Undergraduate Student Training on Thermal Pyrolysis of Low Density Polyethylene for Sustainable Fuel Productions

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Abstract

The use of petroleum based fuels and products in the world has been continuously rising. Plastics are one the most widely used petroleum-based products in both consumer and industrial production markets. Some methods have been developed and used to manage plastic waste around the world. The more traditional and common method is recycling, which changes the form or shape of the plastic into another form without making too much damage to the environment. However, there are some drawbacks on the recycling methods because it requires a high labor cost for the separation process and water contamination that can reduce the sustainability of the overall productions. Compared to the other methods, thermal pyrolysis is not fully developed. It basically changes the molecular structure of the plastic into useful gas and liquid fuels. It mainly removes the plastic waste from the environment and recovers the energy to reduce the fuel dependence. The recovery of plastic into liquid oil through the thermal pyrolysis process has significant potential since the oil produced has a high-energy value comparable to the commercial fuels used today. In this study, recycled and pelletized Low Density Polyethylene (LDPE) pellets were placed into the oxygen free reaction chamber, heated up to the decomposition temperature for a certain time period. Decomposed LDPE produced gas and liquid fuels, second of which reheated to produce diesel like fuel for many applications. Produced gas can be used as a heat source for the future pyrolysis processes. The produced fuel was characterized using different techniques. The purpose of this study is to train undergraduate students for pyrolysis process to convert plastic bags into fuel, and provide them the new developments in the field.

Keywords: Low Density Polyethylene (LDPE), Plastic Recovery, Pyrolysis, Sustainable Fuel.

1.0 Introduction
1.1 General Background

Plastic has played a vital role in enhancing the standard of living for more than 50 years. It is the key to the innovation of many products in various sectors, such as construction, healthcare, paint, electronics, automotive, packaging, and many others. The demand for plastics has been increasing due to the rapid growth of world population and industrialization. The global production of plastics reached about 299 million tons in 2013 and has continued to increase, contributing to the growth in waste accumulation every year (Mallonee et al.,2016; Chinni et al.,2016; Hughes et al.,2016). Based on the 2013 statistics, it was reported that 33 million tons of plastic waste were generated in the U.S. alone. Plastics waste usually falls into two categories: pre-use (production scrap) and post-use plastic/product. Source of pre-use plastic waste is either coming from plastic that has not met the required specifications (e.g., wrong color, hardness, strengths and characteristics), or off-cuts occur during assembly or installation. Post-use plastic waste is appropriate for recycling and falls into five categories; 1) plastic bottles, tubs and trays; 2) plastic films; 3) rigid plastics including pipes and molding / extruding; 4) plastic foams, such as expanded polystyrene (EPS); and 5) flexible plastics, like strapping and cable sheathing (Recoup,2017).

In Europe, 25 million tons of plastic ended up in waste stream during the year of 2012. Based on statistics in Europe, about 38% of the plastic waste went to the landfill, 26% were recycled, while 36% were utilized for energy recovery/conversion. The percentage of plastic waste that ended up in the landfill is still very high and occupies a significant amount of valuable space. Plastics can take up to several hundred years to degrade naturally. They degrade so slowly because the molecular bonds contain hydrogen, carbon, nitrogen, chlorine, oxygen, and others that make the plastic very durable materials for a longer time (Wikipedia, 2017).

Although traditional plastic recycling is able to reduce some amount of the plastic waste, the idea of a more reliable and sustainable method has been established. The demand of plastics continues to grow each year, and the reduction of fossil fuels such as coal, gas, and especially petroleum that make up plastic itself has gained much interest. There has been a push for researchers to discover and develop a possible energy supply from plastics due to the rising energy demand. Energy conversion from plastic waste is being explored as a way to utilize and meet the increasing demand.

1.2 Pyrolysis Process

Pyrolysis is the process of thermally degrading long chain polymeric molecules into smaller ones, less complex molecules through heat and pressure. The process requires intense heat in short durations while in the absence of oxygen in reaction chamber. The three major products that are produced during pyrolysis include oil, gas, and char (or some ashes) which are valuable for many industries, especially manufacturing and refineries (Jangbarwala, 2016). There are two types of pyrolysis: catalytic and thermal. For the present studies, only thermal pyrolysis is used to gather data.

