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Experimental study the influence of zeolite size on pyrolysis of low-density polyethylene plastic waste

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Thermal Science and Engineering Progress <em@editorialmanager.com> Sat, Dec 7, 2019 at 4:59 PM
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Manuscript Number: TSEP-D-19-00313
Experimental study the influence of zeolite size on pyrolysis of low-density polyethylene plastic waste

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Thermal Science and Engineering Progress <em@editorialmanager.com> Wed, Jan 8, 2020 at 6:28 PM
Reply-To: Thermal Science and Engineering Progress <tsep@elsevier.com>
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Manuscript Number: TSEP-D-19-00313

Experimental study the influence of zeolite size on pyrolysis of low-density polyethylene plastic waste

Dear Dr. Susastriawan,

Thank you for submitting your manuscript to Thermal Science and Engineering Progress.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following major revision. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by Feb 07, 2020.

When revising your manuscript, please consider all issues mentioned in the reviewers' comments carefully: please outline every change made in response to their comments and provide suitable rebuttals for any comments not addressed. Please note that your revised submission may need to be re-reviewed.

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Kind regards,
Hussam Jouhara, PhD, CEng, FIMechE. FIEI
Associate Editor

Thermal Science and Engineering Progress

Editor and Reviewer comments:

The article covers interesting and important problem which is plastics utilisation. In particular pyrolysis of LDPE seems to be promising option in waste management. To improve the whole process many catalysts were developed. The size of used zeolite particles potentially has got an influence on the pyrolysis effectiveness, and the researchers decided to check this idea. However, there are several serious problems about the submitted article which are listed below. My overall recommendation is MAJOR REVIEW. I would like the authors to address below comments.
Major comments:
1. Page 6 Experimental section.
* "The setup consists of a Stainless Steel pyrolyzer, a Stainless Steel condenser, a water tank, a LPG burner, K-type thermocouples and a data logger "GrapTech." Are those pieces of equipment are typical, made of any company or they were hand-made by the researchers? It is good idea to add additional pieces of information or, if the whole set-up was described elsewhere - citation. What Bomb Colorimeter was used?
* T1 is described as the temperature of pyrolyser but subsequently appears: "pyrolyzer outlet temperature" (line 51) and "The exit vapor temperature" (line 59). My question is: where exactly the thermocouple was placed? Temperature of outlet vapours can be completely different than temperature inside the reactor.
* Fig. 4. According to the kinetics of LDPE pyrolysis (Aboulkas, El and Bouadili, 2010) the process really begins above 300 °C, and adding a catalyst does not change it significantly (Valde, 2007). This brings me to the conclusion that your experiment conditions were not optimally chosen. Thus the process could not run typically and making conclusion based on that data is not convincing to me.
2. This point comes from the previous one. I would like you to justify below statements considering the characteristics and kinetics of the process as well as proper citations.
* Page 7 line 2-4: "This temperature can be used to predict the pyrolysis temperature at the bottom of the pyrolyzer. The pyrolysis temperature should be higher than 155°C."
* Page 7 line 43-47: "However, very high temperatures give negative effect on oil production. It is because at very high temperature there is a secondary cracking of the volatiles, which results in a higher gas yield." - It should be clearly indicated what exactly temperatures is VERY HIGH for LDPE pyrolysis.
* Page 7 line 52-53: "In the present work, the pyrolysis temperature at the bottom part of the pyrolyzer should be higher than 100°C."

* Page 7 line 58 to page 8 line 2: "Pyrolysis temperature ranges are 378-404°C [16] with maximum temperature of 467°C [17] for High-density polyethylene (HDPE), 225-520°C for Polyvinyl chloride (PVC) [18], 425- 500°C for low-density polyethylene (LDPE) [19], and 350-425 for Polystyrene (PS) [20]." This sentence is simply wrong. First part suggest that temperature of pyrolysis of plastics varies from 378 to 404°C, while the source [16] mentioned pyrolysis of HDPE and did not indicate 378-404 as a range of pyrolysis temperature. What is important, [19] do not claim that 425-500 is a maximum temperature for LDPE pyrolysis. Moreover, [17] says: "Thermal degradation of PP has a maximum at around 720 K, while the highest rate of HDPE decomposition occurs at around 740 K."! That means this is the best, optimal temperature for HDPE pyrolysis. The difference between content of cited sources and your statements is huge.

