Microwave Pyrolysis of Polypropylene with Iron Susceptor

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ABSTRACT

The improper disposal of plastic waste and low recycling rate have caused various environmental issues around the world. Therefore, microwave metal pyrolysis approach is proposed to efficiently convert plastic waste into liquid fuel, wax and gaseous by-products. This study aims to investigate the effect of different parameters such as microwave power and mass of metal on the product formation of the pyrolysis of polypropylene (PP). The experimental study was conducted in a closed glass reactor with a capacity of 500 ml, in a modified 2.45 GHz microwave, at a pressure of 1 atm and nitrogen is flowed at 0.5 L/min. The plastic was mixed with iron (Fe) powder and pyrolysed for 30 min. The produced pyrolysis vapor was condensed in a two-stage condenser where the oil formed was subsequently collected in a flask. The increase in microwave power from 500 to 700 W increased the oil yield of PP with iron powder from 22.4 to 54.5 wt.% and decreased the wax yield from 40.2% to zero. The increase in mass of iron powder from 5 to 10 g improved the oil yield from 20.0 to 54.5 wt.%, while the oil yield slightly decreased to 50.1 wt.% at 15 g. The pyrolysis oil formed has high calorific value of 45-46 MJ/kg comparable with the commercial fuel, thus the fuel can be blended with pure diesel to reduce the portion of fossil fuel in diesel combustion engine application.

Keywords: Microwave pyrolysis, Metal susceptor, Polypropylene, Plastic waste

1 Introduction

Plastic is used in various applications such as packaging, textile and construction due to its versatility, durability, lightness, corrosion resistance and low production cost [1]. According to Geyer, et al. [2], approximately 400 Mt (in million metric tonnes) of plastic is manufactured every year. The global production is projected to increase to 590 Mt by 2050 [3]. Unfortunately, most of the plastic waste generated is disposed in the landfill or dumped in the rivers and sideways, causing various environmental problems. More than 8 Mt of plastic is tossed into the oceans annually, killing 100 million marine lives each year. According to WWF report, in 2019, the lifetime cost of plastic including its pollution and clean-up expenses is US\$3.7 trillion, signifying its huge impact on our economy and environment [4].

The rate of plastic waste recycling worldwide is only 15% due to the difficulties to recycle mixed plastics and unclean materials with food residue [5]. The plastic separation process is expensive as it requires a large amount of workers or complex machinery to mechanically sort the waste [6]. Pyrolysis is an alternative method as it can decompose mixed plastic waste into valuable compounds such as fuel at high temperature [7]. However, due to the inefficient heating normally associated with the conventional pyrolysis, microwave heat source is utilized to provide rapid heating and shorter reaction time [8]. Unlike conventional heating, microwave energy penetrates the inner part of the material and heats it evenly through the molecular interaction with the electromagnetic field. Microwave pyrolysis is considered an efficient technique to pyrolyse plastic to value added products. It may yield up to 81 wt.% pyrolysis oil and 18 wt.% gases that can be utilised as transport fuels [9].



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When microwave radiation hits a surface, it can either be absorbed, reflected or transmitted. A material's ability to absorb microwave energy is directly related to its conductive properties. Plastic is a poor conductor and has a low dielectric constant, thus, it is transparent to microwave and not significantly heated in a microwave [10]. Thus, microwave pyrolysis of plastic requires additional absorbers such as carbon, silicon carbide or metal powders to improve energy absorption and heat transfer. A material can interact with a microwave's electric field, or magnetic field, or both [11]. The dipolar polarisation heating mechanism with the electric field component may occur when a polar molecule such as water is exposed to microwave radiation. The molecules attempt to rotate and realign with the electric field and collide with one another. The resistance to rotation results in friction which is translated to heat energy and heats up the material [8]. Meanwhile, a metal will interact with the magnetic field, generating eddy current and inducing alternating magnetic field which produce heat.

