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Abstract No : 41/1495494861
Abstract Title : A Comparison of Natural Zeolite and Kaolin as a Catalyst in the Isothermal-Catalytic Cracking of Real MSW to Produce Bio-Oil
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# A Comparison of Activated Natural Zeolite and Kaolin as a Catalyst in the Isothermal-Catalytic Cracking of Real MSW to Produce Bio-Oil

Indra Mamad Gandidi[1], M. Dyan Susila[1], and Heri Rustamaji[2]

[1] Department of Mechanical Engineering, Faculty of Engineering, Universitas of Lampung, Jl. S. Brojonegoro, Bandarlampung 35145, Indonesia
[2] Department of Chemical Engineering, Faculty of Engineering, Universitas of Lampung, Jl. S. Brojonegoro, Bandarlampung 35145, Indonesia.

Carresponding Author: indra.mamad@eng.unila.ac.id

**Abstract**
In this work, the real MSW converted into bio-oil have been investigated by pyrolysis method. The pyrolysis experimental was carried out in the isothermal fixed bed reactor at temperature 400 °C with reaction time 60 minutes. The natural activated zeolite and kaolin catalyst were employed in this process with catalyst-MSW ratio 0.5 w/w and influence of activated catalyst to pyrolysis productivity and bio-oil properties were studied. A comparison result between zeolite and kaoline catalytic pyrolysis was examined and bio-oil product was analyzed by GC-MS method which it can be used to identify carbon number range by percentage of peak areas. From this study, yields of liquid fuel or bio-oil were found around 23.6 and 21.4 for kaolin and zeolite catalytic pyrolysis respectively. The carbon range distribution in bio-oil was 65.38% peak area of gasoline range ($C_5$-$C_{12}$) and 33.49% peak area of diesel range ($C_{13}$-$C_{20}$) for activated kaolin catalytic cracking. For activated zeolite has 51.62% peak area of gasoline range ($C_5$-$C_{12}$) and 48.37% peak area of diesel range ($C_{13}$-$C_{20}$). Based on GCMS result, both of bio-oil products were mostly contains of paraffin and olefin. Regarding to the carbon range, the use of kaolin as catalyst was better than the zeolite to convert the real MSW into bio-oil.

**Keywords: MSW, natural catalyst, pyrolytic bio-oil**

## 1. Introduction

The open dumping system of municipal solid waste (MSW) in many developing country like Indonesia has created a lot of impacts to the people and environment in which this is no longer relevant regarding to the limitations in the urban area [1], a habitat for insects, pests and bacteria that can spread various diseases in the open environment and the piles of garbage in the landfill will produce some leachate that contaminates the ground and surface water [2]. Furthermore, it will be degraded biochemically and produce methane gas into the atmosphere where it is known that the potential of methane gas pollution is about twenty times more dangerous than the pollution by $CO_2$ [3]. In addition, many cities around the world have agreed that MSW is one of major contributors for flood and increased of global warming and greenhouse gas effect as well [4].

On the other hand, MSW is a material that has energy stored in the form of chemical bonds between molecules of carbon, hydrogen and oxygen. When the chemical bonds are destroyed, the organic material will release the chemical energy in the form of gas, liquid and solid which is commonly called biofuel [5,6]

The calorific value content is around 20.57 MJ / kg [7] and thus, MSW is one of the potential energy material that can be converted into bio-energy. Several methods for bio-energy production from MSW have been proposed and one of them is pyrolysis method. The pyrolysis method is a thermal degradation in which MSW is converted into solid fraction, liquid fraction consisting of paraffins, olefins, naphthenes and aromatics and gas at elevated temperature in absence the oxygen [8] and usually, the temperature is used in range 400-600 $^{o}$C. This process is intended to break down the long hydrocarbon chain into short-chain hydrocarbon. Hence, the use of municipal solid waste has appeared eligibility to develop as a raw material for bio-oil production by pyrolysis technique [9-12] and MSW converted into bio-oil have attracted much attention of many people due to easily in stored, transported and upgraded.

