tar concentration 92.97 mg $Nm^{3\ -1}$, sample EFB3 was 154.85 °C with tar concentration 359.02 mg $Nm^{3\ -1}$. For the purpose of eliminating the tar from the pyrolysis gas, it needs to be condensed at that temperature range in a condenser.

Table 5 Group of individual organic compounds in tar compounds produced from pyrolysis of EFB3 sample

Tar classes	Compounds	Conc. (%)	Name	Chemical formula	MW (g mol^{-1})	CAS
The primary group of tar compounds (Class 2)	Phenols	0.0210	Phenol (CAS) Izal	C_6H_6O	94	108-95-2
	Guaiacols	0.0029	2-Methoxy-4-methylphenol	$C_8H_{10}O_2$	138.00	93-51-6
	Guaiacols	0.0037	Phenol, 4-ethyl-2-methoxy- (CAS) p-Ethylguaiacol	$C_9H_{12}O_2$	152	2785-89-9
	Syringols	0.0157	Phenol, 2,6-dimethoxy- (CAS) 2,6-Dimethoxyphenol	$C_8H_{10}O_3$	154	91-10-1
	Mixed oxygenates	0.0088	2-Propanone, 1-hydroxy- (CAS) Acetol	$C_3H_6O_2$	74	116-09-6

Table 6 Results of predictive analysis, condensation pressure, condensation pressure and temperature

Sample	Compounds	Chemical formula	CAS	Antoine constant*)			P_{sw} (mmHg)	P_{dp} (mmHg)	T_{dp} (°C)
				A	B	C			
EFB1	Furan	$C_5H_6O_2$	98-00-0	8.21	2120.79	227.76	67238.49	31753.63	204.22
	Guaiacol	$C_7H_8O_2$	90-05-1	7.90	2203.80	234.22	26482.01		
	Phenols	$C_9H_{12}O_2$	108-95-2	8.92	2943.09	252.10	25681.14		
	syringol	$C_8H_{10}O_3$	91-10-1	7.06	1618.53	186.48	19852.11		
	4-Ethylsyringol	$C_{10}H_{14}O_3$	14059-92-8	7.84	2547.16	232.05	6396.09		
	Mixed oxygenates	$C_{11}H_{14}O_3$	116-09-6	8.20	3604.78	373.75	3472.83		
EFB2	Acid	$C_7H_{14}O_2$	111-14-8	7.38	149.41	373.75	15202439.93	10472.14	256.02
	Guaiacol	$C_8H_{10}O_2$	93-51-6	8.62	3468.88	382.59	15340.48		
	Guaiacol	$C_9H_{12}O_2$	2785-89-9	8.92	2943.09	252.10	25681.14		
EFB3	Phenol	C_6H_6O	108-95-2	7.37	1629.40	181.37	27551.75	114559.27	154.85
	Guaiacols	$C_8H_{10}O_2$	93-51-6	8.62	3468.88	382.59	3472.83		
	Guaiacols	$C_9H_{12}O_2$	2785-89-9	8.92	2943.09	252.10	3472.83		
	Syringols	$C_8H_{10}O_3$	91-10-1	7.13	1945.46	197.36	3472.83		
	Mixed oxygenates	$C_3H_6O_2$	116-09-6	8.79	2292.46	241.70	25681.14		

*) Yaw CL (2009)

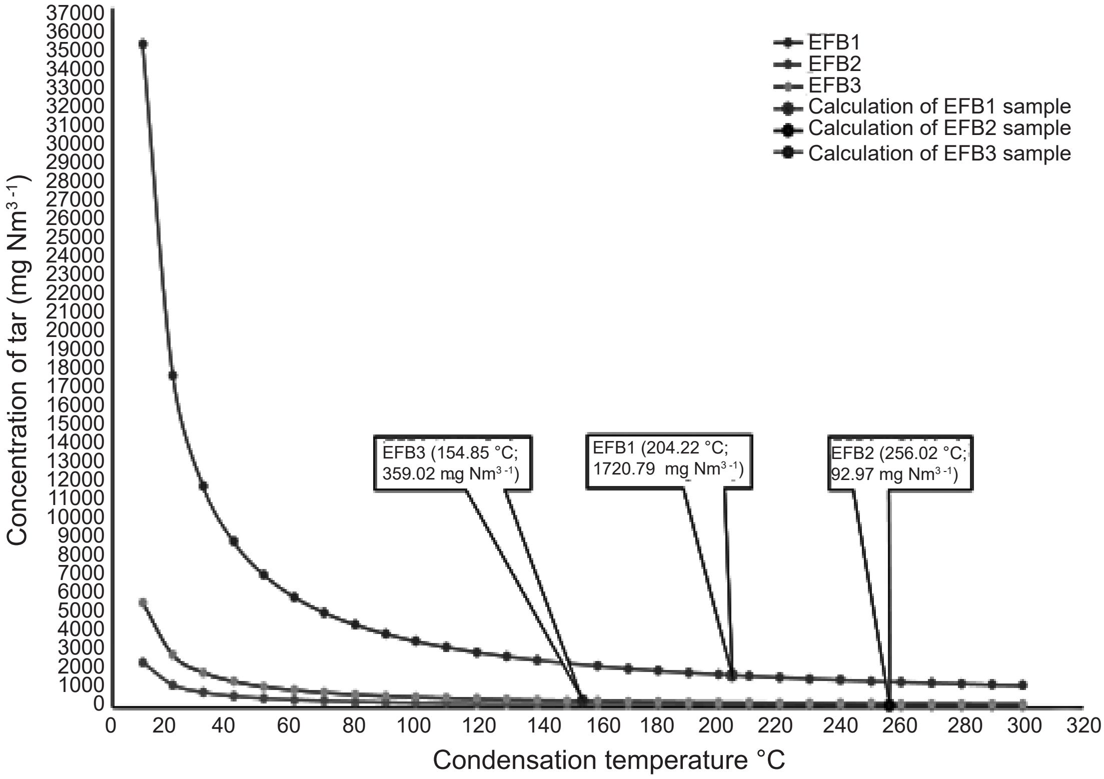

Figure 3 Tar concentration and condensation temperature of pyrolysis EFB .

CONCLUSIONS

The study can be concluded as follows: the proximate analysis is displayed in wet bases (wb), while the ultimate analysis in dry bases (db). The composition of the efb was found to be 52±8% (c), 7.1±0.5% (h), 2.3±0.4% (n), 0.5±0.2% (s) and 35±4 (o); while the ash was 6±2%, fixed carbon (fc) 17±1% and volatile matter (vm)

73±4%; tar compounds produced from pyrolysis at 400 °C, in the form of an oxygenate mixture are classified as first class (class 2) tar compounds which generally consist of phenol compounds (ketones, phenols, and guaiacols); the condensation temperature and the amount of tar concentration in the gas from the pyrolysis of the EFB1sample are 204.22 °C, tar 1720.79 mg $Nm^{3\ -1}$); 256.02 °C, tar 92.97 mg $Nm^{3\ -1}$ (EFB2 sample); and 154.85 °C, tar 359.02 mg $Nm^{3\ -1}$ (EFB3 sample).

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