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Parametric study on catalytic cracking of LDPE to liquid fuel over ZSM-5 zeolite

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ABSTRACT

Pyrolysis or cracking of plastic waste is considered as a potential solution to the environmental problems brought about by plastic waste, with the production of hydrocarbon fuel as a value added benefit. In order to explore the potentials of such process, parametric study have been conducted on the catalytic cracking of LDPE dissolved in benzene in a fixed bed reactor. The five factors studied were temperature (A), catalyst mass (B), feed flow rate (C), carrier gas flow rate (D), as well as concentration of LDPE solution (E), while the responses were LDPE conversion (Y_1) and liquid yield (Y_2). The parametric study showed that four out of five factors (A, B, C and D) have significant effects on Y_1 and Y_2. The optimum conditions that produced maximum responses for Y_1 and Y_2 simultaneously are 600 °C (A), 0.10 g catalyst (B), 1 ml/s LDPE solution (C), 80 ml/min N_2 flow (D). The numerical values for Y_1 and Y_2 were 98.6% and 99.5%, respectively. Analysis on products composition indicated that catalytic cracking of LDPE in fixed bed reaction generally produced high amount of aliphatic branched-chain compounds, together with moderate amount of cyclic compounds. Aromatization of LDPE cracking products is less due to the short retention time of the compounds on the catalysts bed.

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1. Introduction

As the advantages of plastic materials such as light weight, low cost and ease of processing (compared to other materials) are widely employed in all spheres of human endeavours, little attention was given to the detrimental effects of the indiscriminate disposal of post-consumer waste of such materials on the environment. Pyrolysis and catalytic cracking of plastic waste are some of the techniques proposed to curve such negative consequences. These techniques can convert plastic waste to useful products such as fuels and chemical feedstock [1–3]. Due to its significance, research in this area is rewarded by government of most countries in the form of incentives [4,5] since it can convert the source of environmental pollution into renewable energy. However there are still challenges to overcome before such processes can be integrated into large scale plastic recycling industry, as proposed in the literature [6–8]. In order to overcome these challenges, continuous cracking of plastic dissolved in solvent is attempted, inspired by the studies on selective dissolution/reprecipitation of plastics [9] and catalytic cracking of dissolved plastics in fixed bed reactors [10]. However, up to date researchers have very little understanding on the performance of continuous catalytic cracking of plastics, therefore, studies in this area is highly desired.

In order to enable production of fuels from plastic waste, the effects of reaction parameters on the process performance has to be well understood. Systematic study of the process can be achieved by Design of experiment (DOE) due to its versatility. DOE have been widely used in studies on pollution control and resource recovery [11,12], as well as cracking of polymers. Most of the studies on polymer cracking utilized central composite design (CCD) [13–15], while several studies made use of Box-Behnken designs [16,17] and Taguchi method [18]. However, these designs often produce a large number of experimental runs for processes that involve many factors, hence increase the difficulty on the process analysis. The use of two level factorial design [19] overcomes such drawback, as such design requires less experimental runs to obtain satisfactory amount of data for analysis.

It has been shown that catalytic cracking of LDPE solution was able to produce liquid fuel despite short retention times [20,21]. However, to the best of our knowledge, no parametric study has been carried out on catalytic cracking of dissolved polymer. The objective of this study is to determine the effects of five reaction parameters (temperature, catalyst mass, feed flow rate, N_2 flow rate, and concentration of LDPE solution) on the LDPE conversion

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Nomenclature

LDPE	low density polyethylene
HDPE	high density polyethylene
Factor A	temperature
Factor B	catalyst mass
Factor C	feed flow rate
Factor D	carrier gas (N_2) flow rate
Factor E	concentration of LDPE solution
Y_1	LPDE conversion
Y_2	liquid yield
DOE	design of experiment
FTIR	Fourier transform infrared spectroscopy
GC/MSD	gas chromatography coupled with mass selectivity detector
ANOVA	analysis of variance
H_o	null hypothesis

and liquid yield during catalytic cracking of LDPE in a fixed bed reactor. The use of full two level factorial design to determine the optimum conditions involving two responses is also demonstrated in this study.

2. Materials and methods

2.1. Materials

Commercial low density polyethylene (LDPE) was purchased from Titans Chemicals, Johor, Malaysia, while the benzene was purchased from Qrec. The detailed method for dissolution of LDPE in benzene can be found in literature [22,23]. The ZSM-5 zeolite used in this study as catalyst was purchased from Acros Organics, Belgium. Catalytic properties of the zeolite was studied in previous work [24]. The zeolite was pressed and sieved to obtain particles in size of 1.0–1.4 mm for cracking purpose. Prior to the experiment, the catalyst was heated for 4 h at 500 °C to remove the impurities on the catalyst surface. After that, the catalyst was mixed with silicon carbide with particles size of 1.0–1.4 mm, which act as catalyst diluent.

