

Properties of Co-Pyrolysed Palm Kernel Shell and Plastic Grocery Bag with CaO as Catalyst

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Abstract. This study aims to improve quality of pyrolytic oil by co-pyrolysis of palm kernel shell and grocery bag plastic waste with additional CaO as a natural catalyst. Palm Kernel shell was mixed with LDPE grocery bag in the ratio of 1:1 and additional CaO was presented in various weights. The pyrolysis of pure Palm Kernel shell and LDPE grocery bag their self were also observed. The raw material mixture then was heated in the fixed bed pyrolyzer in various temperatures, namely 400 to 500 °C. The obtained pyrolytic oil was analyzed to determine its physical and chemical properties such as density, viscosity, acidity, caloric value and its chemical composition. The results revealed that the increasing pyrolysis temperature affected the increasing of pyrolytic oil yield, from 25.3 % to 48.3 %, with reducing of uncondensable gas yield, from 44.8 % to 25.0 %. The various temperatures also affected the physical and chemical properties. The density increased with the increasing of pyrolysis temperature, however, the pH and caloric value decreased due to the increasing of oxygenated compounds. The pyrolytic oil viscosities were various from 1.4 to 156.4 cP at 60 °C.

1. Introduction

Increasing of energy demand and depletion of energy resources encourages the research to develop alternative fuel which has environment friendly to substitute conventional energy from fossil. One of the alternative energy resources is biomass which abundantly available in the worldwide. Indonesia has abundant biomass resources derived from the solid residue of palm oil production. In 2015, Indonesia has Palm Kernel plantations area of 11.3 million hectares approximately to produce 37.5 million tonnes of palm oil which leaves 8.4 million tonnes of Palm Kernel Shell as solid residue [1]. Palm Kernel Shell is one of high carbon content solid residue which is currently used as a combustion fuel with very limited efficiency [2]. With the modern pyrolysis technology, Palm Kernel Shell can be converted to pyrolytic oil, the useful form of energy.

Pyrolysis is a thermal degradation of material in absence of oxygen to produce pyrolytic oil, char and gas products. Despite pyrolytic oil produced from pyrolysis is a promising alternative fuel, the utilization as liquid fuel is very limited due to the high oxygen content, high viscosity, corrosion and thermal instability [3]. The high oxygen content in the pyrolytic oil provides the low of the heating value.

In another hand, plastic waste increases with the increasing of plastic utilization as grocery bag and bottle. Finally, it is thrown away to the ocean. Indonesia is the biggest country that threw plastic waste to the ocean after China [4]. Plastic is a polymer derived from crude oil which has high heating value namely 42.1 to 49.4 MJ/kg [5]. Pyrolysis of plastic produces liquid products dominated by hydrocarbon



compounds with low oxygenated compounds so that the oil products have a high heating value and low degree of acidity.

Co-pyrolysis is interesting to be studied furthermore. Co-pyrolysis with the blending of biomass and plastic can improve the overall efficiency and increase the quantity of liquid product [6]. Co-pyrolysis of *Laminaria Japonica* and polypropylene resulted pyrolytic oil with the lower water content compared to biomass and increases the hydrocarbon compounds in gasoline groups [7]. Co-pyrolysis of biomass and plastic resulted pyrolytic oil with the high content of aliphatic hydrocarbon compounds and high heating value namely 36.6 MJ/kg [8]. The blending of biomass and plastic in pyrolysis could reduce the oxygenated compounds, acid and phenol on the pyrolytic oil [9]. The conclusion from the previous studies is co-pyrolysis can improve both quality and quantity of pyrolytic oil.

The other method to improve the quality of pyrolytic oil is involving catalyst on the pyrolysis process. Catalysts usually involved in the pyrolysis process are HUSY, Hbeta, Zeolite Y, ZSM-5 and Ni/Al₂O₃ [10]. Since the high price and limited availability of synthesis catalysts, utilizing of natural catalysts such as calcium oxide is an appropriate selection. Calcium oxide contributes to the cracking of heavy compounds to the light oxygenated compounds [11]. In addition, calcium oxide is an effective catalyst to reduce the acid compounds in the pyrolytic oil [12].

Base on the previous studies that Indonesia has abundant resources of Palm Kernel Shell waste and the urgent to reduce plastic grocery bag waste, the study to utilize the both wastes is interesting to be conducted. The involving CaO as catalyst in the pyrolysis is also interesting to be studied furthermore for improving the quality of pyrolytic oil product.

