

Catalytic Co-pyrolysis of Empty Fruit Bunch and High-density Polyethylene

Nadhilah Aqilah Shahdan^a, Vekes Balasundram^{a,*}, Kamyar Shameli^a, Norazana Ibrahim^b, Ruzinah Isha^c, Pramila Tamunaidu^d, Zainuddin Abdul Manan^e

^a Chemical Energy Conversions and Applications (ChECA), Malaysia-Japan International Institute of Technology, Universiti Teknologi Malaysia, 54100 Kuala Lumpur, Malaysia.

^b Energy Research Group, School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia.

^c College of Engineering, Universiti Malaysia Pahang, 26600 Pekan, Pahang, Malaysia.

^d Malaysia-Japan Advanced Research Centre (MJARC), Universiti Teknologi Malaysia (Pagoh), Off Jalan Edu Hub Gunasama 1, Pagoh Higher Education Hub, 84600 Pagoh, Johor, Malaysia.

^e Process Systems Engineering Centre (PROSPECTS), School of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia.
 vekes@utm.my

The main objective of the present work is to investigate the thermal degradation behaviour of the non-catalytic and catalytic co-pyrolysis of empty fruit bunch (EFB) and high-density polyethylene (HDPE) over commercial hydrogen exchanged zeolite socony mobil five (HZSM-5) and rice husk ash (RHA) catalysts via thermogravimetric analyser (TGA). RHA catalysts were produced using the solvent-free method by converting RHA into HZSM-5. XRD characterization was conducted for the synthesized catalysts and RHA catalyst showed less amount of peaks compared to commercial HZSM-5. Non-catalytic and catalytic co-pyrolysis of EFB and HDPE over commercial HZSM-5 and RHA catalysts were conducted using TGA. A fixed EFB-to-HDPE mass ratio of 1:1 and a fixed catalyst-to-feedstock mass ratio of 1:1 were used for the TGA experiments. The sample was heated up under pyrolysis conditions at a heating rate of 20 °C/min until 700 °C. The thermal degradation behaviour of EFB and HDPE did not change significantly when RHA catalysts were used, based on the TG curves. Volatilization of matter was maximum between temperatures 240 °C and 500 °C (Phase II) for all cases of the pyrolysis process, where the highest volatilized matter of 93.2 wt% was produced from the catalytic process over commercial HZSM-5, followed by the catalytic process over RHA catalysts with 92.3 wt% of volatilized matter and non-catalytic process with 83.0 wt% of volatilized matter. When using catalysts, 0.44 wt% of solid residual was left when commercial HZSM-5 was used while 0.38 wt% of solid residual was left when RHA catalyst was used.

1. Introduction

Fossil fuels have been a primary energy driver, especially in the form of fuel for transportation (US Energy Information Administration, 2021). Due to the increase of usage, the fossil fuel industry has been facing several challenges. One of the challenges is resource depletion. A statistical report conducted by the British Petroleum Company (2020) stated that since 2018, total proved oil reserves have been decreasing and the currently available fossil fuel resources can only sustain the population for the next 49.9 y if extraction of resources continues at the current rate, based on the reserves-to-production ratio that was calculated. Fossil fuels have also been a large contributor to the negative impacts on the environment. It has contributed to the increase of greenhouse gas emitted to the atmosphere that has led to a rise of 1 °C in the global average temperature for 2019, the highest rise by far (Ritchie and Roser, 2017). These challenges have driven the interest in finding alternatives to fossil fuels, especially for biomass-derived fuel via pyrolysis.

A thermochemical conversion process called pyrolysis breaks down material in the absence of oxygen at high temperatures, usually in the range of 300 to 700 °C (Bhoi et al., 2020). According to Kan et al. (2020), materials like biomass, especially agricultural residues can be utilized in pyrolysis because they can contain

