MICROWAVE PYROLYSIS OF SCRAP TIRES AND ITS PYROLYSIS OIL PERFORMANCE IN DIESEL ENGINE

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Specially dedicated to *the Almighty, parents*
and *friends*

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ABSTRACT

Microwave assisted pyrolysis of scrap tires allows recovery of energy and useful materials, such as pyrolytic oil, char and gases. Scrap tire were being heated in inert atmosphere at temperature between 400 and 600 °C to produce liquid fuel. In this study, a modified conventional microwave and equipped with a custom made quartz reactor was used in pyrolysis process of scrap tire. Microwave pyrolysis process were performed with and without activated carbon as microwave absorbent. The effect of heating temperature and activated carbon on pyrolysis yield were studied. Pyrolytic oil was characterized for calorific value, composition and compound functional group. Temperature of 500 °C was the optimum temperature for the highest yield of pyrolytic oil at 54.39 wt% was obtained at the run of experiment with activate carbon as microwave absorbent. The obtained tire pyrolytic oil possessed high calorific value in range of 42.09 – 43.07 MJ/kg. The benefit of this thermal treatment was conversion of waste material into high calorific pyrolytic oil, which could be burnt directly in an unmodified diesel engine. Moreover, tire pyrolytic oil was blended with petroleum diesel and biodiesel at different ratio for performance and exhaust emission study. Engine performance such as engine torque, engine brake power, brake specific fuel consumption and brake thermal efficiency were examined with different blend ratio of fuel. Results showed neat pyrolytic oil showed an average of 7.93% lower torque and emission of carbon monoxide (CO), hydrocarbon (HC), nitrogen oxides (NO_x) and sulphur dioxide (SO_2), at an average of 207.4 %, 201.7 %, 42.5 % and 580.7 % higher, respectively than that of petroleum diesel fuel. At an optimum temperature of 500 °C the consumed electrical energy required to produce per unit mass of tire pyrolytic oil was 2.698 kWh/kg.

ABSTRAK

Pirolisis tayar buangan dengan gelombang mikro membolehkan pemulihan tenaga dan bahan berguna, seperti minyak pirolisis, arang karbon dan gas. Tayar buangan telah dipanaskan dalam suasana lengai pada suhu dari 400 sehingga 600 °C untuk menghasilkan bahan api cecair. Dalam kajian ini, ketuhar gelombang mikro konvensional yang diubahsuai dan dilengkapi dengan reaktor kuarza telah digunakan dalam proses pirolisis tayar buangan. Proses pirolisis gelombang mikro telah dilakukan dengan dan tanpa karbon diaktifkan sebagai penyerap gelombang mikro. Kesan suhu pemanasan dan karbon diaktifkan pada hasil pirolisis telah dikaji. Minyak pyrolytic dicirikan untuk nilai kalori, komposisi dan kumpulan berfungsi kompaun. Suhu 500 °C adalah suhu optimum dengan penghasilan minyak pirolisis tertinggi sebanyak 54.39 wt% telah diperolehi pada eksperimen dengan karbon mengaktifkan sebagai penyerap gelombang mikro. Minyak tayar pirolisis diperolehi memiliki nilai kalori yang tinggi dalam lingkungan 42.09 - 43.07 MJ/kg. Proses rawatan haba ini bermanfaat kerana membolehkan penukaran bahan buangan ke dalam minyak pirolisis yang berkalori tinggi dan boleh dibakar secara langsung dalam enjin diesel tanpa diubahsuai. Selain itu, minyak tayar pyrolisis telah dicampur dengan diesel dan biodiesel pada nisbah yang berbeza untuk kajian prestasi dan ekzos pelepasan. Prestasi enjin seperti daya kilas enjin, kuasa enjin brek, kecekapan penggunaan bahan api dan kecekapan enjin yang telah diperiksa dengan nisbah campuran bahan api yang berbeza. Hasil kajian menunjukkan minyak pirolisis tulen menunjukkan purata daya kilas 7.93 % lebih rendah berbanding dengan diesel. Manakala, pelepasan karbon monoksida (CO), hidrokarbon (HC), nitrogen oksida (NOx) dan sulfur dioksida (SO_2), lebih tinggi pada purata 207.4 %, 201.7 %, 42.5 % dan 580.7 %, masing-masing berbanding dengan bahan api diesel. Pada suhu optimum 500 °C, tenaga elektrik yang diperlukan untuk menghasilkan seunit jisim minyak pirolisis tayar adalah 2.698 kWh/kg.