3. Page 9 Fig. 8. How did the measurements of oil amounts was made? What about uncertainty? This cause my concern since you have collected the samples every 15 minutes which gives 12 samples of each oil. If the uncertainty of each separate volume measurement would be ±1ml this could make the results for three different zeolite fractions insignificant.

4. Page 9 line 56 to page 10 line 11. The explanation of the very big difference between result of this work and similar ones did not cover the most important - in my opinion - reason which is too low temperature of LDPE pyrolysis. Work [19] cited in the paper said: "Pyrolysis at 500°C in the absence of any catalyst produced a yield of 95 wt.% oil, negligible char and low gas yield." I am afraid that there is a need to repeat the results under optimal pyrolysis condition or CLEARLY justify why those specific ones were chosen. Maybe there is an explanation why such as conditions were applied.

Minor comments:

1. Page 1 line 59. You claim: "The plastic wastes require billions of years to degrade naturally [2]". My question is: how did you measured this? The above mentioned source said that it may take billions of years to degrade, but there is a huge difference between those two statements. This situation forced me to be very careful about the content of the article and the citations since imprecision like this is unacceptable in a high quality scientific papers. Please, stay consistent with the cited papers and do not overestimate their content.
2. Page 3 line 55-56. There is a language problem with the sentence "The plastic waste mainly comes from bottle mineral water bottles, food plastic packages, and other plastic packages". Should it be "bottled mineral water bottles", should not it?
3. Page 4. Fig. 1. Who is the author of those results? There is a source/sources needed.
4. Page 4 line 34-35. Is it really the MAIN advantage pyrolysis? If you express an opinion, whether your own or someone, you should indicate this.
5. Page 4 line 36, 37: Source [13] do not categorize pyrolysis process this way. Flash pyrolysis is not even mentioned.
6. Page 5 line 2-7: Language problems, repetition is not necessary.
7. Page 5 line 14: IMPROVED not proved
8. Page 5 section Materials: How did you ensure that your waste is LDPE? Did you dry it after washing? How and how long?
9. Page 6 line 6: Quotation mark is missed.
10. Page 9 line 9-13: HHV is not the only one parameter that should be studied prior usage of LDPE pyrolysis oil as a engine fuel.
11. Page 9 line 58-60: Are you using the wt.% or vol.%? Since you measured the volume of oil, there is a little confusion. Are you taking into account the density of the products?
12. Page 10 Fig.11. Is this figure necessary?
13. Page 12 line 45: Wrong year of the article publishing.

References:

Aboulkas, A., El, K. and Bouadili, A. El (2010) 'Thermal degradation behaviors of polyethylene and polypropylene . Part I : Pyrolysis kinetics and mechanisms', Energy Conversion and Management. Elsevier Ltd, 51(7), pp. 1363-1369. doi: 10.1016/j.enconman.2009.12.017.

Valde, F. (2007) 'Catalytic cracking of low-density polyethylene over H-Beta and HZSM-5 zeolites : Influence of the external surface . Kinetic model', 92. doi: 10.1016/j.polymdegradstab.2006.11.007.

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To: "A. A. P. Susastriawan" <agung589e@akprind.ac.id>

Manuscript Number: TSEP-D-19-00313R1

Experimental study the influence of zeolite size on pyrolysis of low-density polyethylene plastic waste

Dear Dr. Susastriawan,

Thank you for submitting your manuscript to Thermal Science and Engineering Progress.

I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following minor revision and modification. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by Mar 04, 2020.

When revising your manuscript, please consider all issues mentioned in the reviewers' comments carefully: please outline every change made in response to their comments and provide suitable rebuttals for any comments not addressed. Please note that your revised submission may need to be re-reviewed.

To submit your revised manuscript, please log in as an author at https://www.editorialmanager.com/tsep/, and navigate to the "Submissions Needing Revision" folder under the Author Main Menu.

Thermal Science and Engineering Progress values your contribution and I look forward to receiving your revised manuscript.

Kind regards,

Hussam Jouhara, PhD, CEng, FIMechE. FIEI

Associate Editor

Thermal Science and Engineering Progress

Editor and Reviewer comments:

Reviewer #1: Thank you very much for addressing almost all my comments. It is possible to accept the revised version into publication. However, I still concern about the unsure temperature of the process that highly possibly is much lower than optimal. Subsequently, your results (especially yields of the pyrolysis products) may confused other researchers without careful reading of the whole paper. Maybe it would be beneficial to change the title in order to give more precise information? I suggest at least adding "low-temperature" before "pyrolysis" in the title. Additionally, I would like you to add a sentence about the influence of process temperature on the products yields in "Conclusion" section, since at the moment your work claims that results are caused by ineffective heat transfer in condenser which is only partially true. The temperature plays major role.