The process can produce high amounts of liquid oil, up to 80 (wt %) at moderate temperatures around 400-500 °C. The pyrolysis process is a very adaptable process, in which the parameters can be manipulated to optimize the products based on predefined constraints and conditions. The liquid oil produced can be used in multiple applications, such as furnaces, boilers, turbines, burners, steamers, and diesel engines without much needs of upgrading or treatment processes (Sharuddin et al., 2016). Pyrolysis is not a source of water contamination and is considered as green technology by some scientists, while others might argue that the potential for air pollution can be considerably high. The gaseous by-product has a substantial energy value that can be reused to assist the overall energy requirement of the process. The pyrolysis process handling is much simpler than the common recycling method since it doesn't need an intensive sorting process. The pyrolysis process can optimally convert 78–84% of plastic by weight into combustible liquid oil / fuel.

Basically, different plastics have different compositions and chemical structures. Proximate analysis is a technique used to measure the chemical properties of the plastic compounds based on four components: moisture content, fixed carbon, volatile matter, and ash content. Volatile matter and ash content are the largest influence for the liquid oil yield during the pyrolysis process. A high amount of volatile matter is preferred for the liquid oil production. Alternatively, a high ash content will decrease the amount of liquid oil produced, while increasing the gas yield and char coal formations based on the product (Sharuddin et al., 2016).

Referring to Table 1: Proximate analysis of different plastic materials the volatile matter present in all the plastics is high while the ash content is relatively low (Sharuddin et al., 2016). Low-density polyethylene (LDPE) has the highe st volatile (~99.7 wt%) content with very little to no ash content (~0.4 wt%) unless some inorganic additives are added during the manufacturing. This configuration indicates that plastics have a high potential for large amounts of liquid oil production through the pyrolysis process. The results from the plastics proximate analysis indicate that the process parameters of the pyrolysis process will strongly influence the liquid oil production.

Table 1: Proximate analysis of different plastic materials (Sharuddin et al., 2016).

Type of plastics	Plastics type marks	Moisture (wt%)	Fixed carbon (wt%)	Volatile (wt%)	Ash (wt%)
Polyethylene terephthalate (PET)	1 PET	0.46	7.77	91.75	0.02
		0.61	13.17	86.83	0.00
High-density polyethylene	2 HDPE	0.00	0.01	99.81	0.18
		0.00	0.03	98.57	1.40
Polyvinyl chloride (PVC)	3 V	0.80	6.30	93.70	0.00
		0.74	5.19	94.82	0.00
Low-density polyethylene	4 LDPE	0.30	0.00	99.70	0.00
		–	–	99.60	0.40
Polypropylene	5 PP	0.15	1.22	95.08	3.55
		0.18	0.16	97.85	1.99
Polystyrene	6 PS	0.25	0.12	99.63	0.00
		0.30	0.20	99.50	0.00
Polyethylene (PE)	7 OTHER	0.10	0.04	98.87	0.99
Acrylonitrile butadiene styrene (ABS)		0.00	1.12	97.88	1.01
Polyamide (PA) or Nylons		0.00	0.69	99.78	0.00
Polybutylene terephthalate (PBT)		0.16	2.88	97.12	0.00

As byproducts, pyrolysis of LDPE plastics also produces char and gas (Syngas). The proportions of the by-products in pyrolysis are influenced by several parameters, such as temperature, heating rate, pressure, catalysts, and reaction time (Rowhani and Rainey, 2016). High temperature and long residence time are the best conditions to maximize gas production in a thermal pyrolysis process. These are opposite of the parameters used to maximize the oil production. Generally, gas production in the process depends on the temperature and type of plastics used in the pyrolysis process. At 425 °C, the pyrolysis of LDPE produces combustible gases for different applications.