useful compounds, in the form of bio-oil. These chemical compounds are similar to that of fossil fuels, especially aromatic compounds like benzene, toluene, ethylbenzene, and xylene (BTEX) (Sanahuja-Parejo et al., 2019) but they are often obtained in low yield and quality. Hassan et al. (2020) studied the pyrolysis of sugarcane bagasse and obtained a bio-oil yield of only 41.0 wt% while Wu et al. (2020) reported a bio-oil yield of only 19.1 wt% obtained from the pyrolysis of corn stover. The low quality of bio-oil from pyrolysis of biomass is attributed to the high oxygen content from compounds such as acids and esters, which causes corrosiveness and contributes to low heating value (Hassan et al., 2020) compared to the standard high heating value of commercial fossil fuels, which is around 45.2 MJ/kg for gasoline and 45.2 MJ/kg for diesel (Hossain et al., 2019). To overcome this problem, biomass is co-pyrolysed with plastic as plastic is rich in hydrogen that helps with the decomposition of biomass cellulose while the oxygen content in the biomass helps with polymer cracking (Ryu et al., 2020), due to the positive synergy that exists (Uzoejinwa et al., 2018). Sanahuja-Parejo et al. (2019) conducted a study on the pyrolysis of grape seeds and discovered when polystyrene was added to the feedstock, bio-oil yield increased from 39.0 wt% to 62.0 wt%. Plastic waste generation has been increasing to the point that it is polluting the environment, especially the waterways and the ocean, and being able to utilize it in pyrolysis not only helps with waste management, but it also helps to retrieve valuable compounds in the plastic that are otherwise discarded. Maisarah et al. (2018) reviewed the appropriateness of using different types of waste for energy and stated that plastic waste is encouraged to be used in pyrolysis compared to incineration as it can be polluting to the environment when incinerated.

The pyrolysis process can be further improved by adding catalysts, which reduces the amount of oxygenated compounds, resulting in improved bio-oil quality (Razzaq et al., 2019). Zeolite catalysts have aluminosilicate structures that provide acid sites for the formation of desired compounds via various chemical reactions, such as deoxygenation, depolymerization, and aromatization (Rahman et al., 2018). Hydrogen-exchanged zeolite Y (HY) (Lee et al., 2015), hydrogen-exchanged zeolite beta (HBeta) (Lee et al., 2015), and hydrogen-exchanged zeolite socony mobil 5 (HZSM-5) (Iftikhar et al., 2019) have been previously used as catalysts for pyrolysis. Among the different catalysts that have been tested in pyrolysis, HZSM-5 is reported to be the optimal catalyst attributed to its unique structural qualities and strong acidity (Kan et al., 2020), favouring the production of aromatic compounds. HZSM-5 is usually synthesized via hydrothermal method (Zhang et al., 2019), requiring dangerous chemical precursors such as tetraethylorthosilicate (TEOS) (Adam et al., 2012), which can be toxic when exposed to and finding a greener approach should be encouraged.

Using natural resources for catalyst synthesis can be an alternative. Rice husk ash (RHA) contains high silica content of around 93.4 wt% (Korotkova et al., 2016), making it an appropriate silica precursor for catalyst synthesis. There have been several studies that focus on catalyst synthesis using RHA. Among the different zeolites that RHA has been converted to, like zeolite beta (Prasetyoko et al., 2006) and zeolite NaY (Mohamed et al., 2012), RHA has also been converted to zeolite ZSM-5 via solvent-free method with a silica and alumina recovery rate of 98.0 % (Zhang et al., 2019). This shows how RHA is a strong candidate for synthesizing catalysts. Despite zeolite ZSM-5 being successfully converted from RHA, studies lack in the application of the catalyst, especially in pyrolysis process. The main purpose of this study is to investigate the thermal degradation behaviour of the non-catalytic and catalytic co-pyrolysis of EFB and HDPE over commercial HZSM-5 catalysts and RHA catalysts via thermogravimetric analyser (TGA).

2. Materials and method

2.1 Feedstock preparation

In this study, the biomass resource and plastic waste resource used consisted of empty fruit bunch (EFB) and high-density polyethylene (HDPE). EFB was obtained in pellet form from a local company in Malaysia. The EFB pellets were dried in a microwave oven at 105 ± 2 °C to remove moisture from the pellets. Using a lab blender (Model 7011HS, Osaka Chemical, Japan), the dried pellets were ground and sieved using a vibratory sieve shaker (Analysette 3 Pro, Fritsch International, Germany) to obtain a uniform particle size of 0.50 mm. To ensure that the moisture was not reabsorbed by the samples, the prepared EFB samples were stored in an airtight container. HDPE was obtained from a local supplier in Malaysia in pellet form. The HDPE pellets were ground using the same lab blender and sieved using the same vibratory sieve shaker to obtain a particle size of 0.50 mm.

2.2 Catalyst preparation and characterization

Two different types of catalysts were prepared for this study, HZSM-5, and rice husk ash (RHA) catalysts. To prepare HZSM-5, commercial ZSM-5 with a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 30, in the ammonium form, was obtained from Alfa Aesar. The solid powder was calcined in a laboratory muffle furnace at a temperature of 700 °C for four hours with a heating rate of 5 °C/min to obtain the protonic form of the zeolite, HZSM-5.