*****

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Experimental study the influence of zeolite size on low-temperature pyrolysis of low-density polyethylene plastic waste

A.A.P. Susastriawan[1*], Purnomo[1], Aris Sandria[2]

[1]Dept. of Mechanical Engineering, Faculty of Industrial Technology, Institut Sains & Teknologi AKPRIND, Indonesia
[2]Undergraduate scholar of Dept. of Mechanical Engineering, Faculty of Industrial Technology, Institut Sains & Teknologi AKPRIND, Indonesia

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ABSTRACT

The plastic wastes may need billions of years to degrade naturally due to their slow degradation rate. The accumulation of the plastic wastes becomes a serious problem. Since a plastic is a polymers of hydrocarbon, it has a potential to be converted into oil fuel. In the present work, zeolite based catalytic pyrolysis of low-density polyethylene (LDPE) plastic waste is performed with different zeolite sizes (i.e. 1, 2, and 3 mm in diameter) at low-temperature. The aim of the work is to investigate the effect of zeolite sizes on pyrolysis of LDPE plastic. The results show that smaller zeolite size increases heat transfer rate, pyrolysis temperature, reaction rate, and oil yield. From 1000 g of LDPE plastic, the oil yields are 138, 134, 126 mL for the use of 1, 2, and 3 mm in diameter of the zeolite, respectively. In order to improve the conversion of vapor into oil, the performance of the condenser have to be improved by lowering the temperature of cooling water or increasing heat transfer surface area of the condenser.

Keywords: plastic; pyrolysis; size; waste; zeolite

1. Introduction

According to Ministry of Environmental and Forestry of Republic of Indonesia [1], plastic waste is the second largest waste after organic waste in Indonesia. As shown in Fig.1, plastic waste grasps 14% of total waste in 2017. The plastic waste mainly comes from bottles of mineral water, food plastic packages, and other plastic packages. The plastic wastes may take billions of years to degrade naturally [2] because their degradation rate is very slow [3]. This leads a huge accumulation in landfill and various natural habitats, i.e. rivers and oceans [4]. This accumulation becomes a problem in the future [5]. Since a plastic is a polymers of hydrocarbon, it has a potential to be converted into oil fuel. One of the promising ways of taking profit of the energetic of the plastic waste is pyrolysis [6]. Pyrolysis is potential process in waste management sector [7]. Many researchers have worked on plastic pyrolysis and have demonstrated the use of the technology to encounter the plastic waste problem [8-11].

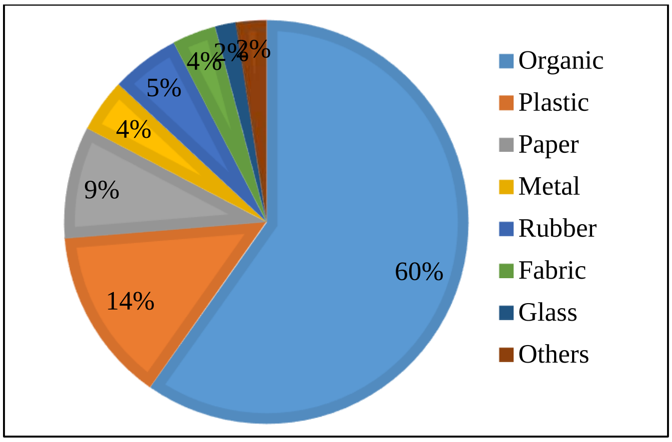

Fig. 1. Percentage of wastes in Indonesia [1]

Pyrolysis is a thermochemical conversion of solid biomass into useful energy in the absence of oxygen, resulting a char, a condensable liquids or tars, and a trace amount of gaseous products [12]. Stated by Dhyani *et al*. [13] and Guedes *et al*. [14], the main advantage of pyrolysis is that liquid fuel produced can be easily stored and transported. Depending on the process conditions, the pyrolysis process can be classified into following categories: fast pyrolysis, intermediate pyrolysis, slow pyrolysis, and hydropyrolysis [13]. Table 1 shows the difference in parameter and major product between slow and fast pyrolysis. Fast pyrolysis is recommended when liquid fuel is the goal of the pyrolysis.