2. Methods

2.1. Material preparation

Palm Kernel shell was obtained from Pandeglang, Indonesia and sieved to 3-6 mm. Palm Kernel Shell was sun-dried for two days to remove unbound water content. LDPE grocery bag was used in this study. It was cut into 2 cm size or less. Palm Kernel Shell to plastic mixture was prepared at the ratio of 1:1. Pyrolysis of pure Palm Kernel Shell and plastic with additional CaO were also performed. Calcium oxide (CaO) used as a catalyst in pyrolysis experiments was purchased from PT. Brataco Indonesia. The proximate and ultimate analysis of Palm Kernel Shell and LDPE were presented in Table 1.

Table 1. Properties of Palm Kernel Shell and LDPE

Ultimate analysis (% db)	Palm Kernel Shell ^a	LDPE ^b
C	45.1 – 50.73	84.98 – 86.35
H	5.1 – 5.97	13.58 – 14.94
O	40.83 – 49.2	0.0 – 0.08
N	0.36 – 0.56	0.0
S	0.04 – 0.95	0.0 – 0.2
Proximate analysis (% db)		
Volatiles	74.0 – 76.0	99.08 – 99.7
Fixed carbon	22.1 – 23.0	0.0
Ash	1.9 – 3.0	0.3 – 0.92
Organic components (% db)		
Cellulose	20.8 – 27.7	
Hemicellulose	21.6 – 22.3	
Lignin	44.0 – 50.7	
Heating Value (MJ/kg)	19.19	46.15

^a data was provided in references [2], [13] and [14]

^b data was provided in references [15], [16] and [17]

2.2. Pyrolysis experiments

Palm Kernel Shell was blended with plastic grocery bag waste in the various ratio. The total mass of Palm Kernel Shell and plastic grocery bag waste sample was 1000 g. Pyrolysis of Palm Kernel Shell and plastic waste mixtures was conducted using a fixed bed reactor powered by an electrical furnace. The experimental apparatus is shown in Fig. 1. Additional catalyst was varied 0, 20 and 30 % .wt of raw material mixtures. After the sample placed into the reactor, the heat was rapidly applied to the desired temperature (400 to 500 °C). The pyrolysis was maintained in the desired constant temperature until the process completed which signed by vapor stop releasing. The releasing vapor was condensed using water-cooled condenser into a liquid product and it was collected for further analysis. The char yield was obtained by calculating the difference between the mass of the solid residue and the quantity of catalyst added. The liquid product was composed organic compounds and water mixture, and its yield was calculated by the total collected pyrolytic oil weight in the condenser system. The gas yield was calculated by difference. The product yields calculations were expressed in equation 1 - 3.

$$Y_{pyrolytic-oil} = \frac{M_{pyrolytic-oil}}{M_0} \times 100\% \quad (1)$$

$$Y_{char} = \frac{M_{char}}{M_0} \times 100\% \quad (2)$$

$$Y_{gas} = 100\% - [Y_{char} + Y_{pyrolytic-oil}] \quad (3)$$

where $M_{pyrolytic-oil}$ is the mass of collected oil product, M_{char} is the mass of char residue and M_0 is the initial mass of raw material fed.

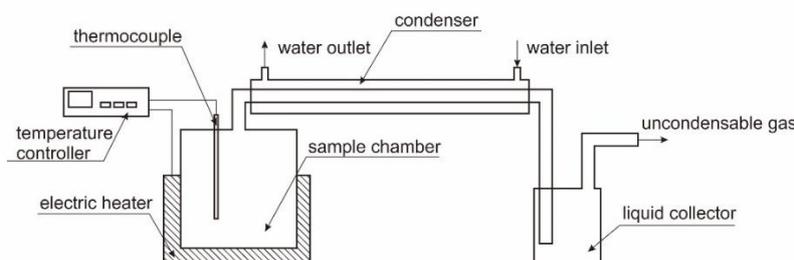


Figure 1. Scheme of experimental apparatus

The collected liquid products were then analyzed their volume and mass to identify the density. The acidity of liquid products are determined by calibrated digital pH meter and those viscosity are determined using rotary digital viscometer (NDJ 8 S Viscometer, WANT Balance Instrument Co., Ltd., Changzhou, China). Amount of 500 ml of pyrolytic oil was located on the glass baker and conditioned at 60 °C using an electric heater. The rotational speed and type of rotor were adjusted to the approximate viscosity. The heating value of liquid products was measured using Bomb Calorimeter Parr 6050 (Parr Instrument Company, Moline, IL, USA). The caloric value was obtained by burning 0.7 ml of top layer pyrolytic oil sample in an oxygen environment in the calorimeter. The components of liquid products are identified using GC-MS (QP2010S Shimadzu, Japan). The oil product detected by GC-MS was used to evaluate the results which divided into two main groups, namely, hydrocarbon and oxygenated compound. Furthermore, they were categorized into subgroups, for example, alcohol, phenolic, acid, aromatic, paraffin, olefin and so on. The percentage of each subgroup yield was defined by totalling of the peak area percentage in the GC-MS spectrum.