For the RHA catalysts, rice husk samples were first obtained from a local market in Malaysia and dried in a microwave oven at 110 °C to remove any moisture. Using the same lab blender and vibratory sieve shaker, the dried samples were ground and sieved to achieve a particle size of 0.50 mm. In a laboratory muffle furnace, calcination was performed on the rice husks at 900 °C for 1 h, converting the sample into RHA. RHA was then converted to zeolite ZSM-5 via solvent-free method applied from the study conducted by Zhang et al. (2019). To achieve this, RHA was mixed with sodium aluminate (NaAlO_2), tetrapropylammonium bromide (TPABr), and sodium carbonate decahydrate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$) in a beaker in a molar composition of 30 $\text{SiO}_2/\text{Al}_2\text{O}_3$: 3.75 TPABr: 9 $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ using a magnetic stirrer for five minutes. The beaker containing the solid mixture was sealed and heated at 150 °C for 72 h. Before the reaction time ended, the solid mixture was washed with water at room temperature and filtered. In an air-circulating oven, the solid catalyst sample was then dried at 80 °C. The dried solid sample is calcined at a temperature of 700 °C for 4 h, with a heating rate of 5 °C/min in a laboratory muffle furnace.

The phase analysis of the synthesized catalysts was conducted via X-ray diffraction (XRD) (Rigaku SmartLab X-ray diffractometer, Japan). Irradiation was conducted on the catalyst sample using $\text{CuK}\alpha 1$ radiation with a wavelength (λ) of 0.15405 nm. This was performed using a tube voltage of 40 kV and tube current of 30 mA. Over a 2θ range of 3° to 60°, the sample scanning was then conducted using a scanning rate of 5 °C/min.

2.3 Experimental setup

The non-catalytic and catalytic co-pyrolysis of EFB and HDPE were conducted in a thermogravimetric analyser (TGA/SDTA851, METTLER TOLEDO, USA). Three different experiment runs were studied, the non-catalytic process, the catalytic process over HZSM-5 catalysts, and the catalytic process over RHA catalysts. All experiment runs were performed at a fixed HDPE-to-EFB mass ratio of 1:1, and a fixed catalyst-to-feedstock ratio of 1:1. Samples of around 5 mg were prepared using mortar and pestle and then heated from room temperature until 700 °C at a fixed heating rate of 20 °C/min. An inert atmosphere for the pyrolysis process was created by flowing nitrogen at a flow rate of 150 mL/min. The result from the TGA was used to study the thermal degradation behaviour of the biomass and plastic waste feedstock over non-catalytic and catalytic conditions using HZSM-5 and RHA catalysts.

3. Results and discussion

3.1 Phase analysis

The characterization of the HZSM-5 and RHA catalysts via XRD can be seen in Figure 1. For HZSM-5 catalyst, very intense peaks are observed at the 2θ position of 7.94°, 8.86°, 23.07°, 23.33° and 23.96°. Du et al. (2020) also observed this occurrence when characterizing HZSM-5 using XRD, where peaks were typically seen at the 2θ position of 8° and 23°. For RHA catalyst, not many peaks are observed and the peak that resembles HZSM-5 is only seen at the 2θ position of 21.9°. This was consistent with the XRD pattern of the RHA catalyst that was reported by Zhang et al. (2019).

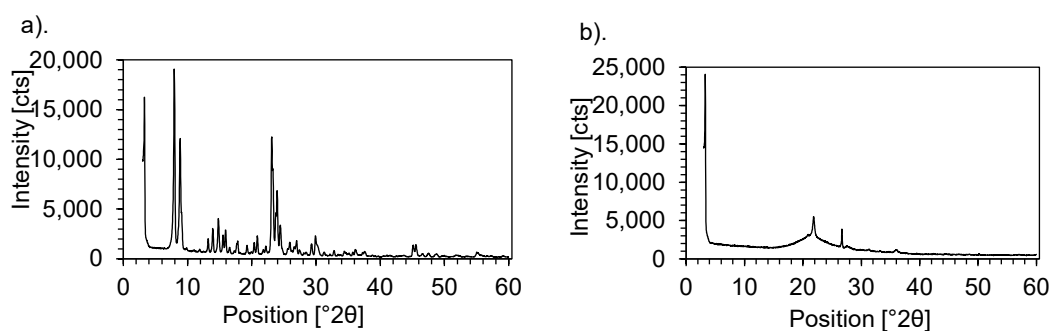


Figure 1: XRD pattern for (a) HZSM-5 catalyst and (b) RHA catalyst

3.2 Co-pyrolysis of EFB and HDPE

The effect of catalyst presence on the co-pyrolysis of EFB and HDPE can be seen from the mass loss with respect to temperature in the TG curves in Figure 2. In Figure 2, the TG curves have been adjusted to display the thermal degradation of feedstock by removing the catalyst mass prior to calculation. The TG curves for all the pyrolysis processes were divided into three different phases adapted from Balasundram et al. (2017). Phase I occurred from room temperature to 240 °C, where moisture and light components were first

