

## CONTROLLED MICROWAVE-INDUCED PYROLYSIS OF WASTE RUBBER TIRES

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### ABSTRACT

Pyrolysis is the thermal degradation of a carbonaceous solid by heat in the absence of oxygen. The feedstocks, such as biomass or solid wastes, are heated to a temperature between 400 and 600°C. The reaction produces three products: gas, pyro-fuel oil, and char. Pyrolyzing solid wastes to make pyro-oil and bio-char provides a renewable, low cost way to produce liquid fuel. The pyro-oil could be mixed with diesel fuel and the fuel properties suitable for a diesel engine generator could be investigated. This work uses a controlled microwave heating technique to pyrolyze used rubber tires into pyro-oil. The thermal treatment of received used rubber tires was done in a modified domestic microwave with a controlled heated stirred bed system. In earlier work, it was found that rubber tires are a poor absorber of microwaves. An appropriate microwave-absorbing material, such as activated char, was added to initiate the pyrolysis process, thus producing pyro-oil. The characteristics of this pyro-oil and the effect of the microwave absorber on the yield of pyro-oil are presented in the paper. Particular attention was paid to the temperature profile during microwave heating of the used rubber tires. The benefit of this application is the conversion of the waste tires into renewable and high calorific pyro-oil. In addition, properties of tire pyrolysis oil have been determined and compared with the results of commercial diesel fuel.

*Keywords:* Characteristics; Microwave; Pyrolysis; Pyro-oil; Waste rubber tires

### 1. INTRODUCTION

In recent decades, worldwide disposal of non-ecofriendly solid wastes, such as polymeric materials, has increased rapidly. Inevitably, it poses very complex environmental problems, such as landfilling and underground water pollution, especially in vast developing Southeast Asian countries, such as Malaysia, Thailand and Indonesia. With the growth of the industrial manufacturing sector, coupled with ignorance of environmental sustainability, a lot of recyclable and reusable solid waste materials are disposed in the environment around the world. For example, with the development of the automotive sector, the number of waste tires being generated increases daily. Conservative estimation of waste tires produced annually are of the order of 2.5 million tons per year, 1.5 million tons per year and 0.5 million tons per year, in North America, the European Union and Japan, respectively (Williams et al., 1990).

Waste tires are disposed of in landfill sites, which pollutes the environment and breeds disease-carrying vermin. However, the negative effects on the environment in landfills can be mitigated by energy recovery and by extracting constituent chemicals by using current technologies (Shah et al., 2008). Moreover, there are multiple alternatives for waste tire management, such as

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retreating, chemical reclaiming, direct incineration and pyrolysis. These are ways to obtain alternative fuels from waste (Murugan et al., 2009).

Pyrolysis is a thermal treatment process in the absence of oxygen and under inert conditions. Pyrolysis processing of waste tires allows complex organic volatile matter to be decomposed into lower molecular weight products constituted of solid, gases and liquids that can be used as fuels, additives or chemical feedstock (Rodriguez et al., 2001). In short, it creates three useful classes of products from waste tires thermally degraded under inert conditions: solid, liquid and gas (Scheirs & Kaminsky, 2006; Kawser & Ani, 2000a).

A conventional pyrolysis furnace consumes considerable electrical energy and results in deficient product quality due to secondary cracking reactions during pyrolysis. Recent microwave pyrolysis technology offers more benefits as compared to conventional pyrolysis methods and has received broad acceptance in the thermal chemistry community (Appleton et al., 2005). In microwave pyrolysis, electromagnetic waves can be transmitted throughout the material instantaneously, unlike in conventional pyrolysis, in which heat energy is transferred based on convection and conduction. Microwave pyrolysis technology brings vital advantages in terms of greater instantaneous volumetric heating, enhanced product quality and better chemical reactivity (Fernandez et al., 2011).

Depending on their dielectric properties, feedstocks are grouped into insulator, conductor, absorber or mixed absorber of microwaves. Good microwave absorbers, such as carbonaceous activated carbon, graphite and char, are generally added as catalysts to induce pyrolysis (Menendez et al., 2010). In the previous research literature, experimental parameters, such as type of pyrolysis reactor, heating rate, temperature, nitrogen gas flow rate and use of catalyst, were commonly investigated. However, insufficient research has been done regarding the microwave heating method. It is important to investigate the impact of microwave heating on yield of waste tires pyro-oil yield.