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LIST OF ABBREVIATION

A	-	Ash
BSFC	-	Brake specific fuel consumption
BTE	-	Brake thermal efficiency
CBSSs	-	Carbon based solid substances
CV	-	Calorific value
FC	-	Fixed carbon
FT-IR	-	Fourier transform infrared spectroscopy
GCMS	-	Gas chromatography mass spectrometry
HHV	-	Higher heating value
LHV	-	Lower heating value
M	-	Moisture
PAHs	-	Polycylic chromatic hydrocarbon compound
TPO	-	Tire pyrolytic oil
VM	-	Volatile matter

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CHAPTER 1

INTRODUCTION

1.1 Introduction

In order to combat petroleum depletion, the use of renewable energies play a mightily important role. As we know the consumption of energy resources are growing gradually and getting demanding day by day. So, both the environmental conservation subjects including the finding alternative sources of energy for the continuously depletion of petroleum reserves and the environmental issue relating to the disposal of solid wastes take part in among important research subject that researchers studied densely on. Furthermore, negative impact of waste rubber tires disposal is getting critical as the growth of automotive industries in recent decades. Regrettably, about 65 – 70 % of these scrap tires are disposed legally or illegally landfills, or are exposed to open air. In fact, both cases cause severe environmental pollutions, threatening situations, and high loss of added value materials (Boxiong *et al.*, 2007b; Galvagno *et al.*, 2002). In sustainable environment, management of waste rubber tires is one of the most important issues that should be controlled. Conservative estimation indicate that over one billion scrap tires are produced annually (RMA, November 2006).

It is crucial to develop a feasible and strategic investigation about practicable innovation together with long-term development in the management of waste rubber tires. Pyrolysis technology is one of the preeminent alternative renewable energy that provide the only source of renewable solid, liquid and gaseous fuels. Moreover, pyrolysis is a favorable ecofriendly alternative source of renewable energy in the context of contemporary energy scenario (Ward *et al.*, 2014). From pyrolysis we can obtain multiple types of renewable fuels and particular attention is given on liquid state high calorific pyrolysis oil. This technology provide the best way mitigation of pollution including reducing greenhouse gases. Pyrolysis oil can be considered as a method of energy security which perform as an alternative of fossil fuels that are shortage in supply. Nowadays, the utilization of this renewable energy expanded around the world. It has high potential to be developed in populated growing regions especially South East Asia. Pyrolysis technology can be a good way to manage disposed scrap tires because it allows recovery of useful materials and energy. These refined materials can be used as source of chemicals or energy in industries.

Among variable types of pyrolysis process, microwave assisted pyrolysis shows a remarkable alternative to conventional heating because microwave can heat rapidly and directly on any microwave absorbing materials with significant reduction of reaction time (Andrea *et al.*, 2011). Microwave assisted pyrolysis is extensively applied to treatment of various types of feedstocks. First, it is widely applied to biomass especially for the production of bio-oil (Abubakar *et al.*, 2013; Salema *et al.*, 2011; Salema *et al.*, 2012), or biochar (Salema *et al.*, 2013). Microwave assisted pyrolysis is also applied in various treatment of polymeric waste, including polystyrene (PS) (Bartoli *et al.*, 2015) and polyethylene terephthalate (PET) (Siddiqui *et al.*, 2012). Gasoline-range hydrocarbons is produced by heating of polyethylene (PE) in the presence of catalyst (Zhang *et al.*, 2015). In order to improve microwave heating efficiency, activated carbon with high surface area promoted a better microwave absorbent is to be used as catalyst to transfer heat energy to polymeric materials including tires (Ani and Nor, 2012). Microwave pyrolysis allows to regulate yields and properties of liquid and gas by using different microwave power (Undri *et al.*, 2013). The production of microwave tire rubber pyrolytic oil and the use of its blend in internal combustion devices had received much attention.