3. Results and Discussions

3.1. Product Yields

The yield distribution of co-pyrolysis products such as pyrolytic oil, char and uncondensable gas in various temperatures and conditions is provided in Fig. 1. The pyrolytic oil yield increased with the

increasing temperature. In another hand, the uncondensable gas product decreased with the increasing temperature, while the char product was changing with no significant decrement. High temperature has potential to decompose the raw material larger, therefore, by the increasing temperature resulted in decreasing char product. This phenomenon was also affected by a heating rate. The higher temperature achieved the higher heating rate occurred on the pyrolysis process that produced higher liquid product yield as mentioned also in another study conducted by Li, et al. [18]. At the desired final pyrolysis temperature 400 °C, during one hour process, the temperature inside the pyrolyzer reached 373.0 °C, however, at the final temperature 500 °C, it reached the higher temperature, namely, 460.1 °C. Therefore, pyrolysis at the final temperature 500 °C has higher heating rate than that at 400 °C, so the highest liquid yield product was achieved by pyrolysis at the final temperature 500 °C.

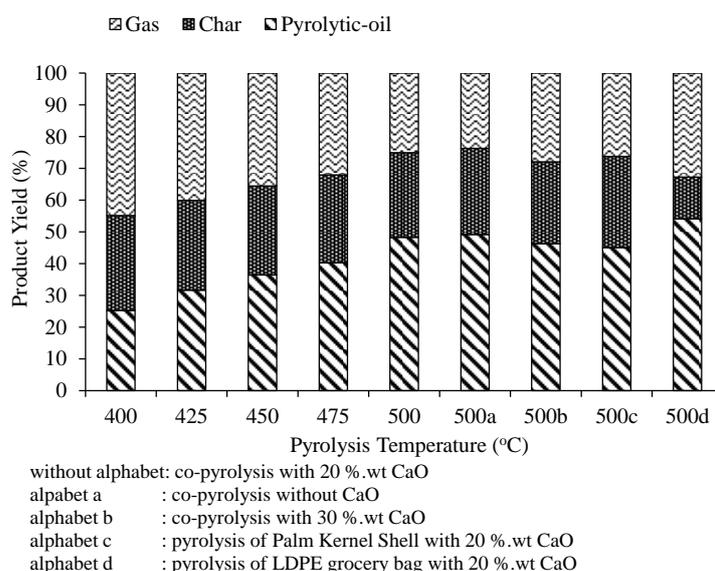


Figure 2. Pyrolysis product yields toward pyrolysis temperatures and conditions

In addition, the higher additional catalyst affected the decreasing of pyrolytic oil yield with the increasing uncondensable product yield as shown in Fig. 1 at 500 °C without alphabet and with the alphabet a and b that indicated pyrolysis with CaO 20%, without CaO and with CaO 30%, respectively. This phenomenon is attributed to the release of oxygen into water form due to the CaO activity that potentially promotes secondary cracking of pyrolytic vapor.

Fig. 1 also shows the pyrolysis product yields with pure Palm Kernel Shell and LDPE grocery bag as raw material with additional CaO. The highest pyrolytic oil product yield was obtained by pyrolysis of pure LDPE grocery bag plastic with the lowest char product yield. Properties of raw material contribute to the products yields. Compared to Palm Kernel Shell, LDPE had higher content of volatile matter. The higher content of volatile matter promotes the higher liquid and uncondensable gas products yield.