The gas composition depends on the composition of feedstock materials. The main gas components produced during the pyrolysis of LDPE plastic are hydrogen, methane, ethane, ethene, propane, propene, butane, and butene (Sharuddin et al., 2016). The gas produced from the pyrolysis process has significantly high calorific value. The gas produced from the pyrolysis of LDPE can have a calorific value between 42 and 50 MJ/kg. Making the gas a potential fuel source in many industrial plants. The gas can also be used in turbines to generate electricity and boilers without the need for flue gas treatment (Fernandez et al., 2011).

A slow heating rate at very low temperatures, and a long reaction time maximize the char formation in the pyrolysis process. The product formation in the early pyrolysis process is mainly low (Sharuddin et al., 2016). The proximate analysis found that volatile matter and fixed carbon were the main components of the char (>97 wt.%) while moisture and ash content are the minor. The components closely related to the proximate analysis of the raw plastic (Table 1: Proximate analysis of different plastic materials show that most plastics are composed of almost 99 wt% volatile matter (Sharuddin et al., 2016).

Char can have a calorific value of approximately 18.84 MJ/ kg. The low sulfur content makes it a suitable fuel in combustion with coal or other organic wastes. However, high inorganic matters in the char can make them difficult to use as a fuel. It can still be used as road surfacing, a building material, and an adsorbent in water treatment to remove heavy metal through an upgrading treatment. The adsorption properties of upgraded chars are mostly a mesoporous and macro-porous material, with adsorption capacity for methylene blue dye is in the range of 3.59 – 22.2 mg/g (Sharuddin et al., 2016). This indicates that the activated chars have good adsorption properties with bulky molecules. Other potential applications for pyrolysis char include feedstock in production of activated carbon and as solid fuel for boilers and heat exchanges.

2. Process Parameters

The process parameters are vital for optimizing the product yield and composition in any of the pyrolysis processes. For LDPE plastic pyrolysis, the process parameters influence the production of the final products, such as liquid oil, gas, and char. These key parameters are summarized as temperature, reactor/high temperature reaction vessel, and time below:

2.1 Types of Polymers

LDPE is a thermoplastic made from the monomer ethylene (Malpass,2010). LDPE is defined by a density ranging from 0.910 to 0.940 g/cm^3, with covalent bonds and van der Waal forces, low toxicity, and a molecular mass of 20,000 – 500,000. It is chemically inert and non-reactive at room temperatures, and can withstand temperatures up to 80 °C continuously, and 95 °C for shorter period of time. It is most commonly made in translucent or opaque variations that are flexible and tough. As is seen in Figure 1, LDPE has more branching (about 2% of the carbon atoms) than High Density Polyethylene (HDPE), so the intermolecular forces (instantaneous-dipole induced-dipole attraction) are weaker and the modulus of resilience is higher. The increase in branching causes weaker intermolecular forces, and lowers the tensile strength and hardness. It has better ductility since the side branching causes the structure to be less crystalline and easily molded. With an excellent resistance to water, it is widely used as plastic bags, wrapping foils for packaging, trash bags, and many others.

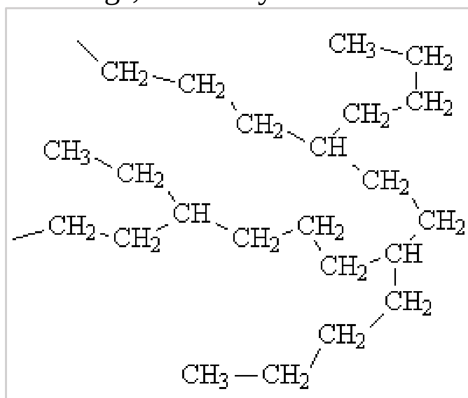

Figure 1: Chemical structure of low-density polyethylene plastic.

2.2 Reactor

The type of reactor has an impact in the mixing of the plastics, residence time, heat transfer, and efficiency towards achieving the final product. Most plastic pyrolysis are performed in batch, semi-batch or continuous-flow reactors to produce gas, liquid and solid products.