1.2 Background of Study

Pyrolysis is defined as thermochemical decomposition of materials at elevated temperatures in range of between 400 °C – 800 °C under the absence of oxygen to produce mainly liquid, solid and gaseous products. In these decades, pyrolysis of waste rubber tires with different method conventional heating were reported in literature. The awareness of renewable energy utilization and waste management are causing development of this technology because of several issues, which include global warming and controlling over dependence of society on fossil fuels. It is also well-known that the high consumption of fossil fuels by our society has driven to its depletion and to a negative consequences on the environment mainly due to the greenhouse gases and the emission of harmful pollutants like sulphur dioxide (SO_2), nitrogen oxides (NOx) and particulates. Parallel to this, disposal of solid waste is increasing as the growth of human population around the world. From the data, world polymeric production in 2009 was 230 million tons, up to 54% of these materials are disposed as wastes (Sienkiewicz *et al.*, 2012). In spite of the fact that majority of these waste materials are non-biodegradable, they are disposed as landfill because recycling is not economically attractive. Used automotive rubber tires is categorized in to this kind of solid waste and it is a burden that adds significant cost over disposal and in many cases acts as a barrier to improve resource efficiency.

Besides that, the shortage of intellectual knowledge in both economic and technical mechanisms in waste reprocessing also causes that scrap rubber tires are considered a severe pollution in terms of waste management. Scrap automotive rubber tires have significant higher value of calorific value than coal as well as remarkable amount of carbon black, it is a great benefit to find an alternative to take advantage of its high calorific value in order to produce alternative fuels, greenhouse gases reduction and pollution mitigation. In recent years, pyrolysis technology is receiving attention and interest to solve the scrap rubber tires disposal issues while allowing energy recovery.

Microwave assisted pyrolysis of scarp rubber tires with the purpose of producing renewable fuel for the usage as a substitution fuel in internal ignition engines can be seen as environmental friendly, hygienic and efficient way of scrap rubber tires management. Microwave assisted pyrolysis (Zabaniotou *et al.*, 2003) is one of the most prospective technology in energy recovery process due to its competency of heating instantly and directly on any microwave absorbing material (Domínguez *et al.*, 2007; Menéndez *et al.*, 2004). Scrap rubber tires contain carbon black up to 30wt% which is perfect microwave absorbent. Carbon black is capable to transform the microwave radiation into heat within seconds, high temperature of 1556 K is reached in 120 s by using a power of 600 W (Tierney *et al.*). The product yield and the constitution of each fraction obtained in the tire pyrolysis primarily depend on the specific characteristics of the pyrolysis process applied (i.e. fluidized bed, jet bed reactor, vacuum pyrolysis reactor, fixed bed reactor, temperature and pressure) (Ucar *et al.*, 2005b; Uçar *et al.*, 2005; Unapumnuk *et al.*, 2008) and less on types of tires, because the essential component of the tires are more or less the same ("http://www.etra-eu.org/.").

In a previous study, a batch in term of ton of scrap rubber tires were pyrolysed to produce char, oil and gas (Williams *et al.*, 1998). In conventional pyrolysis, scrap rubber tires is heated in fixed bed reactor (Williams *et al.*, 2003). Generally the identifiable setup of non-microwave assisted pyrolysis consists of heating reactor, condensing unit and liquid collecting devices. It was reported in the literature (Laresgoiti *et al.*, 2004), scrap rubber tire with sample sizes of 2 - 3 cm wide, representative of a whole car tire, have been heated under flow of nitrogen in a 3.5 dm^3 autoclave at 300, 400, 500, 600 and 700 °C. Meanwhile, at temperature above 500 °C there is no effect of temperature on gas and liquid product yields were about 17 and 38%. Furthermore, catalysts have been applied in several studies for enhancing the product in term of quality and quantity during scrap rubber tire pyrolysis (Boxiong *et al.*, 2007a; Boxiong and Chunfei, 2007b), respectively.