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Table 1. Parameter and major product of the slow, fast, and flash pyrolysis

Parameter	**Pyrolysis**	
	Slow	**Fast**
Temperature	Low	Moderate
Heating rate	Low	High
Residence time	Long	Short
Major product	Char	Liquid

In order to enhance the process and product of pyrolysis, a catalyst is used to increase reaction rate and hydrocarbon distribution in the liquid product and to reduce optimum pyrolysis temperature. Three common catalysts used in the plastic pyrolysis are Zeolite, FCC, and Silica-Alumina catalyst. Among those three, the zeolite catalyst is extensively applied in the plastic pyrolysis [2]. Catalytic pyrolysis of plastic waste using natural and synthetic zeolite catalysts was conducted by Miandad, *et al*. [3]. They obtained that the use of both natural and synthetic zeolite catalysts improved the quality of liquid oil by increasing the light hydrocarbon compounds. Other works on zeolite catalytic pyrolysis have been also reported by other researchers [12, 15].

Several works on the use of zeolite as a catalyst in plastic pyrolysis have been reported, but none of those works discussed the effect of zeolite's particle size on characteristics and product yield of low-density polyethylene (LDPE) pyrolysis. In the present work, zeolite based catalytic pyrolysis of LDPE wastes is performed with different zeolite sizes (i.e. 1, 2, and 3 mm in diameter) at low-temperature. The effect of the particle sizes on characteristics and product yield of LDPE pyrolysis are investigated and discussed. In addition, performance of the condenser is also discussed.

2. Materials and Method

2.1. Materials

LDPE plastic is widely applied as plastic bags [2]. Thus, only waste of plastic bags are collected from local waste disposal site in Yogyakarta to ensure the plastic is LDPE. The plastic bag is washed and sun dried for two days and crushed into smaller pieces as the feedstock of the pyrolyzer. Natural zeolite catalyst is bought from local market and crushed into smaller size of 1, 2, and 3 mm in diameter. Fig. 2 presents the photograph of the LPDE plastic and the zeolite.

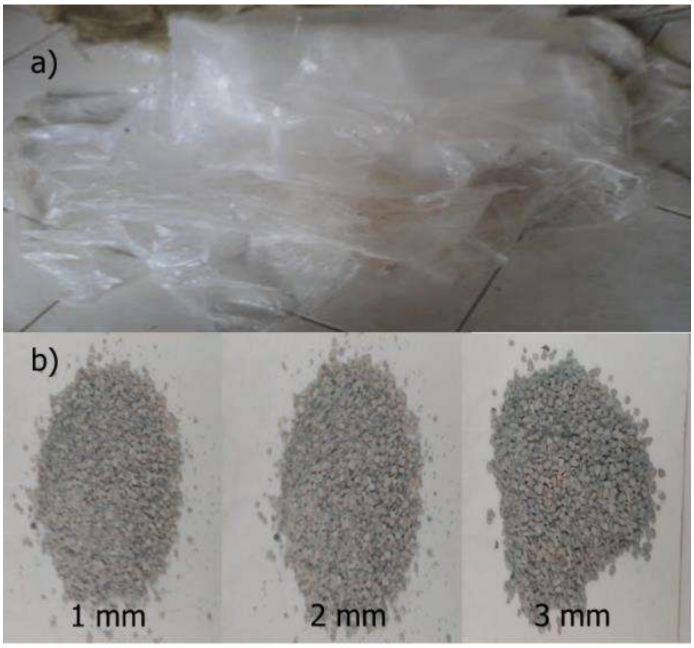

Fig. 2. Photograph of the plastic waste and the zeolite catalyst

2.2. Experimental work

Fig. 3 shows the experimental setup in the present work. The setup consists of a pyrolyzer, a condenser, a water tank, an LPG burner, K-type thermocouples and a data logger "GRAPHTEC GL240". The pyrolyzer, condenser, and water tank are hand-made from Stainless Steel plate with the thickness of 3 mm. The LPG burner and K-type thermocouples are bought from local market in Yogyakarta. The experimental work is performed in batch mode. For 180 minutes batch operation, 1000 g of the LDPE plastic and 500 g of the zeolite catalyst are fed into the pyrolyzer. The pyrolyzer is heated up using an LPG burner. Temperature at upper part of the pyrolyzer (T_1), temperature of the vapor at the condenser inlet (T_2), and temperature of the cooling water (T_3) are measured using the thermocouples and logged into the data logger. The plastic oil is collected every 15 minutes and weighted. Data of the temperatures and the plastic oil yield for the use of 1, 2, and 3 mm zeolite are analyzed and discussed. Higher heating value (HHV) of the oil is analyzed using Bomb Calorimeter. In addition, performance of the hand-made condenser is also discussed.