3.2. Pyrolytic Oil Properties

Liquid is an interesting form of energy that easy to be used and stored. The properties is important to be considered because every combustion equipment requires appropriate fuel properties such as density, viscosity and heating value. Fig. 3-5 represent the properties of pyrolytic oil with various temperatures and conditions. Distribution of pyrolytic oil density is shown in Fig. 3. The density was higher by the higher pyrolysis temperature. The higher temperature contributed to the more degradation of the raw material, especially for Palm Kernel Shell that has high lignin content that requires high temperature to decompose into pyrolytic vapor. High density is contributed by the presence of amount oxygenated compounds contained in pyrolytic oil in which oxygen has higher atomic mass than carbon and hydrogen. Therefore, the higher temperature promotes Palm Kernel Shell to release the pyrolytic vapor that rich of the oxygenated compound that has high density. The lowest and highest density of pyrolytic

oil were obtained from pyrolysis pure Palm Kernel Shell and LDPE grocery bag, namely, 1001.3 kg/m³ and 775.07 kg/m³, respectively. The study reported by Abnisa, et al. [19] reported that the density of pyrolytic oil from Palm Kernel Shell was 1051 kg/m³.

Fig. 3 also showed that the pyrolytic oil density produced by several additional CaO had no significant difference than others. In general, CaO works through three pathways to remove acid: neutralization, thermal cracking and catalytic cracking reactions as shown in equations 4-7 [12]. Acid can be converted to calcium carboxylate with the releasing of water. Calcium carboxylate then decomposes to calcium carbonate and ketones. Acid can also cracks to be hydrocarbons and carbondioxides by thermal cracking reaction. In Addition, CaO can remove the acid compounds by catalytic cracking that produces calcium carbonate and hydrocarbons. In thermal cracking, heavy hydrocarbon can be converted to light hydrocarbon that has low density and viscosity. However, CaO also provides the neutralization reaction with the releasing of water that has a high density.

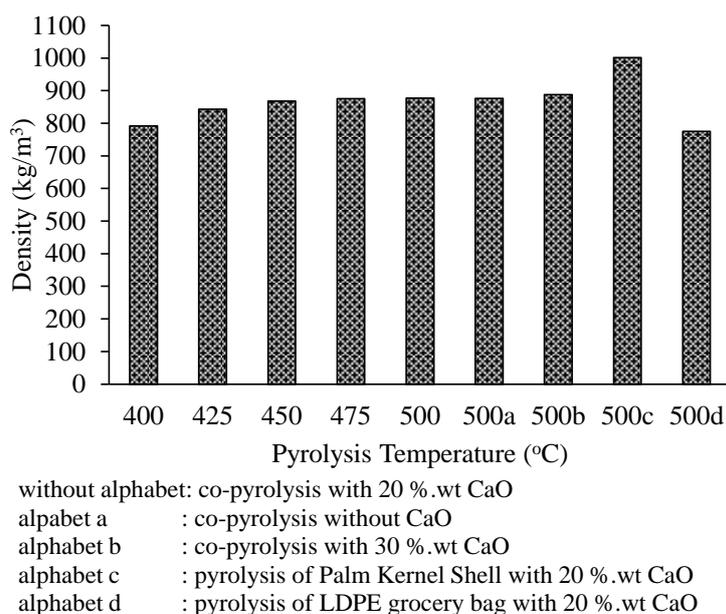
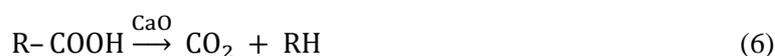
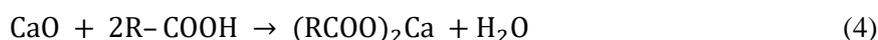


Figure 3. Density of pyrolytic oils towards pyrolysis temperatures and conditions

Viscosity of pyrolytic oil had various value, ranging from 1.4 to 156.4 cP at 60 °C depending on its pyrolysis temperature and conditions as shown in Fig. 4. Pyrolytic oil viscosity measurement required heating the oil until 60 °C due to its great viscosity. As shown in Fig. 4 that temperature affected the viscosity of the pyrolytic oil. The highest viscosity was produced by co-pyrolysis at 450 °C. This phenomenon was attributed to the wax formation on the co-pyrolysis process. The wax formation increased with the increasing temperature, however, when temperature increase higher than 450 °C, the wax formation decreased due to the secondary reaction on pyrolysis. Therefore, the viscosity of pyrolytic oil was lower with the greater temperature.