Despite that, previous studies provides limited amount of data over the state of are of microwave assisted pyrolysis of different types of materials. However, the research studies are mainly targeted on the experimental and apparatus set up but less studies are concentrated on the use of the product in internal combustion devices. Therefore, this research consists a thorough studies of the governing variables influence on both yield and quality of the pyrolytic liquid products, including the effect of both use of activated carbon as catalyst and pyrolysis temperature. The aim is to provide essential studies to understand the microwave assisted pyrolysis process applied to scrap rubber tires, including the physicochemical properties of the products and their performance in internal combustion devices. In this studies, special attention is given on the scrap rubber tires management problem and the current alternative to reuses it. The literature review also includes information about pyrolysis technology and the properties of scrap rubber tires as feedstock for this process. Besides that, types of pyrolysis reactor and experimental condition for classifying the category of pyrolysis are also studied. In the literature, the governed experimental variables in the tire pyrolysis are including temperature, carrier gas flow rate, heating rate and volatiles residence time. However, temperature in microwave assisted pyrolysis plays an important roles in the pyrolysis yields. Special attention is given to the liquid yield in microwave assisted pyrolysis of scrap rubber tires, highlighting its properties as alternative fuel in compression ignition engines.

However, the efficiency of pyrolysis oil in internal combustion engines remains an issues makes it tough for this alternative energy to compete with conventional petroleum fuel. The utilization of internal combustion engines is the major contributors to the formation of greenhouse gases and pollutants. Emissions from internal combustion engines also causes negative impact on the environment due to their intrinsic toxicity, which release gases direct or indirect destroying the environment. Incomplete combustion in internal combustion engines releases a series of pollutants such as carbon monoxide, aldehydes, sulphur oxides, nitrogen oxides, polycyclic aromatic hydrocarbon, unburned hydrocarbon and heavy metals. In European countries, engine emissions such as hydrocarbon, carbon monoxide and nitrogen oxide are strictly regulated. In order to meet the European Emission Trading System (EU ETS) operational policies commencing from December 2007 and ending in December 2020, the Environment Agency has legislated more stringent controls regarding tolerable limits of exhaust gas emissions and diesel engine characteristics. Exhaust emission is highly affected by engine characteristics and fuel types.

In order to mitigate environmental issues caused by pollution and shortage of fossil fuel so development of alternative fuel must be taken into consideration. Development of tires pyrolysis oil is absolutely necessary for reducing the dependence of society on diesel fuel and mitigating land pollution caused by tire disposal. The mass collection of scrap rubber tires for tires pyrolysis oil production as alternative fuel also reduces the exploitation of petroleum fuels for energy generation and cost reducing. Tires pyrolysis oil has similar calorific value compared with diesel, which makes it a high potential fuel alternative to fossil fuel, and can be used in blends with diesel fuels in different proportions or neat, in the unmodified compression ignition engines. In spite of the fact that tire pyrolysis oil brings numerous remarkable positive aspects, numerous disadvantages should be evaluated, i.e. capability of conventional diesel engine to run on various fuels, engine emissions, power efficiency and chemical content of tire pyrolysis oil. Besides that, some properties to be considered are elemental content, ash content, moisture content and viscosity. These properties are closely related to the chemical composition of the scrap rubber tires used. Previous studies shows various blends of tires pyrolysis oil in diesel fuel are utilized in conventional diesel engine (İlkılıç *et al.*, 2011).

The studies shows when high blend of tire pyrolysis oil in diesel fuel is combusted in diesel engines, the engines shows downtrend in both efficiency and output performance due to the differences in physical properties between diesel fuel and tires pyrolysis oil. Meanwhile, emission test results of diesel engine also shows a significant difference along various proportion of blends between tires pyrolysis oil and diesel fuel. Basically, tires pyrolysis oil has slightly lower calorific value compared with diesel fuel. With the presence of elements such as heavy metal and Sulphur, these will affect the properties of tires pyrolysis oil, causing another issues on environmental pollution and tires pyrolysis oil's feasibility in internal combustion devices. Presence of Sulphur also changes physical and chemical properties of fuel combustion in term of decreasing pH value leading to an increase in corrosiveness. However, the high cost of input energy to produce per unit volume of tires pyrolysis oil can make tires pyrolysis oil unfeasible to completely replacing petroleum fuel. In the literature, different studies have been performed on pyrolysis oil production by using various method including conventional pyrolyzer, fluidizer bed, fixed bed, rotary kiln, but not as much studies was performed in diesel engine performance and emission using tires pyrolysis oil made from microwave pyrolysis technology. Tires pyrolysis oil produced by microwave heating has high potential to be used in internal combustion devices and bringing environmental benefits. It is observable scrap tires pyrolysis will control solid waste and it does not consume food crops like production of biodiesel. The purpose of current study is to evaluate the feasibility of scrap tire pyrolysis oil production by using microwave technology with addition of coconut activated carbon as catalyst. The series of research studies conducted are characterization of feedstock and products, liquid yield efficiency analysis, gases emission and combustion performance of its blends in unmodified compression ignition engines. The studies were operated in laboratory scaled, in the following steps; firstly characterization of feedstock, secondly production of scrap rubber tires microwave pyrolysis oil with catalyst, then finally the engine performance and emission characteristics of Yanmar N70 diesel engine using different ratio of tires pyrolysis oil, diesel and biodiesel blends were evaluated.