Microwave pyrolysis can provide higher heating rate and shorter reaction time compared to conventional pyrolysis while producing a high amount of product yield. Since plastics are poorly heated in a microwave, in this research, PP plastic will be mixed with metal powder (iron) to efficiently convert plastic waste into liquid fuel and wax. In this paper, the effect of microwave power and amount of iron powder on the product yield and distribution of microwave-metal assisted pyrolysis of plastic is investigated.

2 Materials and Methods

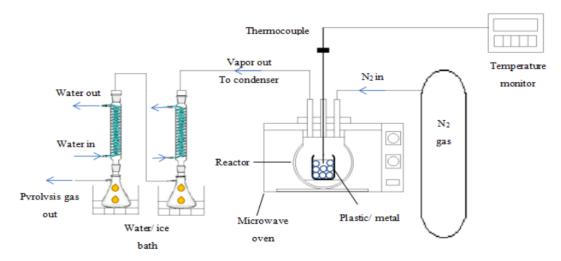


Figure 1: Microwave pyrolysis set-up

A 2.45 GHz domestic microwave oven manufactured by Samsung as shown in Figure 1 was modified to allow the entry and exit of gas from the top of the microwave. Nitrogen gas was flown at 0.5 L/min to the reactor for 2 min before the experiment to maintain an inert atmosphere. The wide neck flask reactor vessel was custom-built and made of borosilicate glass, with an internal diameter of 15 cm, a height of 12 cm and a wall thickness of 2 cm and had a capacity of 0.5 L. The PP plastic sample 50.0 g was mixed with iron powder (Figure 2) and put inside an alumina crucible and placed in the reactor. The sample was heated inside the microwave for 30 min. The microwave power was varied at 500, 600 and 700 W. The mass of iron powder was varied at 5.0 g, 7.5 g, 10.0 g and 15.0 g which were equivalent to 10.0, 15.0, 20.0 and 30.0 wt.% respectively. The pyrolysis vapor was condensed to form pyrolysis oil which was collected in a flask while the non-condensable vapor remained as gas. The reaction temperature was monitored using a thermocouple type-K covered with silicone sheath to protect the metal thermocouple from arcing. The product yield of wax and liquid were weighed, and the mass of gas was determined from the difference between mass of plastic and mass of wax and liquid.

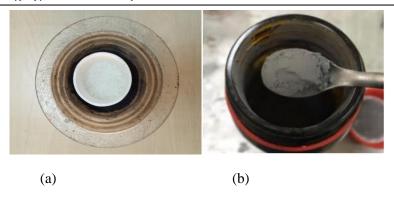


Figure 2: (a) Plastic sample and (b) iron powder

Pyrolysis oil product formed was analysed using Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography Mass Spectrometry (GC-MS) to identify the components present inside the oil. Energy content was also determined using bomb calorimeter to study the feasibility of the liquid product as fuel in diesel engine.

3 Results and Discussion

3.1 Effects of microwave power on the pyrolysis of PP

Table 1: Effects of various microwave power on temperature and product yield

Type of sample	Mass of plastic (g)	Mass of metal (g)	Power (W)	Time (min)	T _{max} (°C)	Max rate (°C/min)	Mass of wax (wt. %)	Mass of oil (wt. %)	Mass of gas (wt. %)
PP + Fe pw	50.0	10.0	700	30	460	110	-	54.5	45.5
PP + Fe pw	50.0	10.0	600	30	440	90	2.8	44.7	52.5
PP + Fe pw	50.0	10.0	500	30	<400	70	40.2	22.4	37.4

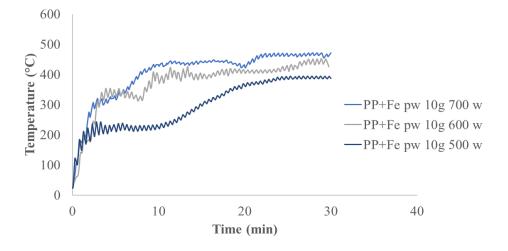


Figure 3: *Temperature vs time for various microwave power* (500-700W)