Based on the above points, we selected microwave heating temperature and the use of a catalyst as parameters to examine in this study. In this study, automotive waste tires were pyrolyzed at temperatures between 400°C and 600°C, with or without activated carbon. The effects on pyro-oil yield and on the properties of products were studied. The calorific values of liquid products were investigated for the representative temperature of 600°C. The physical and chemical properties of the raw materials were examined by several techniques, namely, elemental analysis and proximate analysis. In this study, the effects of microwave cavity temperature and use of a catalyst on both pyro-oil mass and energy yields were investigated.

## **2. MATERIALS AND METHODS**

### **2.1. Materials**

The waste tire crumb used in this experiment was obtained from a solid waste recycling company located in Sarawak, Malaysia. Waste tires were ground to crumbs of 2.0 mm size. Fine coconut-activated carbon of 3.0 mm size obtained from the Gaia Group of Companies, Malaysia, was used as a microwave absorber to initiate heating. The difference in particle size of the activated carbon and the waste tire crumb facilitated the separation of the activated carbon for reuse after pyrolysis. Both the proximate analysis and the elemental analysis are tabulated in Table 1, while the thermogravimetric analysis (TGA) and derivative thermogravimetric (DTG) analysis are illustrated in Figure 2.

### **2.2. Detailed Method and Experimental Procedures**

All of the pyrolysis experiments were carried out on a lab-scale pyrolysis system. Fabrication and microwave rig modification were similar to the setup in previous experiments (Abubakar et al. (2013) However, a proportional–integral–derivative controller (PID) controller was added to

control the temperature. The main parts of the pyrolysis system were modified to include a conventional microwave with a maximum adjustable power of 800W and a quartz rig. The quartz rig, 150 mm in length and 100 mm in diameter, was used as a reactor to contain feedstock. The liquid product collecting and condensing system consisted of two glass traps (cylindrical in shape and connected to each other with pipelines) filled with cooling solution conducted from a Lauda water-cooling system.

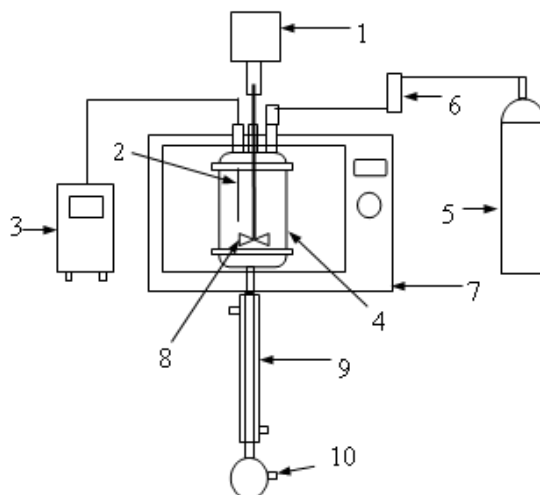


Figure 1 Setup of Microwave assisted pyrolysis system: (1) Electrical motor; (2) Thermocouple; (3) PID temperature feedback controller; (4) Quartz rig; (5) Nitrogen gas tank; (6) Flowmeter; (7) Conventional Microwave; (8) Stirrer blade; (9) Condensing unit; (10) Liquid collector

Two series of experiments were performed in order to investigate the effects of heating temperature and the use of a catalyst on the yields of pyrolysis products (pyro-oil, char and gases) and on the physical properties of pyrolytic liquids. For each series of experiments, about 100 g of feedstock was loaded directly into the reactor (for the various heating temperature runs) or was physically blended with activated carbon in ratios of 1:1 by weight and placed in the quartz rig (for the effect of catalyst runs). Figure 1 shows the quartz rig was covered with three inlet channels on the glass lid at the top and a single outlet on the lid at the bottom. The stirrer was introduced from the central channel of the lid, while the other two channels were used as a thermocouple and a nitrogen gas inlet. A fine wire mesh of 60 microns was used to support the feedstocks loaded into the quartz rig and also was used to filter vapors before the condensation process at a condensation unit. A double-bladed steel stirrer with a shaft length of 20 cm was powered by a 90W electrical motor with an adjustable angular speed. The internal temperature of the reactor was controlled by the PID controller with a K-Type thermocouple. Temperature control of the internal reactor was possible as the microwave field in the cavity could be controlled based on a duty cycle or via ON/OFF mode with the aids of PID controller.

The condensing system was precooled to 5°C before the commencement of the experiment and the loaded quartz rig was put inside the microwave. Then the air inside the rig was eliminated with a nitrogen flow of 1 L/min. The stirrer was set to an angular speed of 50 rpm to ensure systematic heating of the feedstock. During the heating process, the PID controller was set to the desired temperature (final holding temperature and duration) under certain conditions (a constant increment of heating temperature at 10°C/min and nitrogen flow rate at 0.5 L/min). The duration of pyrolysis was set to 60 mins to ensure completion of the waste tire pyrolysis. After the experiment, the liquid products (pyro-oil and water) condensed in the collector were weighed. Then, after cooling the reactor, the char was collected and weighed.