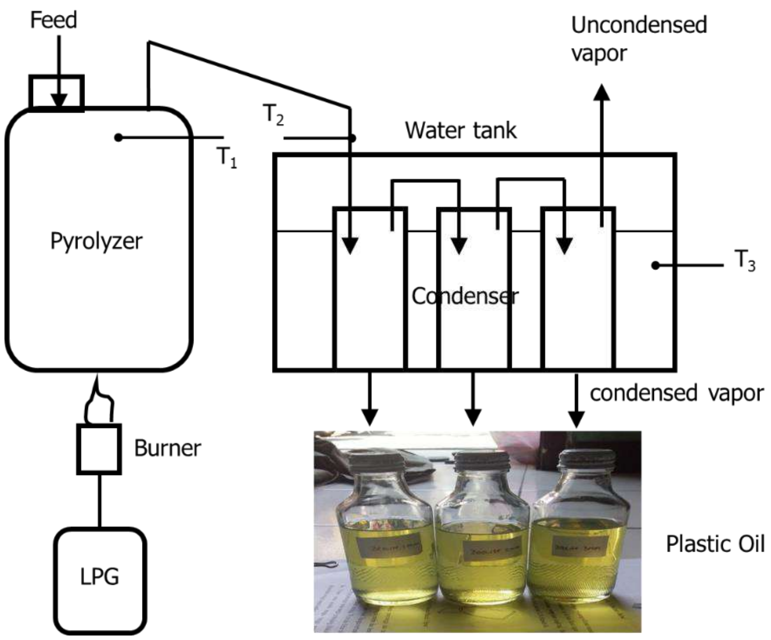

Fig. 3. Experimental setup and photograph of the plastic oil

3. Results and Discussion

Fig. 4 presents the temperature profile at upper part of the pyrolyzer (T_1) during 180 minutes batch operation time. The curves indicate that the temperature at upper part of the pyrolyzer increases faster with the use of the 1 mm zeolite. Heat transfer rate to the feedstock is higher when using the 1 mm zeolite. This is due to larger heat transfer area with the use of 1 mm zeolite than with the use of 2 and 3 mm zeolite. Increasing heat transfer rate to the feedstock causes the pyrolysis temperature increases faster. The temperature at upper part of the pyrolyzer reaches 120°C within 90 minutes for the use of the 1 mm zeolite. At the same minutes, the temperatures observed are 90°C and 50°C for the use of 2 mm and 3 mm zeolite, respectively. At 180th minute, the temperatures at that location are nearly similar for all zeolite sizes, such that 155°C. According to Aboulkas *et al.* [16], LPDE pyrolysis starts to produce oil at temperature of 300°C. The temperature does not change significantly by adding a catalyst [17]. In the present work, the heat for pyrolysis is obtained from LPG burner. Thus, it is difficult to maintain a proper pyrolysis temperature. However, the temperature at upper part of the pyrolyzer (T_1) can be used to predict the pyrolysis temperature at the bottom of the pyrolyzer. The pyrolysis temperature should be higher than 155°C. Conduction heat transfer occur in the pyrolyzer from bottom to the top (i.e. from higher temperature to lower temperature). Theoretically, the temperature at the bottom part of the pyrolyzer (i.e. pyrolysis temperature) is higher than 155°C and probably near to 300°C.