2.2. Reactor setup

The catalytic cracking of LDPE solution was performed in a self-assembled fixed-bed reactor, which was made of ½ in. 316 SS tubing with length of 27 cm. The catalyst bed was located 15 cm from top, supported by stainless steel mesh. During the reaction, the temperature of the catalyst bed was monitored by a K-type thermocouple located above the catalyst bed, which was connected to a temperature controller. The column was heated by a cylindrical refractory heater. Due to the carcinogenic nature of benzene solution, the reactor was situated in a covered metal frame equipped with exhaust fan for safety purpose. Fig. 1 shows the reactor setup in the laboratory.

2.3. Catalytic cracking of LDPE dissolved in benzene

Prior to the reaction, the reactor was purged with nitrogen for 5 min to ensure an inert atmosphere for cracking reaction. Then, the reactor was heated to desired temperature, and the flow rate of nitrogen gas was adjusted to desired flow rate. When the reactor reached the required temperature, a single syringe pump (Cole Parmer 74,900 series) was switched on to allow the flow of LDPE solution into the mixing point with carrier nitrogen gas. The LDPE solution was allowed to pass through the fixed catalyst bed in a down-flow direction. The products formed during the cracking flowed down the reactor into a glass condenser unit (cooled to 0.5 °C using a circulating chiller (FIRSTEK, MODEL-B401L)), and then into a gas liquid separator. All the liquid products were collected and weighed. After weighing, the sample bottles were kept at 5 °C in a refrigerator to prevent the loss of highly volatile hydrocarbon molecules during storage.

The yield of liquid product collected in the separating funnel in each run was calculated using Eq. (1). In order to ensure repeatability of the result, each run was repeated twice, and the average value of the liquid yield from the two runs was taken to represent the actual value. Calculation showed that the percentage errors for the readings on liquid yield are less than 3% in all cases.

$$Liquid\ yield = \frac{mass\ of\ liquid\ collected\ after\ cracking}{mass\ of\ LDPE\ solution\ fed\ into\ the\ reactor} \times 100\% \tag{1}$$

2.4. Product analysis

The unconverted LDPE in the liquid products was quantified through FTIR analysis. Each liquid product was scanned by IR-Prestige-21 Fourier Transform Infrared Spectrophotometer (Shimadzu) to obtain the absorbance at $\sim$2918 cm^{-1}. By using a premade calibration curve, the absorbance was converted to concentration of unconverted LDPE in the solution. The liquid product was analysed by gas liquid chromatography with mass selectivity detector (GC/MSD) using Agilent 6890N Network GC system through method described in a work by Ates et al. [25] on plastic pyrolysis. The GC/MSD was equipped with a 30 m × 0.25 mm capillary column coated with a 0.25 μm thick film of 5% phenyl-methylpolysiloxane (HP-5). Helium was employed as a carrier gas at constant flow rate of 1.2 ml/min. The initial oven temperature was 45 °C held for 2 min, ranging from 45 to 290 °C at 5 °C/min and then held for 10 min. Splitless injection was applied at 290 °C. Prior to injection, the liquid samples were filtered using syringe filters with pore size of 0.22 μm. Chromatographic peaks were identified by means of NIST standard reference database.

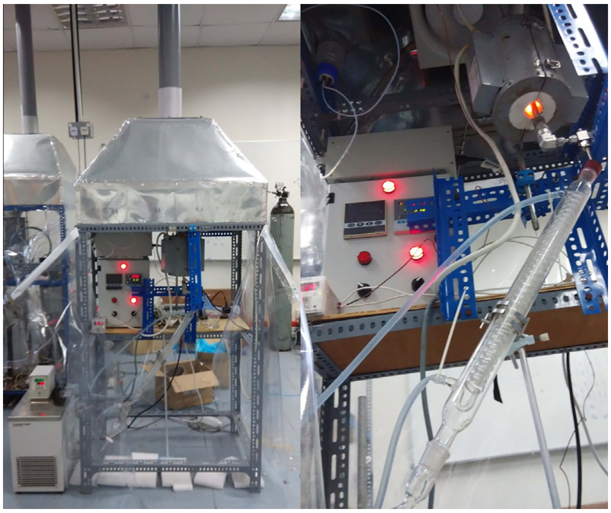

Fig. 1. Setup of fixed bed reactor for LDPE cracking.

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