Recently, some researchers have focused on using of catalysts to reduce the activation energy on the pyrolysis process and to improve thermal efficiency due to endothermic reaction at high temperature and the decomposition process improvement during catalytic cracking as well. The presence of catalyst will generate the secondary cracking to the solid and liquid. Thus, the higher pyrolytic product can be desired. Chika M, et al. [13] was carried out the catalytic pyrolysis by using Y-zeolit and ZSM-5 as catalyst to convert electric and electronic plastic waste into bio-oil, Aida, et al. [14] by using fluidized catalytic cracking (FCC) catalyst to convert plastic waste, Norbert M, et al. [15] was perform catalytic pyrolysis by Y-Zeolite, FCC, $MoO_3$ and HZSM-5 to convert MSW and MPW, Almeida D and Marque MDF. [16] was perform the comparison study between thermal and HZSM-5-catalytic cracking to the municipal plastic waste, Funda A, et al. [17] was studied of catalyst efficiency (Y-zeolite, β-zeolite, equilibrium FCC, $MoO_3$, Ni–Mo-catalyst, HZSM-5) on the catalytic cracking of MSW and MPW. Anyhow, all of researchers above have used synthetic or commercial catalysts in the pyrolysis process in which it will take the high costs of production caused faster deactivation time of catalyst by MSW impurities or contaminant and it is unfavorable effort in the viewpoint of economic issues.

Nevertheless, the natural catalysts that available and abundant such as zeolite, kaolin, dolomite and etc have been investigated as a catalyst on pyrolysis process instead of commercial catalysts. Wenger Jorn. [18] was performed the experiment of catalytic pyrolysis to produce bio-oil from mixed plastic waste by using clay as catalyst, Kyaw K T, et al. [19] was carried out of pyrolysis experiment to make a comparison between clay and dolomite as a catalyst, Panda KA, et al. [20] was using kaolin in the polypropylene pyrolysed and Mochamad S, et al. [21] was carried out two stage pyrolysis of HDPE and PP as raw material with the natural zeolite to produce of bio-oil. However, most of the papers cited above performed single component pyrolysis or MSW that was specified such as pure and mixed plastic material for bio-oil production. While, in generally, the MSW in developing country characteristically includes a huge variety of mixed waste and it is impossible to collected different kinds separately because of the large quantities. Also, more information due to bio-oil characteristics from the real MSW (unseparated) pyrolysed was necessary before it will be used as an engine fuel. Moreover, natural catalysts have difference characteristics with each other and it is very dependent with the mining location and chemical composition of natural catalyst. For instance, Si/Al ratios in the catalyst will strong effect to the pyrolytic products. Significant effect to the decomposition process will be obtained under lower Si/Al ratio that it will increase the acidity of catalyst [22].

Furthermore, the selection of appropriate natural catalyst for real MSW pyrolysed has been required and so far kaolin and zeolite has not been used as catalyst in the pyrolysis process of real MSW.

Therefore, this research was focused to investigate the productivity and selectivity behavior of natural zeolite and kaolin as a catalyst on pyrolysis process to produce bio-oil from the real MSW in which it is a mixture of some kinds of biomass waste, several plastics waste, textile waste, papers waste and rubber waste. The pyrolysis bio-oil that obtained was analyzed to study the characteristics of bio-oil included the products composition and yield, chemical and physical properties of bio-oil. Afterward, a comparison result between pyrolysis and conventional oil (diesel-48 and gasoline-88) was performed to examine a possibility to substitute the conventional fuels

2. Material and Methods

a. *Waste and catalyst for pyrolysis*

The MSW that was applied in this experiment collected from the final disposal site located on Bakung district, Bandarlampung city. This MSW consist of some kind of waste, i.e. vegetable waste from the traditional market, garden waste, household waste, LDPE, HDPE, paper/cardboard, PS, rubber waste and textile waste. All of the samples were dried around 3-5 days by the sun then separated to account the composition of each component.  The dry MSW was then chopped into smaller pieces than 5-10 cm to provide the proportional size with reactor capacity. The composition (dry basis) and physical form of MSW is shown in Fig. 1.