2.3 Temperature

Temperature is one of the most significant operating parameters in the pyrolysis process, and controls the cracking reaction of the polymeric chain (Sharuddin et al., 2016). The molecules are attracted by van der Waals forces and this prevents them from collapsing. When the temperature of the system increases, the vibration of the molecules increases, causing the molecules to evaporate away from the surface of the LDPE (vapor forms). This occurs when the energy by the internal forces along the polymer chains becomes greater than the enthalpy of the C–C bonds in the chain, resulting in a broken carbon chain into different by-products.

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2.4 Reaction Time

Residence time can be defined as the average amount of time that the plastic spends in the reactor, and its influence on product distribution (Sharuddin et al., 2016). A longer residence time will increase the conversion of the product, thus creating a more thermally stable product, such as light molecular weight hydrocarbons and non-condensable gases. However, the temperature must reach a certain point in the process before the residence time has much effect on the product distribution.

3. Experiment

3.1 Materials

LDPE plastic bags of different size and shape were collected locally, and utilized in the pyrolysis process to produce oil, gas and solid (mainly char/ashes). No further treatments were applied on the collected plastics prior to the process.

3.2 Methods

Design materials and supplies were obtained from local home improvement store to build pyrolysis prototype. For this experiment, a batch style reactor that was under atmospheric pressure was constructed using a 2.6 gallon propane cylinder (**Error! Reference source not found.**). Outside of the reactor was contained fire bricks (**Error! Reference source not found.**) with a high-pressure propane burner (**Error! Reference source not found.**) placed directly underneath the tank. During the chemical reactions at high temperature, gas and liquid products were immersed into a cold water container through a copper pipe to condense the liquid products.

a) b) c)

Figure 2: The images showing a) outside of assembled pyrolysis reactor, b) inside of the reactor with a used propane tank, and c) high temperature burner.

a) b) c)

Figure 3: Images showing a) the LDPE plastic bag used in the pyrolysis process, b) vapor and initial oil, and c) produced liquid oil.

The high pressure burner is rated at 150,000 BTUs/hr with a regulator operating at 5 psi. The bricks can withstand temperatures above 600 °C without any damages. This experiment was conducted with about 1,100 grams of the LDPE in the form of plastic shopping bags (Figure 3: Images showing a) the LDPE plastic). The plastic was packed into the reactor and heated to approximately 415°C for 45 minutes. It was observed that a dense vapor and a small amount of liquid oil formation started at temperatures of 360 – 385 °C (**Error! Reference source not found.**), and the oil conversion of LDPE started at about 400 °C (**Error! Reference source not found.**). A brown waxy material also formed in the oil at temperatures below 415 °C, indicating that the conversion to oil was incomplete. During the reactions, a large amount of gas was produced. Note that this by-product gas can be utilized to heat the reaction chamber as a heating energy sources. The byproduct burns like natural gas.

4. Results and Discussion
4.1 Quantitative Recovery of Process

After the first pyrolysis process was completed, it was discovered that paraffin and glycerin type materials were largely present in the collected liquid oil. This outcome was also stated in previous studies (Joo and Guin,1997). This waxy like substance has a low melting temperature (~ 100 °C), so it could easily be converted into a liquid in a warm bath or under a flame. It could, however, go back to a solid / waxy state if the temperature of the product fell below 70 °C. The mixture of paraffin, glycerin and oil was reheated second time in the same reactor at about 380 °C to produce more lighter and sable fuel. This step resulted in a cleaner more purified oil with a natural liquid state. Figure 4 shows the obtained oil after the first and second runs of pyrolysis process.

a) b)

Figure 4: The obtained wax / oil after a) the first and b) second run of pyrolysis process. The first step is more likely to be waxy, while the second sept is clear oil/fuel (more diesel like).

In addition to the paraffin and oil products, the pyrolysis process also converted some of the LDPE into char/ash and gas. Table 2 exhibits quantitative results of the first and second runs of pyrolysis process. As an input, overall 1,100 g of LDPE plastic bags was used for the first run of the pyrolysis process. After the first run, amounts of obtained paraffin/oil, char and gas are 643g, 294g and 175g, respectively. Percent yielding of paraffin/oil, char and gas was 58%, 27% and 15%, correspondingly. In the second run of the pyrolysis process, paraffin and oil which were obtained from first run pyrolysis process were used. Out of 643g paraffin and oil mixture; 312 g oil, 170g char and 161g gas was obtained in the second run of the pyrolysis process. Final percent yielding of the materials are about 49% oil, 27% char and 25 % gas. There might be some leakage during the reactions, but at minimum level.