Table 1 Proximate, elemental analyses and HHV of scrap tires

Elemental analyses	Values (wt%)	Proximate analyses (wt%)	Values (wt%)	Higher heating value (HHV) (MJ/kg)
Carbon (C)	78.1	Moisture content	1.05	29.80
Hydrogen (H)	8.3	Ash content	18.90	
Nitrogen (N)	0.4	Volatile	57.81	
Sulfur (S)	3.2	Fixed Carbon	22.24	
Oxygen (O)	10.0			

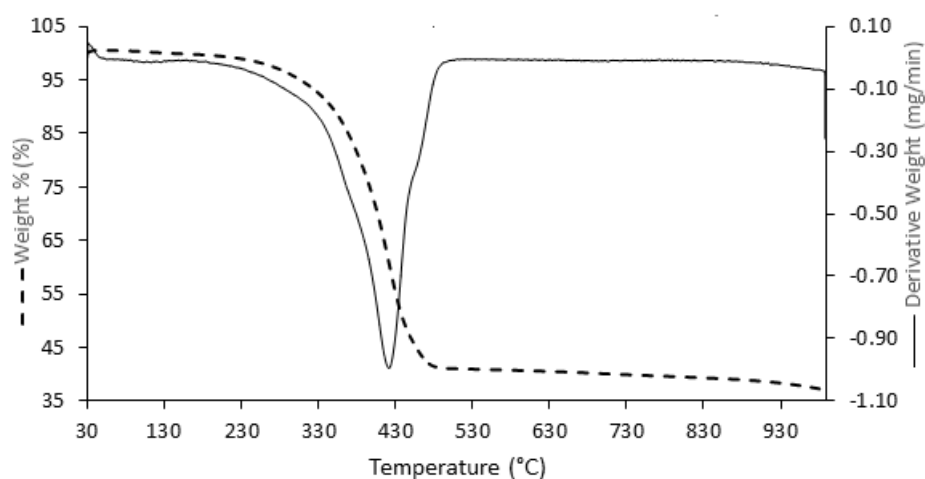


Figure 2 TGA and DTG curves of waste tire

### 3. RESULTS AND DISCUSSION

#### 3.1. Microwave Heating Temperature

The microwave heating temperature during the pyrolysis run was recorded. Figure 3 illustrates the temperature profile during the pyrolysis experiment for the heating temperature of 500°C. It can be observed that with activated carbon as the catalyst, the temperature in the rig rose sooner. Compared to runs of this experiment without activated carbon as the catalyst, the heating temperature of microwave pyrolysis without the catalyst increased slightly slower. However, the rate of temperature rise during pyrolysis without a catalyst remained steady because waste tire is already a carbonaceous material and to a reasonably good absorber of microwaves. During the holding period, temperature in the reactor only increase slightly after adding activated carbon. This is because of the selective heating nature of activated carbon at a high mixing ratio (1:1). Namely, the activated carbon gets heated due to localized heating and the energy fails to be transferred efficiently to the feedstock (Abubakar et al., 2013).

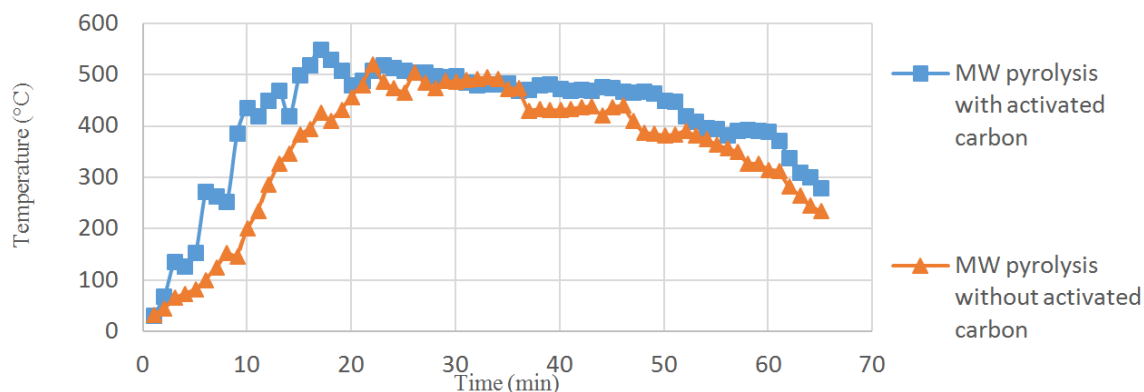


Figure 3 Comparison of the temperature profile of microwave pyrolysis with and without activated carbon