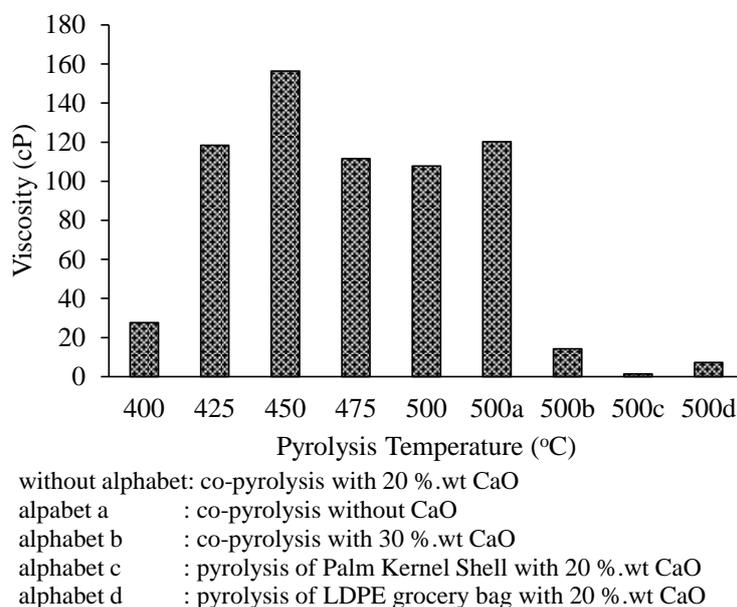


Figure 4. Dynamic viscosity of pyrolytic oils towards temperatures and conditions

In general, wax formation occurs on co-pyrolysis of biomass or coal with plastic base material and it is not formed on pyrolysis of pure biomass itself. The wax is contributed by melting plastic covering the Palm Kernel Shell surfaces that hold up to the condenser by pyrolytic vapor of Palm Kernel Shell force. Palm Kernel Shell consists of hemmicellulose, cellulose and lignin that decomposes at wide range temperature. Hemmicellulose and cellulose decompose at low temperature (lower than 400 °C) and lignin decomposes at high temperature. In other hand, LDPE decomposes at high temperature [17]. Therefore, Palm Kernel Shell decomposes faster than LDPE that those pyrolytic vapor hold up the melting plastic to the condenser and collected as pyrolytic oil so that the viscosity of pyrolytic oil obtained from co-pyrolysis of Palm Kernel Shell and LDPE was high. The phenomenon was illustrated in Fig 5. The prominent viscosity reduction was obtained by co-pyrolysis with additional CaO 30%. Its additional catalyst could reduce viscosity from 120.3 cP to 14.2 cP due to CaO activity that cracks heavy compounds to the light oxygenated compounds so that the viscosity becomes low.

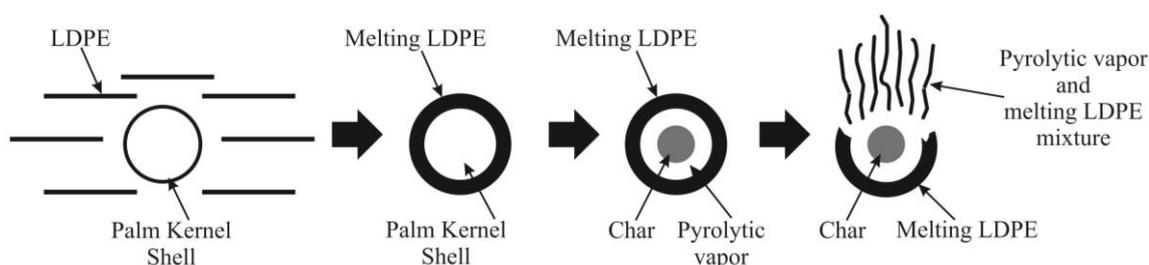


Figure 5. Illustration of co-pyrolysis Palm Kernel Shell and LDPE phenomenon

Acidity of a liquid fraction can be determined in pH unit. The pyrolytic oil is generally acid and it makes pyrolytic oil difficult to be used in boilers, engines, and refinery equipment due to its corrosivity. Fig. 6 represents the relationship between pyrolysis parameters and pH. The temperature was not affected significantly the acid removal. However, additional CaO made the pyrolytic oil had higher pH than that without additional CaO. CaO is a natural base catalyst that performs to remove acid from the pyrolytic oil. The pyrolytic oil obtained from co-pyrolysis at 500 °C without CaO, with CaO 20% and with CaO 30% had pH 3.6, 4.1 and 4.7, respectively. Pyrolysis of pure Palm Kernel Shell and LDPE grocery bag with additional CaO 20% had pH 3.5 and 6.9, respectively. The acidity of pyrolytic oil is mainly due to the presence of organic acids such as acid and phenolic groups, which are the major components of

pyrolytic oil. The phenolic compounds are mainly obtained from the decomposition of lignin, the main ingredient of Palm Kernel Shell.