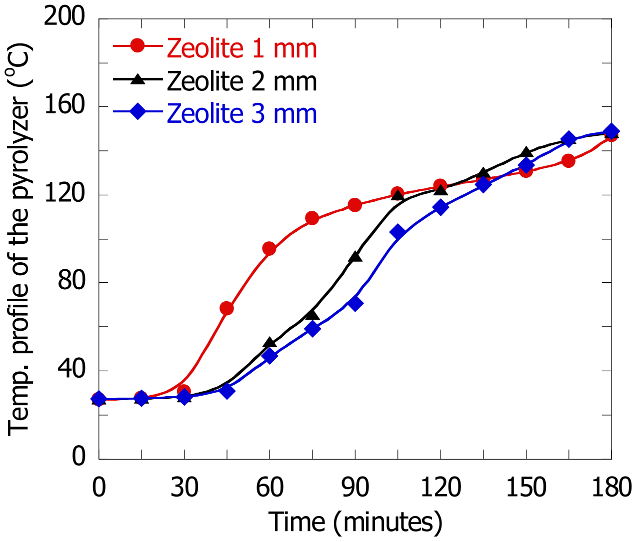

Fig. 4. Temperature profile at upper part of the pyrolyzer

In general, the trend of oil yield is similar to the pyrolysis temperatures trend for all zeolite size as shown in Fig. 5 to Fig. 7. Comparing all three curves in Fig. 5, it can be noticed that oil production starts after 80, 90, and 100 minutes for the use of 1, 2, and 3 mm zeolite, accordingly. The oil production begin when the temperature at upper part of the pyrolysis reaches 100°C. Since the pyrolysis temperature increases faster for the use of 1 mm zeolite, shorter time is required to start the production of the plastic oil. The increase in temperature affects the oil production positively. The temperature in the pyrolysis process indicates the necessary heat for the decomposition of the plastic bonds. The conversion efficiency increases with increasing temperature [13]. However, very high temperatures give negative effect on oil production. It is because at very high temperature there is a secondary cracking of the volatiles, which results in a higher gas yield. Pyrolysis temperature of LDPE is considered high when the process was conducted at temperature higher than 550°C. Marcilla *et al*. [18] conducted LDPE pyrolysis at temperature of 550°C. No other reported work on LPDE pyrolysis was performed at temperature higher than 550°C. Besides increasing heat transfer area, reducing size of the zeolite also improves contact area between the LDPE and the zeolite. Improving contact area leads in enhancing the pyrolysis reaction rate.

In the present work, the pyrolysis temperature at the bottom part of the pyrolyzer should be higher than 300°C According to Aboulkas *et al*. [16], pyrolysis temperature of LDPE at which oil production begin was 300°C. Pyrolysis temperature range at which conversion of the plastic begin can be indicated by their thermal degradation temperature which is depended on plastic type [2]. Various pyrolysis temperatures of specific plastic have been investigated. For high-density polyethylene (HDPE), the pyrolysis temperatures observed are 300°C-400° [19], 450-550 [9]. Meanwhile for low-density polyethylene (LDPE), Onwudili *et al*. [20] performed LDPE pyrolysis at temperature of 425°C, Bagri and William [21] conducted LDPE pyrolysis at temperature of of 500°C, and LDPE pyrolysis was conducted at temperature of 550°C by Marcilla *et al*. [18].