### 3.2. Mass Yield of Pyro-oil, Char and Gases

In these experiments, the two operational parameters used were microwave pyrolysis heating temperature and the use of activated carbon as a catalyst. The two series of experiments were carried out at heating temperatures from 400 to 600°C. For the run without using activated carbon as catalyst, the yield of liquid product ranged from 19.03 to 28.63 wt%. The total yield of pyro-oil without use of activated carbon increased and was enhanced by higher temperatures, as shown in Figure 4. The optimum temperature of microwave pyrolysis without activated carbon was between 550 and 600°C.

In contrast, the results for the experiment with activated carbon as a catalyst showed the highest liquid yield of 54.39 wt% at 500°C. Figure 5 showed that for the run with activated carbon as the catalyst, the yield of liquid product increased from 27.46 wt% at 400°C to 54.39 wt% at 500°C, but decreased to 39.61 wt% at 600°C. The experimental results showed that utilization of activated carbon for catalysis enhanced the yield of liquid product when the heating process was initiated.

Meanwhile, the literature reports that the optimum temperature for microwave pyrolysis with activated carbon as a catalyst is between 400 and 500°C (Ani and Nor, 2012). In conventional pyrolysis, the effect of temperature on scrap tire pyro-oil yields follows a normal distribution curve in which the pyro-oil yield first increases and then decreases with temperatures. The optimum temperature for scrap tire pyro-oil yield falls at the temperature of 500°C. The curve is caused by the upward reaction that produce oil from scrap tire and the downward reaction that produce gases from the pyro-oil. As the temperature increases after the optimum point, the yield of pyro-oil decreases gradually. This is because above the optimum temperature, the high temperature reaction convert liquid to gases becomes significant. This basic nature of pyrolysis process is the reason behind the normal distribution curve was observed.

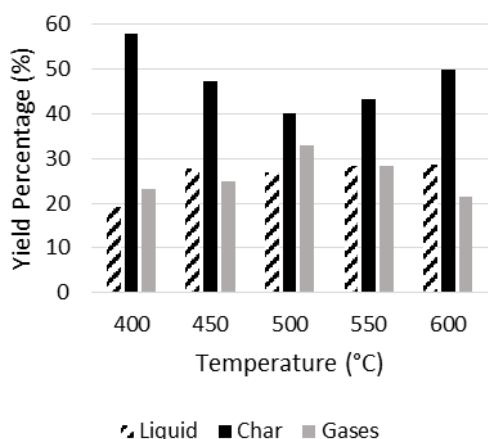


Figure 4 Percentage of product yield from waste tire pyrolysis without activated carbon

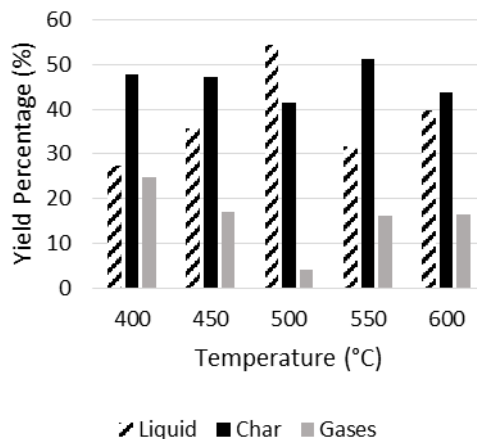


Figure 5 Percentage of product yield from waste tire pyrolysis with activated carbon

### 3.3. Fuel Properties

The scrap tire pyro-oil was tested for the fuel properties, including elemental composition, calorific value, density and viscosity. For the experimental run without activated carbon as the catalyst, a calorific values with increasing reaction temperatures were in a downward trend from 43.67 to 42.09 MJ/kg, as shown in Figure 6. Using activated carbon as the catalyst, the calorific values of pyro-oil showed that pyrolysis at 400°C yielded higher calorific values at 600°C and the calorific values for downward trend as the temperatures increases with slightly higher calorific values of 43.70 to 42.59 MJ/kg.

Previous studies have shown that at higher pyrolysis temperatures cause an increase in aromatic compounds and a decrease in aliphatic compounds (Cypres et al., 1989). In addition, temperature rises cause secondary reactions of propene, ethylene and butadine. The process also result in the production of polyaromatic and aromatic compounds such as naphthalene and benzene.