The effect of microwave power was determined by carrying out the pyrolysis of polypropylene (PP) in a borosilicate glass reactor containing iron powder at various power of 500 - 700 W. Based on Figure 3, the reaction temperature increased rapidly in the first few minutes and gradually stabilised at the end of the experiment (30th min). The iron powder absorbed the microwave radiation and heated up the plastic. The PP plastic sample was fully converted to either oil, wax, or gas.

The results of the effect of variation in microwave power on temperature and product yield was tabulated in Table 1. The increase in microwave power from 500 to 700 W increased the oil yield from 22.4 to 54.5 wt.% and decreased the wax yield from 40.2% to zero. The temperature and heating rate rose with the increase in power which was in accordance with the theory. The increase of input power improved the electromagnetic density and intensified the alternation of electric field. This increased the energy density of the electrons escaping from the metal and made the electrons and the surrounding gas medium collided more frequently [12]. At higher microwave power of 700 W, the temperature reached 460 °C and provided more energy to break the bonds between the plastic carbon chain, thus, more oil (shorter carbon chain) was produced. At 500 and 600 W, the lower temperature caused a lower degradation rate, thus producing greater amount of wax (longer carbon chain) and less oil.

3.2 Effect of mass of iron powder on PP

Plastics are normally microwave-transparent, that is, they cannot be heated up significantly in microwave since they have low dielectric constant. Thus, the microwave heating rate and temperature depend on microwave-metal interaction instead of plastic. PP Plastic was further pyrolysed with iron powder of 5-15 g for 30 min at microwave power of 700 W. The metal powder was a good absorber of microwave, unlike bulk metals which would reflect most of the microwave radiation. For most metals and good electrical conductors, the magnetic loss was more dominant, and they were more efficiently heated in the magnetic field (H) than the electric field (E). The iron powder was heated through eddy current loss and magnetic reversal loss mechanism, allowing it to reach high temperature of more than 400 °C [13].

Table 2: Effects of mass of iron powder on temperature and product yield

Type of sample	Mass of plastic (g)	Mass of metal (g)	Power (W)	Time (min)	T _{max} (°C)	Max rate (°C/min)	Mass of wax (wt. %)	Mass of oil (wt. %)	Mass of gas (wt. %)
PP + Fe pw	50.0	5.0	700	30	396	35	-	20.0	80.0
PP + Fe pw	50.0	7.5	700	30	392	48	-	32.6	67.4
PP + Fe pw	50.0	10.0	700	30	460	110	-	54.5	45.5
PP + Fe pw	50.0	15.0	700	30	420	69	-	50.1	49.9

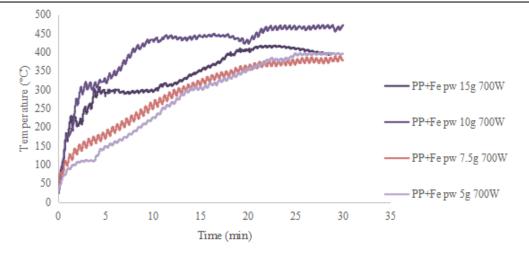


Figure 4: Temperature vs time for pyrolysis of PP with iron powder 5-15 g at 700 W