Table 2: Quantitative results of the pyrolysis process after first and second run of the tests.

First run of the pyrolysis process	**LDPE (**1,100 grams**)**		
	Type of outcomes	Amount (g)	Yield (%)
	Paraffin & Oil	643 g	58.4%
	Char	294 g	26.5%
	*Gas	175 g	15.1%

Second run of the pyrolysis process	**Paraffin & Oil** (643 grams)		
	Type of outcomes	Amount (g)	Yield (%)
	Oil	312 g	48.5%
	Char	170.3 g	26.5%
	*Gas	160.75 g	25.0%

(* approximate value)

4.2 NMR Characterization

Nuclear Magnetic Resonance (NMR) tests were conducted on the liquid samples to characterize their properties. The liquid sample collected from the second process was considered during these tests. This study was performed on the samples to help determine about what the oil products were or their compositions. Figures 5 and 6 show the NMR test results of the standard gasoline and diesel samples (Thermofisher, 2017; Process NMR Associates LLC., 2017). These results will be used to compare the oil produced in the present study. The NMR result of the pure gasoline sample indicates that aromatics, alcohols, methylated aromatics and aliphatics are presented in the sample (Figure 5). The same NMR tests conducted on the pure diesel shows that di-aromatic-H, mono-aromatic-H, alpha-CH_2, alpha-CH_3 and CH_3 groups are formed in diesel.