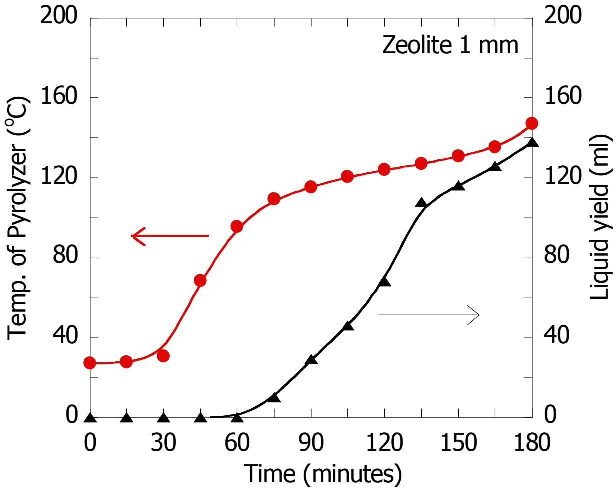

Fig. 5. An effect of pyrolysis temperature on oil production using 1 mm zeolite

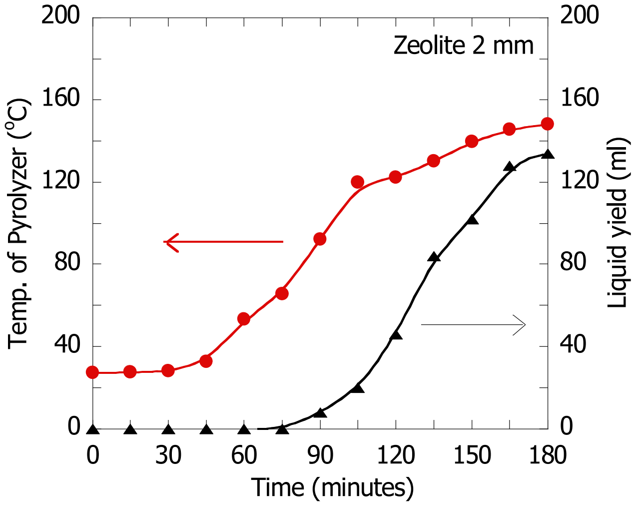

Fig. 6. An effect of pyrolysis temperature on oil production using 2 mm zeolite

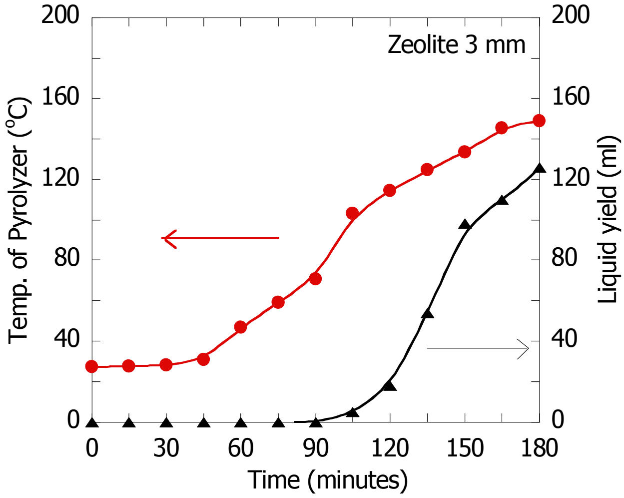

Fig. 7. An effect of pyrolysis temperature on oil production using 3 mm zeolite

Fig. 8 and Fig. 9 display the total oil yield and weight percentage of the product and residue after 180 minutes pyrolysis. From Fig. 8, the oil yields are 138, 134, 126 mL for the use of 1, 2, and 3 mm zeolite, respectively. Those volume of oils have a weight of 103.5, 100.5, and 94.5 g, accordingly. In weight percentage, the highest oil yield is 6.9 wt.% which is obtained for the use 1 mm zeolite as shown in Fig. 9. The highest oil yield obtained at 1 mm zeolite is due to the highest reaction rate with the use of 1 mm zeolite. The oil yield in the present work is much lower than that obtained by Marcilla *et al*. [18] who obtained oil yield of 93.1 wt.% when carried out LDPE pyrolysis in a batch reactor at 550°C with heating rate of 5 °C/min. This may due to uncontrolled heating rate and much lower of pyrolysis temperature in the present work. To obtain more precise measurement of oil yield, the digital measurement device should be used. This may reduce uncertainty in device reading during the experimental work.