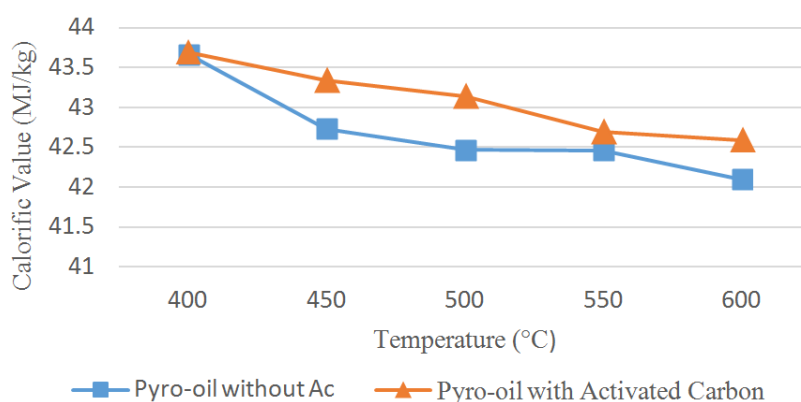


Figure 6 Calorific value of pyro-oil at various pyrolysis temperatures

Table 2 shows the physical properties of pyro-oil derived from microwave pyrolysis of waste tires; the fuel properties of pyro-oil and commercial diesel are also tabulated. The comparison shows that the calorific value is close to that of commercial diesel and reflects the feasibility of using pyro-oil as a substitute for diesel fuel. The density of the pyro-oil is 0.962 kg/L, which is slightly higher than that of commercial diesel fuel. Both microwave pyro-oil without, and pyro-oil with, activated carbon as a catalyst have a high calorific value at 43.67 MJ/kg and 43.70

MJ/kg, respectively. The significant calorific values indicate that it would be feasible to use pyro-oils directly as fuel substitutes or petroleum refinery feedstocks (Dai et al., 2001).

Table 2 Characteristics of the results for microwave pyro-oil in comparison to pyro-oil reported in the literature, commercial diesel and bio-diesel

Content analysis % wt	Waste tyre pyrolysis oil	Tire rubber pyrolysis oil*	Diesel fuel**	Bio-diesel fuel***
Carbon	83.0	82.98	84.76	-
Hydrogen	9.20	7.63	14.96	-
Nitrogen	0.50	0.12	0.01	-
Oxygen	5.98	8.57	0.00	-
Sulphur	1.31	0.50	0.28	-
C/H	9.13	10.87	5.67	-
Ash	0.10	0.050	-	-
Calorific value (MJ/kg)	43.70	43.41	45.50	40.87
Kinematic viscosity at 50°C	6.53	6.66	-	3.19
Density at 15°C, kg/L	0.96	0.93	0.84	0.87

(\*Kawser & Ani, 2000b; \*\* Yi & Ani 2012; \*\*\*Motasemi & Ani, 2011)

Moreover, pyro-oil has a low viscosity of 6.53 cST at 35°C, a beneficial feature for use in a conventional diesel engine. However, this value is slightly higher than those of several ranges of conventional diesel fuels (Islam et al., 2006). In short, properties of pyro-oil, such as elemental composition, density, calorific value and viscosity (see Table 2) are similar to those of conventional diesel fuel. The physical characterization of current microwave pyro-oil shows that the carbon content and calorific value are close to various results in previous pyro-oil studies (Islam et al., 2006; Ucar et al., 2005). Thus, good physical characteristics of pyro-oil increase the plausibility of directly using pyro-oil or of blending it with conventional diesel fuel (Kawser & Ani, 2000c).

#### 4. CONCLUSIONS

The study implies the mitigation of environmental pollution by conversion of waste tires into useful fuel. Microwave pyrolysis is capable in converting waste tires into various useful products, such as pyro-oil, char and gases. However, the calorific values of both series of pyro-oils achieved their maximum value of 43.70 MJ/kg. So, this is a promising substitute for petroleum fuel. The research has proven the ability of microwave pyrolysis to convert waste tires into a useful fuel. The motivation to use scrap tires with activated carbon is an attractive option to reduce carbon dioxide emission and step forward towards clean energy utilization. Setup of large scale including pilot scale and industrialization could decrease land pollution. Addition of activated carbon as a catalyst in microwave pyrolysis has the potential to enhance the yield of liquid and saving input energy. This is because the heating temperature rise faster and reduce the heating time, so saving in term of electrical energy.

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