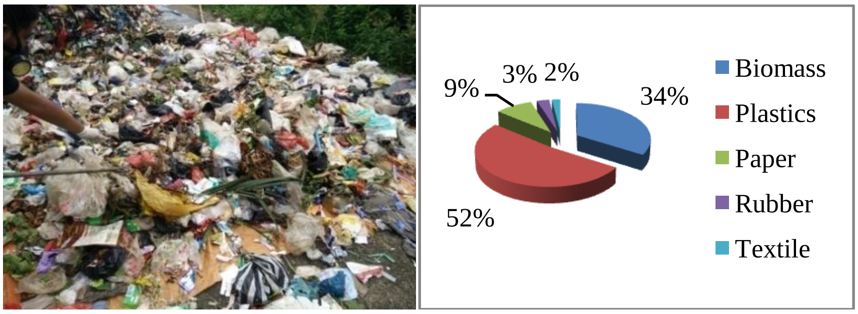


Fig.1. The composition and physical of MSW used in this experiment

The natural zeolite and kaolin that used in this study was obtained from the local source in Lampung province, Indonesia and then grinded by hummer mill and sieved by 20-30 mesh. Afterward, zeolite and kaolin were activated thermally of 500 $^{o}$C for 2 hour to improve the active site and crystallinity behavior of catalyst. To examine the structure and surface of natural zeolite and kaolin, the XRD method was employed and the composition and X-ray patterns of the activated natural zeolite and kaolin was shown in Tables 1 . It could be seen that the hight amount of $SiO_2$ and $Al_2O_3$ compound contented in natural zeolite and kaolin has shown that zeolite and kaolin were very likely to used as a catalyst in the pyrolysis process. These compounds could increase the cracking efficiency and increase the pyrolytic product. Among of them, activated zeolite has seen preferably than the kaolin to decomposition process of real MSW.

Tabel 1. X-Ray analysis for chemical composition of activated zeolite and kaolin

| Kind of Catalyst | Unit | Compound | Concentration |
|---|---|---|---|
| Zeolite | % | $SiO_2$ | 68.5 |
| | % | $Al_2O_3$ | 13.17 |
| | % | $Fe_2O_3$ | 2.98 |
| | % | MgO | 1.15 |
| | % | CaO | 2.47 |
| | % | $K_2O$ | 1.80 |
| | % | $Na_2O$ | 1.06 |
| | % | $TiO_2$ | 0.14 |
| Kaolin | % | $SiO_2$ | 43.12 |
| | % | $Al_2O_3$ | 46.07 |
| | % | $Fe_2O_3$ | 0 |
| | % | MgO | 0.27 |
| | % | CaO | 0.3 |
| | % | $K_2O$ | 0.01 |
| | % | ZnO | 0.0064 |
| | % | $TiO_2$ | 0.74 |

Source: CV. Mina Tama, Bandarlampung

Figure 2. is showing the X-pattern for both of catalyst. The peak sharply was showed by both pattern in which it is implies that natural zeolite and kaolin have a good crystallinity behavior and possibly to give a strong effect in the decomposition of MSW and stable at high temperatures.