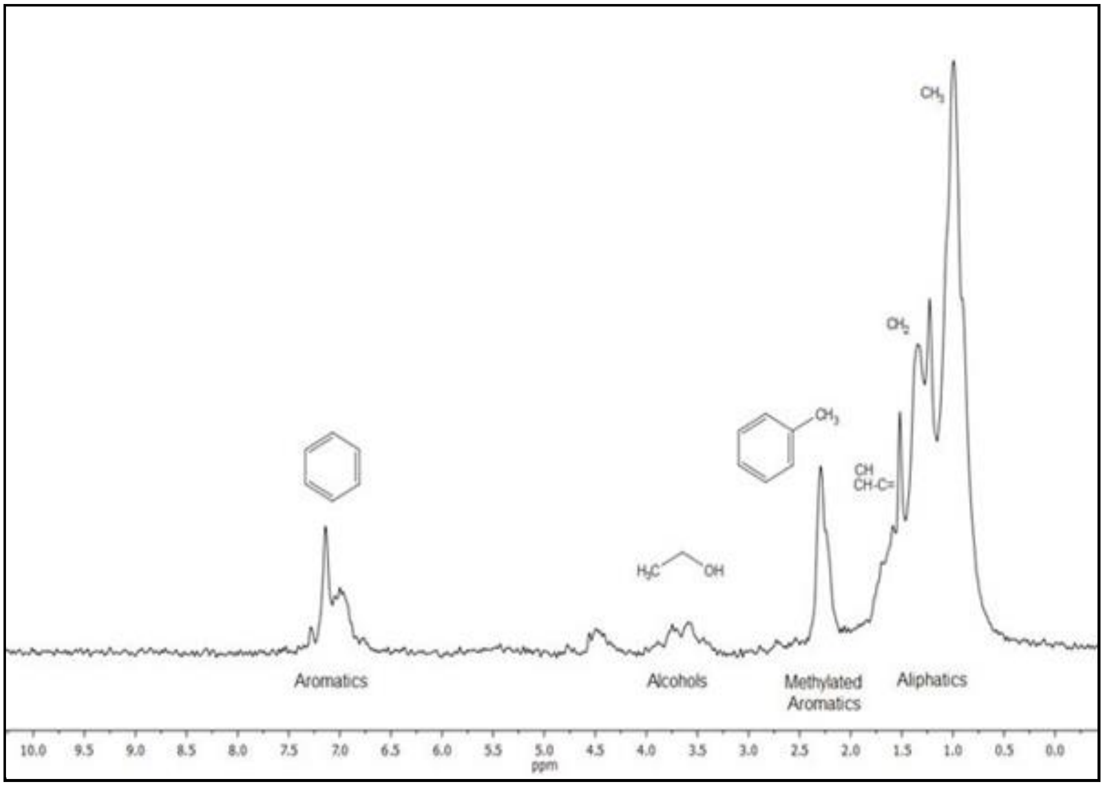

Figure 5: NMR test results of the standard gasoline samples (Thermofisher, 2017)

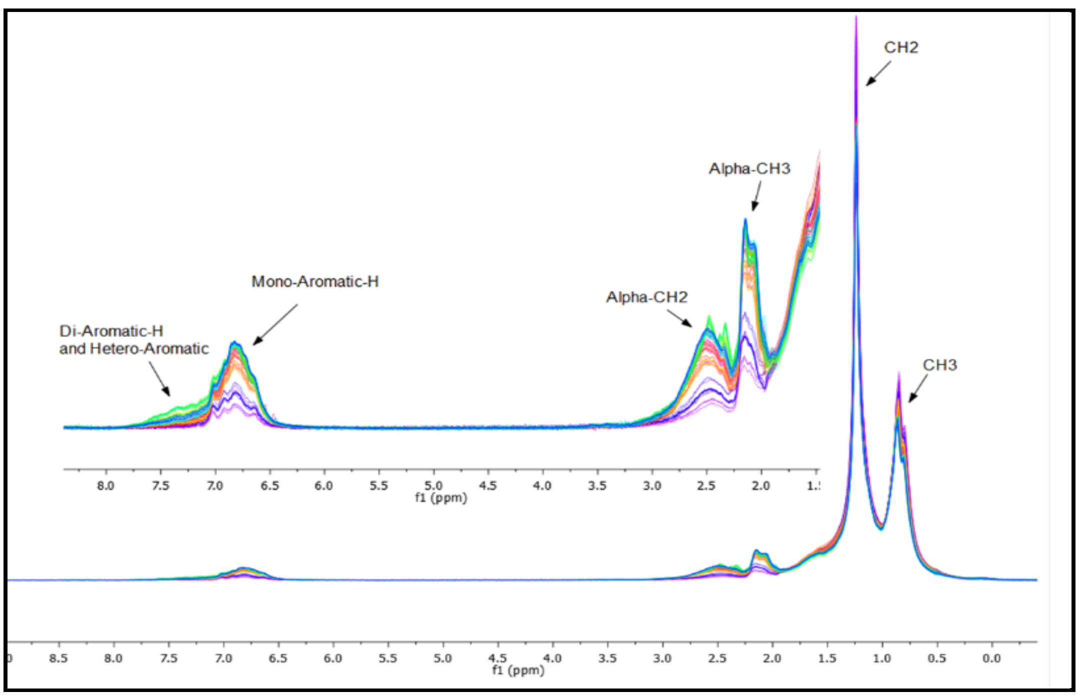

Figure 6: NMR test results of the standard diesel samples (Process NMR Associates LLC., 2017)

In order to compare the test results with the pure diesel and gasoline, a number of tests were conducted on the prepared liquid samples. Figure 7 shows the NMR results of the present oil produced in the second step of the reactions using the LDPE pyrolysis process. The tests results confirmed that the LDPE pyrolysis process oil after the second distillation process contains aromatics, solvents, olefins, alpha-H, CH3, aliphatic, and C=CH groups. These tests indicated that the oils produced using the LDPE pyrolysis process were similar to the previous liquids (mainly diesel and less likely gasoline; perhaps 80:20 ratio). It can be concluded that the LDPE pyrolysis process oil may be considered as a fuel for different engines after some optimization studies. Table 3 gives the properties of the LDPE oil vs. conventional diesel properties which was extracted from Sharuddin et al., 2016.