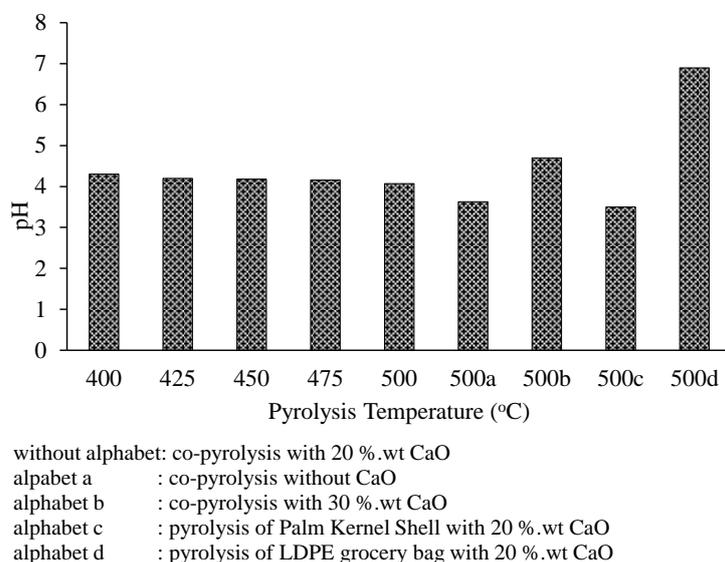


Figure 6. pH of pyrolytic oils towards pyrolysis temperatures and conditions

Heating value is also the important qualification of pyrolytic oil. The heating values of pyrolytic oil in this study were vary depending on its pyrolysis temperature and conditions as exhibited in Fig. 7. The increasing temperature from 400 °C to 500 °C caused the decreasing of heating value from 44.3 MJ/kg to 39.35 MJ/kg. The high temperature provides high energy to decompose raw material, especially for Palm Kernel Shell that needs high temperature to degrade. The higher temperature makes Palm Kernel Shell decompose larger that releases rich oxygenated compounds of pyrolytic vapor. Oxygenated compounds in pyrolytic oil cause the decrement of heating value. Fig. 8 shows that the oxygenated compounds increased from 14.24 % to 24.42 % at co-pyrolysis temperature 400 °C to 500 °C. The highest heating value was obtained by pyrolysis of pure LDPE grocery bag, namely, 45.89 MJ/kg, while, Palm Kernel Shell had the lowest heating value, namely, 18.37 MJ/kg. The study obtained by Hussain, et al. [20] resulted that the heating values of pyrolytic oil from Palm Kernel Shell were 11.94 MJ/kg.

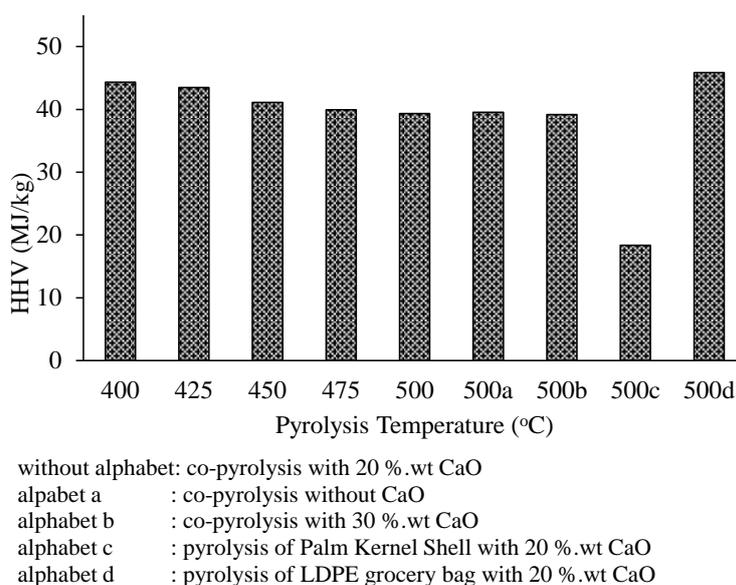


Figure 7. Heating value of pyrolytic oils towards pyrolysis temperatures and conditions

3.3. Pyrolytic Oil Compositions

Table 2 represents the main compositions of pyrolytic oil obtained from many parameters. The major components of pyrolytic oil obtained from pure Palm Kernel Shell were phenol and acetic acid. Phenol is primarily obtained from the decomposition of p-coumaryl alcohol derived from the lignin. Lignin is a different chain from hemicellulose and cellulose. It consists of phenyl propane monomers and it has a complex structure with multiple functional groups, such as methoxy, hydroxy and carbonyl. The main components of all type of lignin were p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol [13]. In other hand, acetic acid is not obtained from lignin decomposition. However, it obtains by significant cracking of the O-acetyl groups in the cellulose and hemicellulose in the raw material [21].