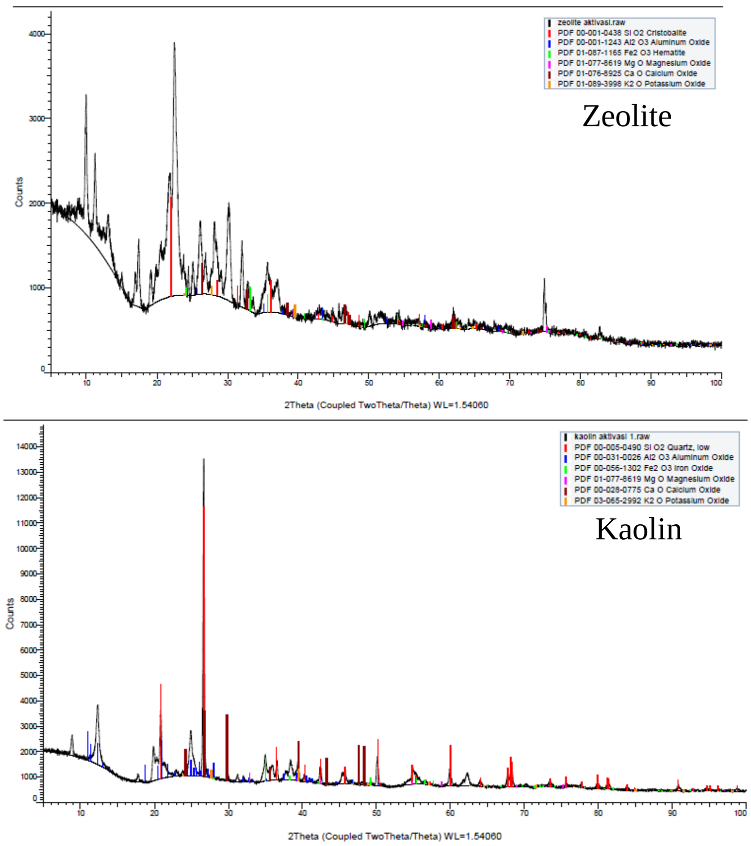

Fig. 2. X-Ray patterns of the activated zeolite and kaolin

*b. Pyrolysis Experiment and Bio-oil Analysis*

The MSW sample was pyrolysed in a laboratory scale using fixed bed vacuum reactor that equipped with air lock valve between reactor and hopper to avoid the air takes the place of entry into the reactor. The reactor is made from the stainless cylinder with the dimension of 310 mm in height, 160 mm in ID and the electrical heating jacket that connected by thermo controlled and thermocouple was taped around the reactor. The reactor was set vertically that connected by nitrogen tube with the rubber tube and then $N_2$ gas was introduced into the reactor for 3 minutes from the top and flow out passed through the top of the reactor. The flow of nitrogen replaces the air from the reactor and permits the pyrolysis reaction under inert vacuum condition.

In the experiment procedure, firstly nitrogen gas was introduce into the reactor and then the reactor was heated until reactor temperature reaches the set value of 400 $^{o}$C with 12 $^{o}$C heating rate and followed by feeding around 750 g of mixed catalyst-MSW were fed into reactor by opened air lock valve. The ratio of catalyst to MWS was 250:500 g. Set the time reaction was 60 minutes for both of pyrolysis experiment. The gas produced from the pyrolysis was flow out from the top of rector and it flow into vacuum gas cleaner as a first condenser then the gas went to shell and tube condensers at 20 °C as a secondary condenser to separate process between permanent gas and liquid. Condensation product from the vacuum cleaner and condenser are mixture of bio-oil and water, then this mixture separated by precipitation and filtration process. The gaseous products were burned off to prevent emission from hydrocarbon gases. The char produced was remained in the reactor and collected after the pyrolysis reaction was finished. The installation of experimental apparatus is shown in the Fig. 3.