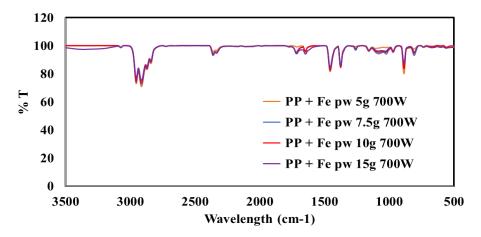
The variation in temperature and yield with mass of iron powder were presented in Table 2. The temperature for 10 g of iron powder was the highest at 460 °C followed by 15 g, 7.5 g and 5 g at 420, 396 and 392 °C respectively (Figure 4). As the mass of iron powder increased from 5 to 10 g, the temperature and heating rate increased due to the availability of more susceptors to absorb microwave radiation. But as the mass of iron powder increased from 10 to 15 g, the temperature decreased due to the bulk heating effect of the metal. The pyrolysis of PP with iron powder 10 g yielded 54.5 wt.% oil which were the highest, compared to 20.0%, 32.6% and 50.1% for 5, 7.5 and 15 g respectively. No wax was produced from the pyrolysis of PP with all mass of iron powder.

3.3 Calorific content of pyrolysis oil

The calorific value of pyrolysis oil from the microwave pyrolysis of PP was found to be 45-46 MJ/kg, which was comparable to diesel (42-46 MJ/kg) and petrol (44-46 MJ/kg). The heating value is also similar to the value of pyrolysis oil from PP obtained by Arshad, et al. [14] which is at 46.7 MJ/kg.

3.4 FTIR

Figure 5 showed the curve of FTIR analysis for the sample. The spectrum was representative of C-H stretching of CH₂ (2958 cm⁻¹), CH₂ symmetric and asymmetric stretching (2914 cm⁻¹), C=C stretching (1456 cm⁻¹), C-H bending (1375 cm⁻¹) and C-H out of plane bending (887 cm⁻¹). Thus, the pyrolysis oil mainly consisted of alkane, alkene and cycloalkane functional groups.



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Figure 5: FTIR analysis of pyrolysis oil from the microwave pyrolysis of PP

3.5 GC-MS

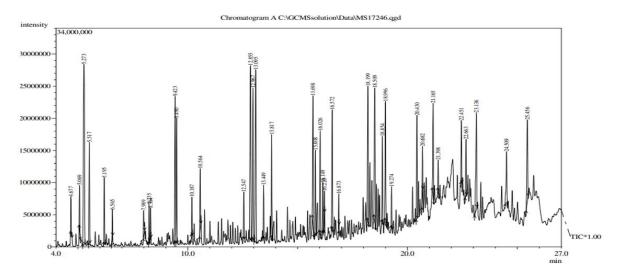


Figure 6: GCMS of pyrolysis oil from microwave pyrolysis of PP with Fe powder

The pyrolysis oil of PP with iron powder contained aliphatic hydrocarbon (alkane, alkene, cycloalkane) such as 2,4-dimethyl-1-heptene, hexacontane, 2,5-dimethyl-2-undecene and cyclohexane, 1,2,3,5-tetraisopropyl, but did not contain any aromatic compounds. The oil also contained alcohol (~OH group) compound. However, the PP structure (C₃H₆) n did not contain any oxygen in the PP chain. The oxygen might come from the atmospheric gas, where oxygen was not fully removed by the nitrogen gas at the beginning of the experiment. Besides, the oxygen traces might come from the metal powder which oxidized in the environment.

4 Conclusion

Microwave pyrolysis with iron susceptor serves as an effective process for the conversion of PP plastic into value added material such as fuel like oil compared to conventional pyrolysis. The oil yield of PP with iron powder increased from 22.4 to 54.5 wt.% with the increase of microwave power from 500 to 700 W. Simultaneously, the wax yield decreased from 40.2% to zero. 10 g is the optimum value of iron powder addition to produce the highest oil yield with no wax formation. The obtained pyrolysis oil contained aliphatic hydrocarbon (alkane, alkene, cycloalkane), but did not contain any aromatic compounds. The pyrolysis oil exhibit a high calorific value of 45-46 MJ/kg comparable with the commercial fuel, thus it has high potential to be used in diesel engine application.

5 Declarations

5.1 Acknowledgements

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5.2 Competing Interests

There is no conflict of interest.

5.3 Publisher's Note

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