Table 2. Main chemical constituents of pyrolytic oil

Components	Pyrolysis Temperature (°C)						
	400	450	500	500a	500b	500c	500d
Acetic acid	-	-	-	-	-	22.33	-
1-Hexene	1.95	1.67	3.47	-	-	-	3.08
Benzene	2.40	2.00	0.38	0.91	2.46	-	1.5
1-Heptene	2.09	1.86	-	4.64	2.63	-	4.03
Toluene	3.93	3.51	0.45	2.19	4.08	-	2.5
1-Octene	2.69	2.51	1.81	3.04	2.21	-	2.99
Octane	1.61	1.52	0.99	1.55	1.6	-	1.94
2-Furancarboxaldehyde	0.60	0.56	-	-	0.57	6.16	-
2,4-Dimethyl-1-heptene	3.00	2.80	0.84	2.59	2.74	-	-
Ethylbenzene	0.86	0.81	0.15	0.53	0.94	-	0.31
Cyclohexanemethanol	0.32	0.29	-	0.31	0.2	-	-
1-Nonene	4.22	3.39	2.28	4.08	4.41	-	3.43
Nonane	1.99	0	1.76	1.77	-	-	1.72
Phenol	4.28	4.29	0.73	2.67	5.97	31.35	-
1-Decene	4.88	4.51	3.87	5.23	3.97	-	-
Decane	4.70	1.65	1.87	1.56	2.09	-	1.86
Indene	0.57	0.53	-	0.3	0.86	-	-
Mequinol	1.50	1.46	-	-	-	-	-
1-Undecene	3.53	3.42	3.84	0	2.47	-	5.9
Undecane	1.92	1.89	5.54	0	2.15	-	8.93
1-Dodecene	4.61	4.40	5.40	0	8.5	-	14.64
Dodecane	5.13	8.16	3.88	0.23	6.32	-	2.71
1,13-Tetradecadiene	1.82	0.70	4.00	2.3	1.5	-	-
1-Pentadecene	7.95	5.84	3.83	-	5.36	-	-
1-Hexadecene	2.61	4.15	7.64	0.62	1.42	-	9.66
Pentadecane	2.59	-	0.80	-	-	-	-
1,15-Pentadecanediol	1.13	1.55	8.50	2	0.78	-	1.26
1-Octadecene	0.88	2.31	0.33	3.5	0.75	-	1.69
1-Docosene	1.17	3.92	0.80	-	1.25	-	-
Heptadecane	0.76	3.05	5.45	0.4	-	-	2.22
Tetratetracontane	0.19	0.23	-	-	0.64	0.29	-

without alphabet : co-pyrolysis with 20 %.wt CaO

alphabet a : co-pyrolysis without CaO

alphabet b : co-pyrolysis with 30 %.wt CaO

alphabet c : pyrolysis of Palm Kernel Shell with 20 %.wt CaO

alphabet d : pyrolysis of LDPE grocery bag with 20 %.wt CaO

The pyrolytic oil obtained from LDPE grocery bag was dominated by alkenes and alkanes chains such as 1-Dodecene, 1-Hexadecene, 1-Heptene and Undecane as listed in Table 2. Plastic is produced by

polymerization of hydrocarbon. It indicates that the decompositions of LDPE obtain significant quantities of hydrocarbon in pyrolytic oil. The mixture of raw materials in pyrolysis results in hydrocarbon and oxygenated compounds mixtures such as benzene, toluene, 1-nonene, 1-decene, decane, 1-pentadecene, phenol and 1,15-Pentadecanediol.

In general, the higher temperature obtains the decreasing of hydrocarbon with the increasing of oxygenated compounds as represented in Fig. 8. The highest hydrocarbon composition was obtained by pyrolysis of pure LDPE grocery bag, and pyrolysis of pure Palm Kernel Shell consisted of little compounds of hydrocarbon with the oxygenated compounds domination. The prominent phenomenon in this trend was that by the increasing temperature olefin compounds decreased with the increasing of paraffin compounds. The additional CaO was not affected significantly the changes of olefin and paraffin compositions. Olefin consists of double bonds of hydrocarbon that may disengage due to the energy absorption in the compound that breaks the double bonds into a single bond and it furthermore forms paraffin compounds.