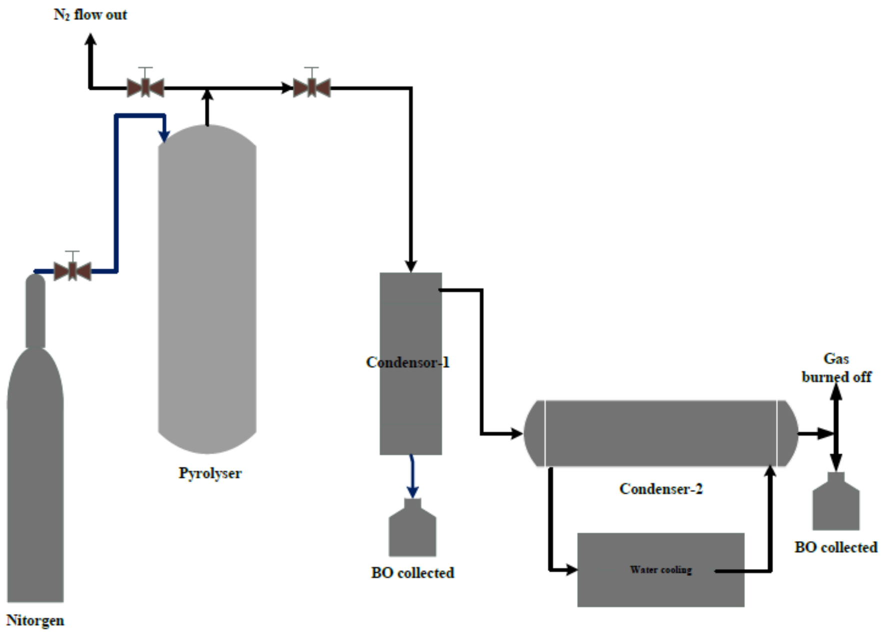

Fig. 3. The installation of experiment apparatus

Regarding to pyrolytic products, the products obtained from the pyrolysis can be divided into non-condensable gas fraction, solid fraction and liquid (bio-oil) fraction. From the bio-oil yield can be recovered hydrocarbon chain in the gasoline range ($C_5$-$C_{12}$), diesel ($C_{12}$-$C_{20}$), heavy oil (> $C_{20}$) that consist of paraffin, olefins, naphthenes and aromatics [23]. In order to examine the bio-oil, pyrolysis liquid was analyzed by using a gas chromatography mass spectrometry (GC-MS, QP2010S Shimadzu) which it can be used to identify carbon number range and chemical composition by percentage of peak areas. The column was DB-1 (Agilent J 100% dimethyl polysiloxane) capillary column, 30 m length with 0.25 mm diameter and 25 µm film thickness. Helium was used as the gas carrier. The temperature was set in initial temperature 60°C for 5 min followed by a heating rate of 5 °C/min to 280 °C and held for 51 min. To estimation the heating value of bio-oil, it was approached with the carbon-hydrogen weigh ratio [24] in which it has been explained that the paraffin has highest heating value and then followed by iso-paraffin, olefin, nepthenes and aromatic (PIONA). Also, the distributions of PIONA in the each range of hydrocarbon were calculated to determine the selectivity behavior of natural zeolite and kaolin catalysts in the pyrolysis process.

## 3. Results and Discussion

### a. *Physical Properties of Bio-oil*

Table 2. is showing the physical properties of bio-oil such as density and viscosity for conventional fuel and bio-oil which it were analyzed based on ASTM methods. It can be seen that the both of bio-oil from the natural catalytic cracking have similar in the density value and significantly different on the viscosity property. The kinematic viscosity of natural kaolin was lower than the natural zeolite. It is indicated that the natural kaolin has good cracking reaction compared the natural zeolite. However, both of bio-oil has properties close to diesel-48 and acceptable to be considered as a future energy. Due to solid and water content in the bio-oil, both of bio-oil have solid and water content highly regarding to impurities in the unwashed real MSW and direct catalytic as well. The very high of solid content in the bio-oil was also be affected by ash from the biomass material.

Tabel 2. The physical properties of bio-oil

| **Propertis** | **Units - Standard** | **Diesel** | **Gasoline** | **Kaolin** | **Zeolite** |
|---|---|---|---|---|---|
| *Cetane Octane Number* | | 48 (*cetane*) | 88 (*octane*) | - | - |
| *Density@15ºC* | g/cm3 ASTM D-1298 | 0,815-0,87 | 0,7-0,78 | 0.8038 | 0.8076 |
| *Kinematic Viscosity@40ºC* | cSt ASTM D-445 | 2,0-5,0 | 0,4-0,8 | 2.0345 | 2.4775 |
| *Flash Point* | ºC ASTM D-93 | min 60 | min 40 | min 5 | min 5 |
| *Pour Point* | ºC | max 18 | - | - | - |
| *Water Content* | %Vol ASTM D-1744 | max 0,05 | - | 3 | 2.5 |
| *Oil Content* | %Vol ASTM D-1744 | - | - | 86 | 90 |
| *Solid Content* | %Vol ASTM D-482 | - | - | 11 | 7.5 |

*b. Products Yield and Chemical Properties of Bio-oil*

The product from the real MSW pyrolysed was shown in Figure 4. Catalytic cracking to real MSW was produced pyrolysis bio-oil around 23.6 and 21.4% by use natural kaolin and zeolite respectively. The natural kaolin has a higher yield compared the natural zeolite. This yield was indicated that natural kaolin has productivity behavior better than the natural zeolite in the pyrolysis process. This behavior can be explained due to its active site and acidity, which facilitate the cracking reaction in the pyrolysis. However, the highest yield on the gaseous product and appearance of water content in the bio-oil was imply that the further drying for real MSW was required and temperature of condensation environment around 20 $^{o}$C was unfavorable. Moreover, the quite low on bio-oil yield can also be associated to the time reaction shortly and long residence time of gas in the reactor [25]. The similar result was also presented by Panda, KA. [26] that use of kaolin as a catalyst in plastic waste pyrolysed.