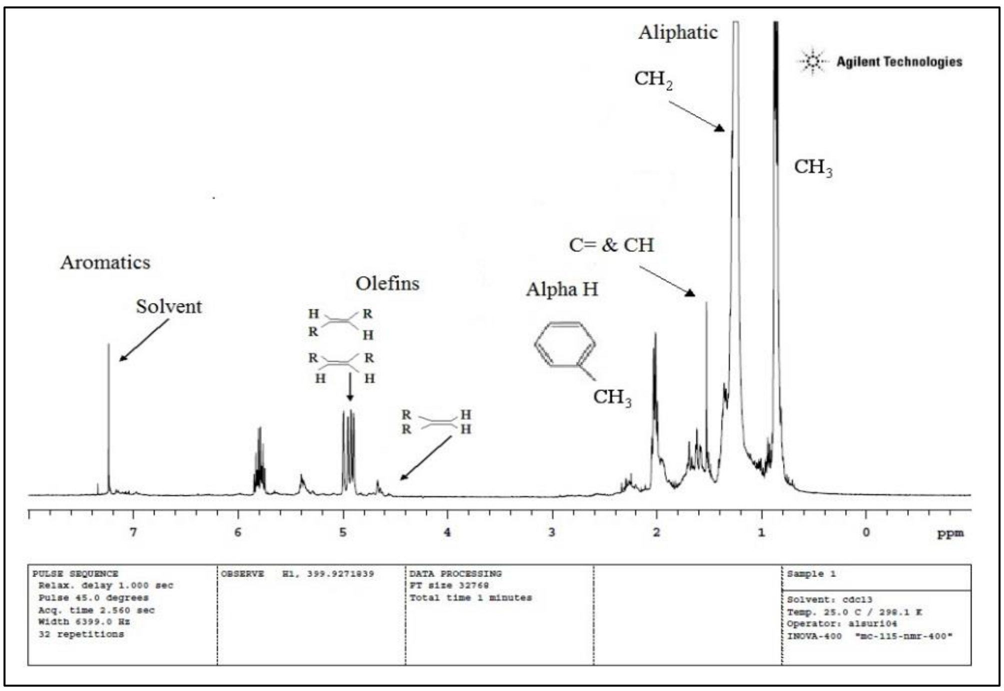

Figure 7: The NMR results of the present oil produced in the second step of the reactions using the LDPE pyrolysis process.

Table 3: The properties of the LDPE oil vs. conventional diesel properties. (Sharuddin et al., 2016)

Parameters	Units	Liquid oil vs. conventional diesel	Value
Density	g/cm^3	LDPE oil Conventional diesel	0.7787 0.815-0.870
Viscosity	mm^2/s	LDPE oil Conventional diesel	1.89 2.0-5.0
Kinetic viscosity	cSt	LDPE oil Conventional diesel	- 2.0-5.0
HHV	MJ/kg	LDPE oil Conventional diesel	38-39 46.67
Pour point	°C	LDPE oil Conventional diesel	- 6
Flash point	°C	LDPE oil Conventional diesel	41 52
Boiling point range	°C	LDPE oil Conventional diesel	- 150-390

4.3 Process Modifications and Optimizations

Many process parameters can be used to improve the outcome of the experimental results. First, the reactor temperatures can be adjusted to optimize gas and liquid productions from the recycled plastics. Research shows that the maximum liquid oil yield occurs at temperatures between 469 and 494 °C for some plastics (Alarifi, et al.,2016). The optimum temperature to obtain the highest liquid oil for LDPE is 420-425 °C, so it would be possible to reach various temperatures to maximize the by-product yields from different plastics (Sharuddin et al., 2016).

According to Sharuddin et al., 2016; introducing pressure is another modification that could speed the process up and increase its output. Pressure can affect the carbon number distribution of the liquid oil by shifting it to the lower molecular weight side as it rises, and the rate of double bond formations (Murata et al.,2004). The rate of double bond formations decreased when the pressure increased, suggesting that pressure directly affect the scission rate of C–C links in the LDPE polymer. It was also discovered that pressure had greater impact on residence time at lower temperatures. As the temperatures increased more than 430 °C, the effect of the pressure on the residence time became considerably less (Sharuddin et al., 2016).