The observable advantages of using scrap rubber tires pyrolysis oil in diesel engines are (İlkılıç and Aydın, 2011):

i. Fully renewable and environmental protective by reducing solid waste disposal.
ii. In low blend with diesel can be utilized in compression ignition engine without any modification.
iii. The low volatility makes the pyrolysis oil easier for storage as conventional diesel.
iv. Non explosive due to high flash point makes the storage safe.

However, some of the disadvantage of utilizing scrap tires pyrolysis oil as fuel are (İlkılıç and Aydın, 2011):

i. Relative higher viscosity, flashpoint and sulfur content compared to diesel fuel.
ii. Relative higher sulfur dioxide (SO_2), carbon dioxide (CO), oxide of nitrogen (NO_x), unburned hydrocarbon (HC) at emission compared to diesel fuel.
iii. Generate lower engine torque and power due to lower calorific value compared to diesel fuel.
iv. Scrap tires pyrolysis oil cannot completely replace diesel fuel.

1.3 Problem Statement

Polymeric materials are referred to synthetic or naturally occurring polymer. Polymers can be categorized according to their types of source. These included the scrap rubber tires which is produced from matrix of natural rubber and synthetic rubber, synthetic plastics which are made from fossil fuel and also naturally occurred polymer including biomass substances. Waste polymeric materials can be treated as a renewable energy sources, because these high energy content substances can be reused and process into pyrolysis oil. Scrap rubber tires can be considered as alternative renewable energy source. The remarkable calorific value of scrap rubber tires made this polymeric material a strong alternative to become an energy source. Occasionally, scrap rubber tires is used directly as solid fuel in heating of steam boiler to power the steam turbine. However, the unhandy situation of scrap rubber tires has made inconvenience in storage and reusing it as solid fuel. As result of that, most of waste rubber tires are usually abandoned or thrown. This issues rise an alarming situation in waste management.

Generally landfilling is the ordinary way to eliminate excess waste. However, awareness of environmental sustainability and establish of stringent laws have led to the reducing of landfill method. Besides that, open air combustion is another usual way of eliminating waste rubber tires. However, open air combustion releases toxic gases and brings severe negative effects to the environment. In order to mitigate problems as mentioned, effective countermeasure have to be implemented in order to improve the management of waste rubber tires. Retreatment of waste rubber tires might be one of the best alternative in solving the problem as rubber tires possess high calorific value. Thermal treatment such as microwave pyrolysis transforms the waste rubber tires into liquid fuel, solid char and gaseous products which possess higher market value. The product of pyrolysis such as tire pyrolytic oil can be utilized in extensive types of application as previous studies have confirmed that the rubber tires pyrolysis oil possess comparable calorific value to conventional diesel fuel. However, the incurred cost in producing the pyrolysis oil is higher than the cost used in producing the equivalent amount of fossil fuel. In short, microwave pyrolysis with the aid of activated carbon as catalyst might be a solution to improve the liquid yielding efficiency. In addition, more studies have to be done to investigate the efficiency of tire pyrolysis oil in term of chemical characteristics and it combustions in internal combustion devices.

1.4 Objective of Study

The following objectives of research studies were set in order to achieve the aim:

i. Evaluation of physical, chemical properties of scrap tire and microwave tire pyrolysis oil.
ii. Perform pyrolysis of scrap tire by using microwave heating.
iii. Evaluation of diesel engine performance and emissions using different blend of diesel and biodiesel with various proportions of tire pyrolysis oil.

1.5 Scope of Study

i. Producing tire pyrolytic oil from granular form tire pellet with size of 1- 5 mm^3
ii. Perform pyrolysis of automotive tires using conventional microwave
iii. Perform engine performance test of tire pyrolytic oil by using a single cylinder Yanmar diesel engine with displacement of 0.32L

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