Fig. 8 also represents the pyrolytic oil composition groups of co-pyrolysis of Palm Kernel Shell-LDPE grocery bag and pyrolysis of pure Palm Kernel Shell. The blending feeding favored reductions of acid, phenolic and ketone groups with the alcohol group improvement. Hydrogen transfer from the scission of LDPE can be provided during pyrolysis that promotes the degradation of oxygenated compounds groups such as acid, phenolic and ketone. The pyrolytic oil obtained from co-pyrolysis of Palm Kernel Shell and LDPE grocery bag contained much smaller quantities of oxygenated compounds which indicates that co-pyrolysis is compatible to improve the quality of pyrolytic oil. In another hand, the alcohol group of pyrolytic oil increased. It can be explained that the activated radical site of hydrocarbons from LDPE grocery bag contributes in the interaction with hydroxyl fragments from the initial cellulose degradation in biomass structures that results in the formation of alcohol [22].

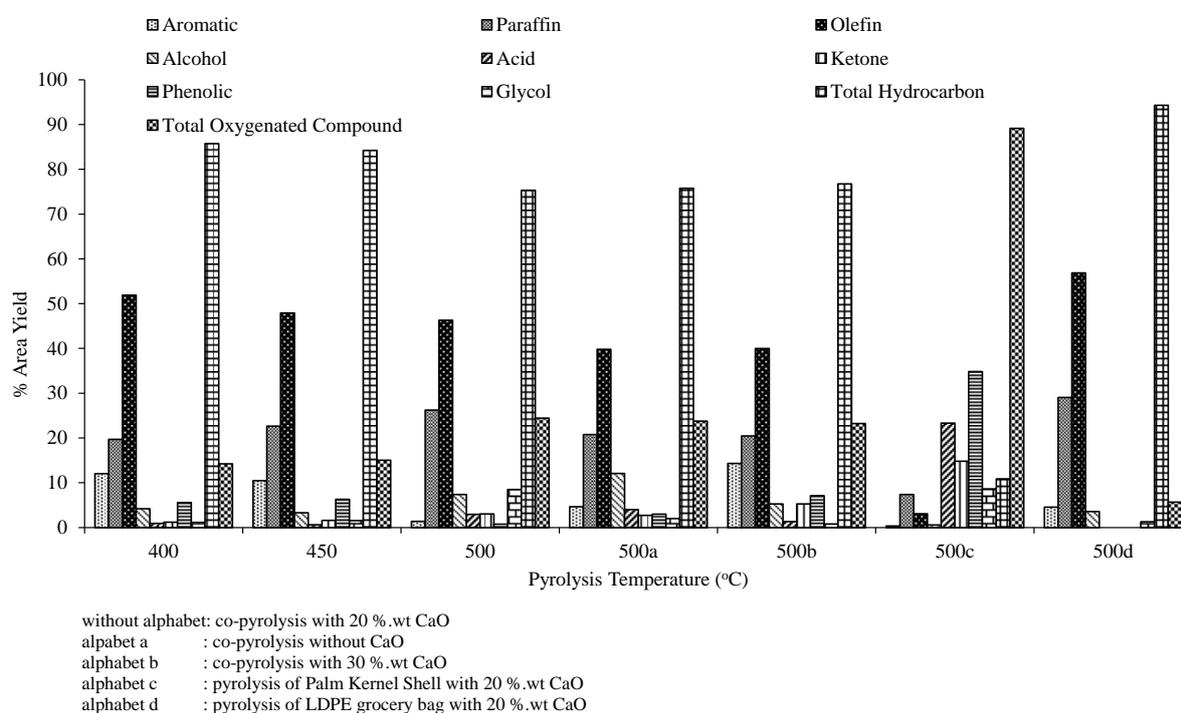


Figure 8. Composition groups of pyrolytic oil

4. Conclusions

In this study, properties of pyrolytic oil were affected by many parameters such as temperature, raw material and catalyst involved. The greater temperature favored higher liquid yield with lowering gas and char product yields. The higher temperature also provided decreasing of heating value due to the

increasing of oxygenated compounds in the pyrolytic oil. The blending Palm Kernel Shell with LDPE grocery bag could improve the quality and quantity of pyrolytic oil such as pyrolytic oil yield, density, heating value and acidity. However, the blending had the significant effect on the great viscosity of pyrolytic oil due to the wax formation. Additional CaO as catalyst contributed significantly to acid removal and viscosity decrement of pyrolytic oil.

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