Agitations of the reactor during the decomposition process at higher temperatures can improve the product output, along with selecting materials that have both a high heat tolerance and conductivity. Agitation of the catalyst materials (e.g., zeolite, clay, and other silicates) in the reactor will improve the production rates (Manos et al.,2001). Further research is needed for developing a process that pretreats the plastic for different conditions. This would also maximize the amount of space used, while removing unwanted voids and oxygen from the process.

Collecting the gas that is produced during the process is another way to increase the overall efficiency of the pyrolysis system. An increase in process temperature increases the production of the gases. Generally, 1 kg of plastic feedstock produces within the range of 13 – 26.9% gases by weight (Sharuddin et al., 2016). The produced gases from plastic waste have high calorific values, and it can be put back into the reaction system as a heat source or be stored and used in other applications. It could also make the process more environmentally friendly and safe.

4.4 Hands-on Experiences for Engineering BS Students

Because of the environmental and health concerns of many fossil fuels, new generations of energy sources have been considered more worldwide. Recycled plastic-based fuels (e.g., diesel, gasoline and jet fuel) can be the major sources of the renewable energies for transportation industries. Department of Mechanical Engineering at WSU has more than 600 undergraduate students and a big portion of them are considering hands-on engineering experiences during their studies. Two of the students (Mr. Nathan M. Schneider, and Mr. Mark Janzen) were involved in the present study, learned many new techniques and gained a lot of new skills and knowledge about recycling, renewable energy sources, pyrolysis, and other related characterization techniques. These students have used these research activities as their own Engineer of 2020 requirements in the College of Engineering. Both of them are also co-authors of the present study and made significant contributions during the experiments. It is believed that these hands-on trainings and new technologies will enhance the knowledge of many BS engineering students to perform more detail studies in their future.

5. Conclusions

Thermal pyrolysis has the potential to convert energy from LDPE plastic waste into valuable liquid oil, gas, and char/ash. It could be one of the best alternatives for plastic waste conversion and recycling. The amount of global plastic waste is reaching hundreds of millions of tons yearly. This process can be adapted for larger industrial scales with profitable outcomes (e.g., diesel, gasoline, char, Syngas, etc.). It can also be scaled down to a more household sized process. The LDPE thermal pyrolysis method makes waste management more efficient, with less capacities in landfills, while lowering pollution and positively affecting the economy. The dependence on fossil fuel as the non-renewable energy could be reduced through the present studies. Two of the engineering students designed, developed and performed the tests to produce liquid, gas and solid products from the recycled shopping bags, and gained enormous research experiences. These practices will benefit undergraduate students for their future academic studies in different universities.

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Biographical Information

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Nathan Schneider is the Mechanical Engineer student at Wichita State University (WSU). After the graduation, he will be focusing on renewable and sustainable energy sources, such as solar and wind energies and recycling and reusing of engineering materials.

Mark Janzen

He graduated from Cowley County Community College in Spring 2012 with an Associate of Arts Degree. He is a BS student in Mechanical Engineering at WSU. He is expecting to join the graduate school for his further studies in renewable energy and other related technologies.

Dr. Eylem Asmatulu

Dr. Asmatulu is currently an Engineering Educator in the Department of Mechanical Engineering at WSU and actively involving in teaching, research, and scholarship activities in the same department. She received her PhD degree from the Department of Industrial and Manufacturing Engineering at WSU in May 2013, which was mainly focused on the "Life Cycle Analysis of the Advanced Materials". Prior to the WSU, she also worked in the Environmental Health and Safety at WSU and Composite Manufacturing Laboratory at NIAR of WSU. Throughout her studies, she has published 7 journal papers and 23 conference proceedings, authored 7 book chapters, presented 8 presentations, and reviewed several manuscripts in international journals and conference proceedings. Dr. Asmatulu is currently conducting research on "e-waste recycling, algae based biofuel productions and CO_2 capturing.