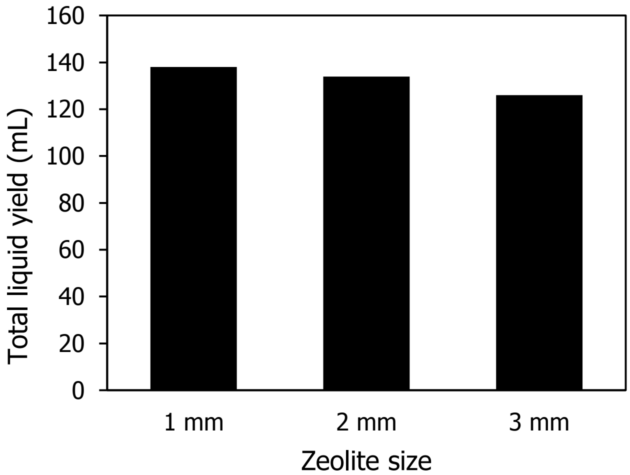

Fig. 8. Total liquid yield

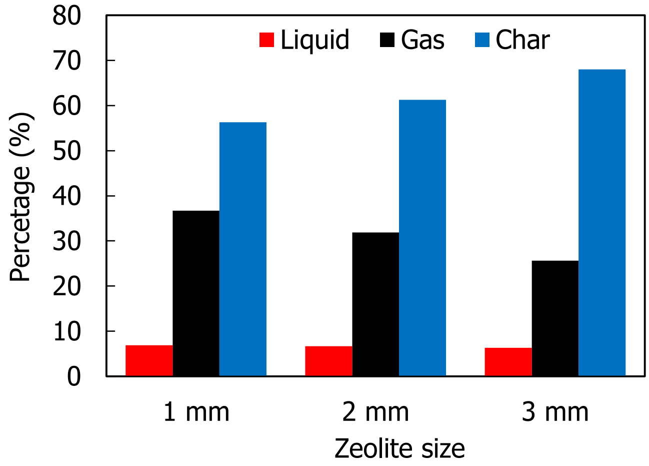

Fig. 9. Percentage of liquid, gas, and char

Meanwhile, Fig. 10 presents higher heating value (HHV) of the plastic oil. The use of 1 mm zeolite produces plastic oil with the highest HHV. The HHV of the oil with the use of 1, 2, and 3 mm zeolite are 45.47, 45.08, and 45.08 MJ/kg. These HHVs are nearly similar to the HHV of gasoline and diesel fuel. In terms of density, oil from LDPE pyrolysis seems comparable with the commercial standard value of both gasoline and diesel [2]. Desai and Galage [22] found that LDPE oil has a density of 0.78 which is similar to that of gasoline [19]. From measurement of volume and mass of the oil yield, the density of the oil is calculated to be 0.75 in the present work. This value is nearly similar to that of gasoline. Since the HHV and density of the oil are almost similar to that of gasoline, the oil obtained from LDPE pyrolysis in the present work could be used as a fuel of spark ignition engine.

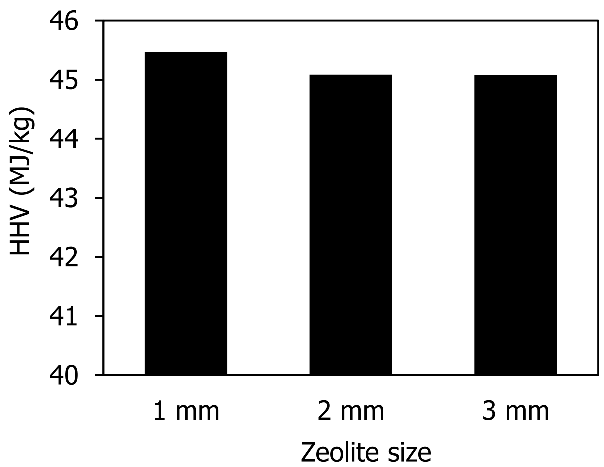

Fig. 10. HHV of the liquid product

In addition, overall performance of the condenser is analyzed based on Fig. 9 and Fig. 11. Weight percentages of the oil are lower than that of the gas and the char for all zeolite sizes. Much lower the oil percentage is due to low condensation rate of the vapor in the condenser. Heat transfer rate from the vapor to the cooling water is low. This phenomenon is indicated by the temperature difference between vapor temperature at the condenser inlet and the temperature of cooling water which is relatively small as shown by Fig.11. In order to increase oil yield, performance of the condenser could be improved by lowering the temperature of the cooling water to 15°C [23] or increasing heat transfer surface area of the condenser. Improving heat transfer rate will enhance condensation rate, resulting more vapor is converted into oil.

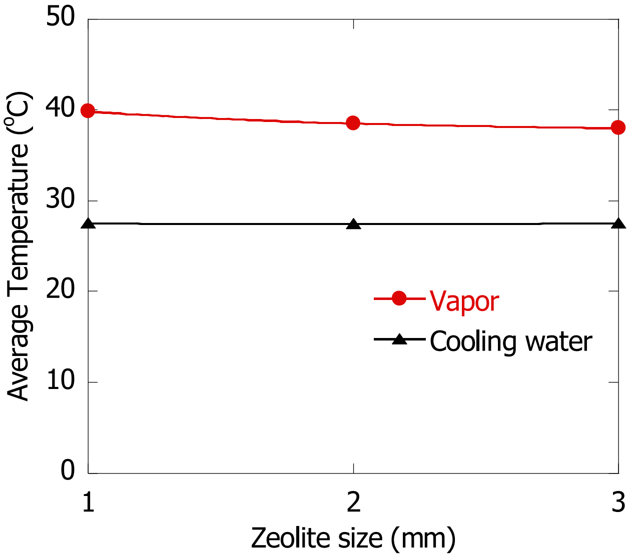

Fig. 11. Average temperature of vapor and cooling water of the condenser

4. Conclusion

Experimental work of catalytic pyrolysis of the LPDE plastic waste is been conducted in batch pyrolyzer using different zeolite size (i.e. 1, 2, and 3 mm in diameter) at low-temperature. It can be concluded that zeolite size impacts the pyrolysis process and oil yield. Smaller zeolite size increases heat transfer rate, pyrolysis temperature, reaction rate, and oil yield. The pyrolysis temperature plays major role in oil yield. Oil yield increases as increasing in pyrolysis temperature. However, oil yield percentage is relatively low compared to gas yield and remaining char. Condensation rate of the vapor in the condenser is very low. The performance of the condenser have to be improved by lowering the temperature of the cooling water or increasing heat transfer surface area of the condenser.

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