vaporized. Phase II occurred between 240 °C and 500 °C, where hemicellulose and cellulose were then devolatilized, including HDPE where the thermal degradation of HDPE occurs around 400 °C (Aboulkas et al., 2010) with lignin decomposition occurring at the end of Phase II. Finally, Phase III occurred at temperatures from 500 °C to 700 °C, where decomposition starts to plateau. This agrees with the literature as cellulose and hemicellulose degrade at temperatures lower than lignin (Rocha et al., 2020). The division of these three phases can be visualized in Figure 2. When catalysts were added, for both cases of HZSM-5 and RHA catalysts, the thermal degradation curve of EFB and HDPE was lower compared to the non-catalytic process (refer Figure 2). This indicates that EFB and HDPE degraded faster with the addition of the catalysts. The degradation of EFB and HDPE without catalyst was completed at 500 °C while with catalysts, the degradation was complete at around 475 °C, when either HZSM-5 or RHA catalysts were used.

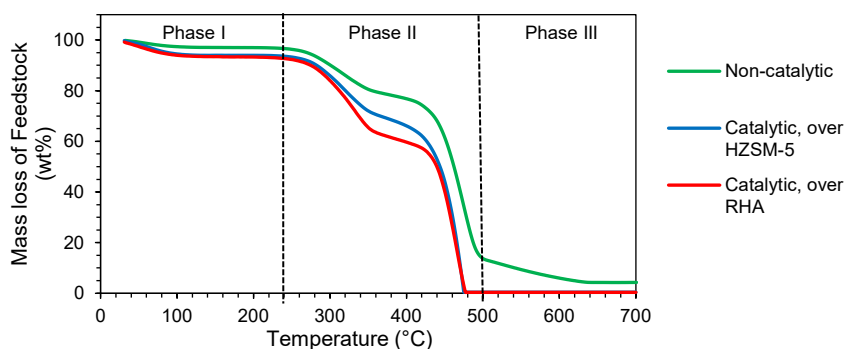


Figure 2: TG curves of non-catalytic and catalytic co-pyrolysis of EFB and HDPE

In Figure 3, the derivative TGA (DTG) curves show the mass loss events that occur in the different phases. Similar to the phases in the TG curves, the first, second, and third peaks in Figure 3 can be associated with the vaporization of moisture, the decomposition of hemicellulose and cellulose, and the decomposition of polymer and lignin. The highest points of the peaks in the DTG curves represent the maximum rate of mass loss (Mensczel and Prime, 2009). The difference in the intensity of the peaks is seen significantly at the third peaks for all the cases of non-catalytic and catalytic pyrolysis processes. The highest rate of mass loss in the third peak is observed for the catalytic process over HZSM-5 catalysts while the least rate of mass loss in the third peak is seen for the non-catalytic process. The catalytic process over RHA catalyst also showed a higher rate of mass loss in the third peak compared to the non-catalytic process.