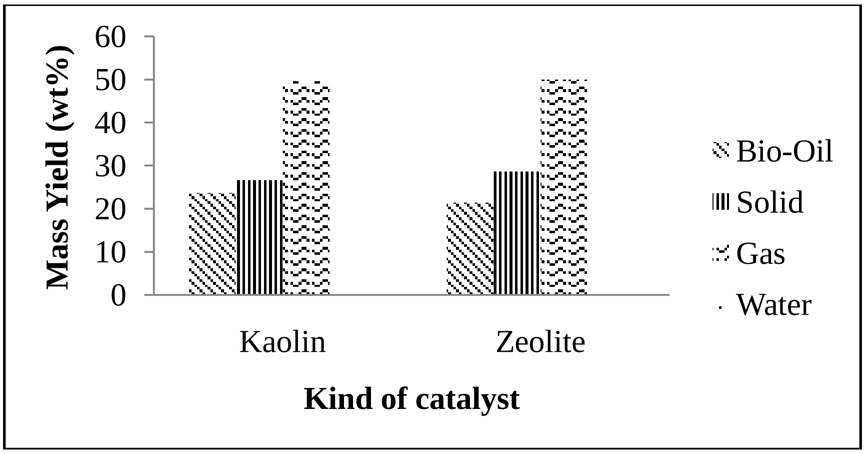


Fig 4. Mass yield of pyrolytic products

Figure 5. is presenting the carbon distribution range in the conventional and pyrolytic oil. The pyrolytic oil from real MSW pyrolysed is mostly contain of gasoline ($C_5$-$C_{12}$) and then followed by diesel ($C_{13}$-$C_{20}$) and small amount of heavy oil (C>20).

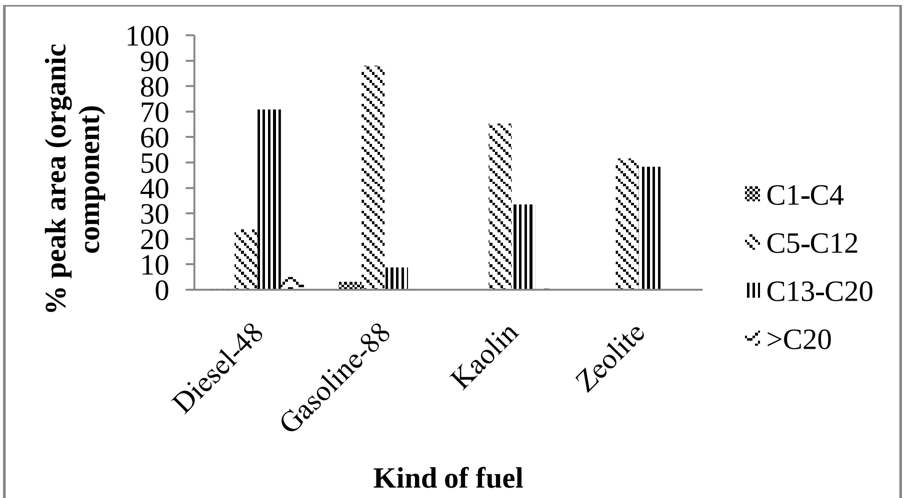


Fig 5. Carbon distribution range on conventional and pyrolysis fuel

Bio-oil by natural kaolin catalytic was consisting of 65.38% peak area of gasoline fraction and 33.49% peak area of diesel fraction. While bio-oil by natural zeolite has 51.62% peak area of gasoline fraction and 48.37% peak area of diesel fraction. Clearly, natural kaolin has shown better properties as a catalyst for the production of bio-oil from municipal solid waste compared with natural zeolite although natural zeolite has not heavy oil fraction in the bio-oil. The microstructure and pore size of the natural catalyst can be used as a reason to this distinction.

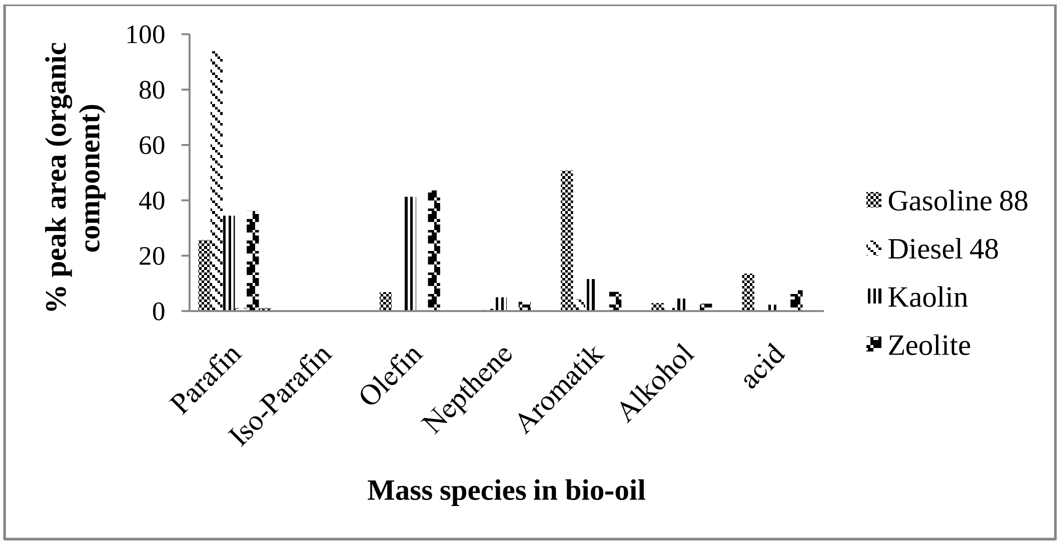


Fig 6. A comparison of mass species between bio-oil and conventional fuel

Hereinafter, figure 6. is reveal the organic component in the bio-oil in which most of organic component in the bio-oil is paraffin and olefin with small amount of aromatic, alcohol and acid.  There is only slight difference in the amount of paraffin and olefin between natural kaolin and natural zeolite in catalytic cracking process. Consequently, bio-oil from natural kaolin and natural zeolite catalytic pyrolysis have the same caloric value and it was illustrates that both of natural catalyst at 400 $^{o}$C and 60 minutes have similarities in the selectivity behavior.

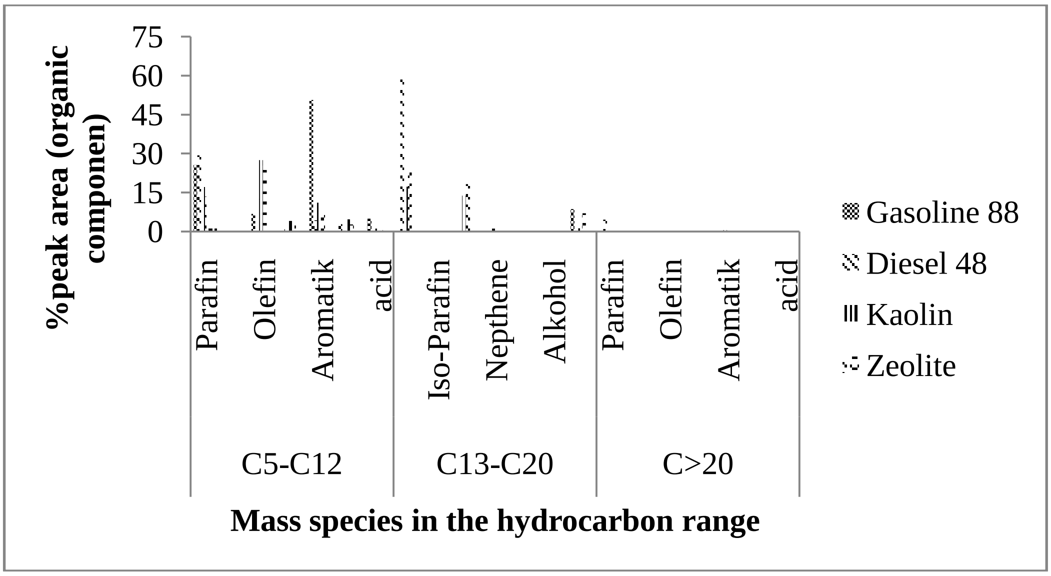


Fig 7. Number of mass species in each hydrocarbon range

Figure 7. is describing the PIONA contents in the each hydrocarbon range on the pyrolytic bio-oil in which shows that gasoline fraction was contain more olefin than the paraffin and otherwise with the diesel fraction for both of pyrolytic bio-oil. This composition was significantly affected by kind of real MSW especially plastics and biomass in which as a determinant of chemical reaction during the process of pyrolysis. The presence of impurities and natural catalyst in the real MSW was also allegedly playing an important role to the bio-oil composition [27]

4. Conclusion

The real MSW is the great potential raw material that can be converted into hydrocarbon range as a paraffin and olefin by thermal-catalytic cracking. By use activated natural catalyst (kaolin and zeolite) can increase the product of bio-oil (productivity behavior) but quite low in the selectivity behavior. Among of them, kaolin catalyst has demonstrated a better capability for pyrolysis process. The upgrading process to enhance the pyrolysis oil yield is necessary to obtain a bio-oil with gasoline range and more. Currently, the influence of time reaction, temperature and particle size of catalyst are investigating. Also double stage pyrolysis is studied.

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