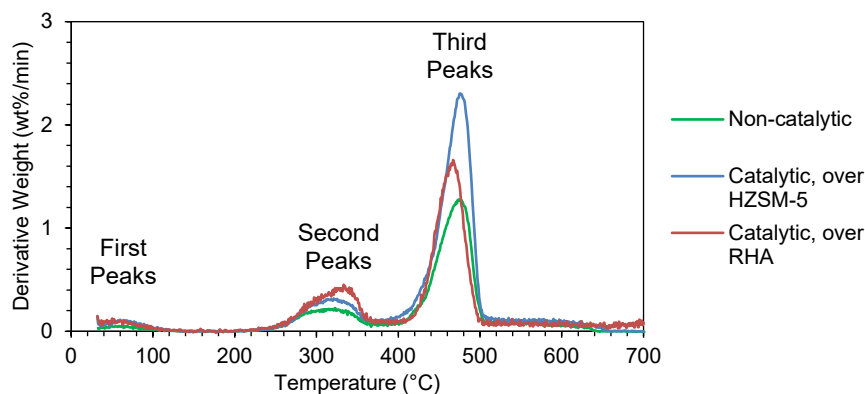


Figure 3: DTG curves of non-catalytic and catalytic co-pyrolysis of EFB and HDPE

For all the cases of non-catalytic and catalytic processes, mass loss was significant in Phase II. As seen in Figure 4, higher percentage of mass loss occurred in Phase II for the catalytic process over HZSM-5 catalyst and the catalytic process over RHA catalysts compared to the non-catalytic process. This is due to the majority of the components of the feedstock where hemicellulose, cellulose, lignin and HDPE (long-chain hydrocarbons) all degrade within the temperature range of Phase II. It is unknown from the TGA data whether the volatilized matter in Phase II was in the form of bio-oil or syngas. Furthermore, the major difference between the TG curves can be seen in the weight percentage that remained in Phase III. Based on Figure 4, only 4.3 wt% of matter remained when the temperature reached 700 °C. For both the cases of the catalytic

pyrolysis over HZSM-5 and RHA catalysts, very little matter remained, where 0.44 wt% of solid residual remained when HZSM-5 catalyst was used while 0.38 wt% of solid residual remained when RHA catalyst was used. This shows that the catalysts have improved the degradation of EFB and HDPE.

Comparing between HZSM-5 and RHA catalysts, although the rate of mass loss is higher in Phase II when RHA catalyst was used as seen in Figure 2 and 3, the percentage of mass loss itself was similar to that when HZSM-5 was used. This can be seen in Figure 4 where 93.2 wt% of mass loss occurred when HZSM-5 was used while 92.3 wt% of mass loss occurred when RHA catalysts were used. Further analysis should be done to observe how RHA affects the chemical composition of the volatilized matter as compared to HZSM-5. Based on the results obtained, RHA has the potential to be a catalyst source for pyrolysis as it improves the thermal degradation behaviour of the catalytic co-pyrolysis of EFB and HDPE.

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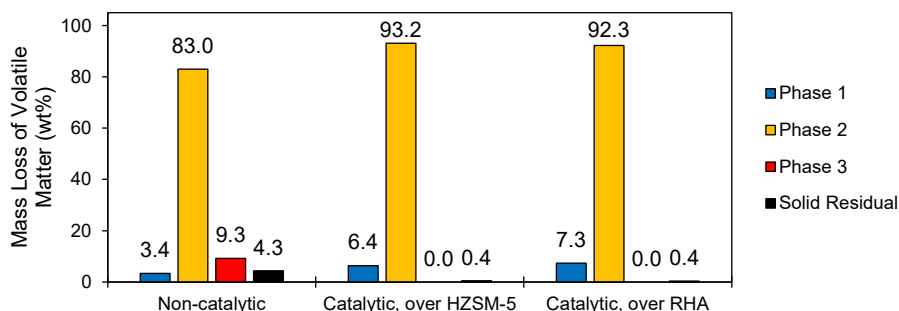


Figure 4: TG volatile matter of mass loss of EFB and HDPE feedstock sample

4. Conclusion

RHA catalyst has been successfully applied to the catalytic co-pyrolysis of EFB and HDPE via thermogravimetric analyser (TGA). Results from using TGA in this study have shown that using HZSM-5 or RHA catalysts improves the thermal degradation behaviour of EFB and HDPE although the two catalysts have different structures based on XRD. Volatilized matter was maximum in Phase II of the pyrolysis process for all non-catalytic and catalytic processes. After accounting for the catalyst mass in the calculations, it was found that very little solid residual remained for both cases of catalytic process using HZSM-5 and RHA catalysts. Further analysis using pyrolyser-GC/MS should be conducted to identify the chemical composition of the volatilized matter to see how using RHA catalysts affect the chemical composition compared to using HZSM-5. As most of the matter was volatilized in Phase II, it is possible that it was volatilized either in the form of bio-oil or syngas and further research should be conducted to determine how much bio-oil or syngas that was produced from the pyrolysis process. Despite the increase of solid residual observed, this study has shown that catalytic pyrolysis can be conducted using RHA catalysts with the additional benefit of being a renewable resource that is low-cost compared to HZMS-5 catalyst. Further research can be done in enhancing the catalytic performance of the RHA catalyst.

Acknowledgments

A special thanks to Universiti Teknologi Malaysia (UTM) for the financial support to carry out this research project under the Research University Grant - UTM ER (Vot number: Q.K130000.3843.19J46). The authors also would like to thank the Frontliners of Malaysia, who have been working day and night during the COVID-19 pandemic.

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