COMPARATIVE ANALYSIS OF MICROWAVE AND THERMAL PYROLYSIS OF USED TYRES TO PRODUCE LIQUID FUEL

BY

BETT, RONALD KIPKEMOI

A Project Report submitted to the School of Engineering, Department of Mechanical, Production and Energy Engineering in Partial fulfillment for the requirements of the Award of the Degree of

Master of Science

In

Energy Studies (Renewable Energy Resources)

MOI UNIVERSITY

2022.

DECLARATION

Declaration by Candidate

I declare that this project is my original work and has not been submitted for the award of a degree in any other university. No part of this thesis may be reproduced without the prior written permission of the author and/or Moi University.

Signed……………………… Date……26 November 2022…………

Bett Ronald Kipkemoi

Eng/Mes/16/19

Declaration by the Supervisors

This thesis has been submitted for examination with our approval as university supervisors.

Signed………………… Date……1 November 2022…………….

Prof. Anil Kumar

Department of Chemical & Process Engineering,

School of Engineering,

Moi University, Kenya

Signed…………………………………… Date……2nd November 2022……….

Prof. Siagi Zachary

Department of Mechanical, Production and Energy Engineering,

School of Engineering,

Moi University, Eldoret, Kenya

DEDICATION

This project report is dedicated to my wife Irine Chepkoech Bett who gave me moral support since I began my studies despite a tight schedule and other challenges. Besides, I dedicate to my daughters Victoria and Rita who wished I could be with them all the time but I had to leave them for some time as I went to do the experiments.

It is also dedicated to World Bank, ACE II PTRE for funding my studies and research. I would not have enrolled for masters without them.

Finally, I dedicate to Moi University, school of Engineering for creating an enabling environment for all students to study and carry out their research.

ACKNOWLEDGEMENTS

Fast and foremost I would delight in giving thanks the Almighty God, the source of knowledge and wisdom, for His providence and guidance from the inception to completion of this research work. Besides, I would like to appreciate ACEII- PTRE for the funding of my studies including research Special thanks goes to the Centre Leader Prof. Ambrose Kiprop on behalf of all centre staff. I would also like to express my sincere gratitude and much appreciation to my able projects supervisors: Prof. Anil Kumar and Prof. Z. Siagi both from Moi University school of Engineering for their unceasing guidance, support, timely advice, and constructive criticism at every stage of this research. I am overwhelmed to appreciate all members of the graduate school board, school of Engineering for their continuous encouragement during this work. I am also thankful to Mr. Tonui, laboratory technician from the department of Mechanical, Production and Energy Engineering, Moi University, for assisting me to use their facilities in the Automotive Engineering Laboratory during the entire project. I deeply appreciate my parents who were of full support to me, both financially and emotionally. I can’t end without appreciating any person who impacted on this project and by so doing motivated steps towards its completion.

ABSTRACT

Used tyres are non-biodegradable, and the current methods of disposal pose a threat to environment. They can be valorized through decomposition to produce alternative fuel using thermal pyrolysis technique. Microwave pyrolysis is an alternate method which uses microwave irradiation, saves energy, and is environmentally friendly. The main objective of this study was to perform microwave pyrolysis of used tyres (Triangle 1000/R 20/10.00X20) to produce liquid fuel and compare with thermal pyrolysis. The specific objectives were to study the effects of pyrolysis operating variables, optimization of liquid fuel yield for microwave pyrolysis and compare with thermal pyrolysis and characterization of liquid fuel. Microwave pyrolysis variables were microwave power, reaction time, and particle size while thermal pyrolysis variables were reaction temperature, reaction time, and particle size. Process variable levels for microwave were power levels of 20, 30, 40, 50, 60, 80, and 100%; reaction time as 8, 13, 18, 23, and 28 minutes. Thermal pyrolysis reaction temperatures were 200, 300, 400, 500, 600 and 700°C; reaction time 10, 20, 30, 40, 50, 60, 70, 80, 100 and 120 minutes. Particle sizes for both processes were 25, 50, 60, 100, 125 and 200mm^2. A domestic 900W microwave oven was modified to be used for microwave pyrolysis, while the thermal pyrolysis was carried out in a furnace, fabricated using furnace clay, rated 600W. In both processes, 500 ml round bottomed flask was used as a reactor. Design Expert 13 was used for data analysis and optimization, Gas chromatography–mass spectrometry (GC-MS) was used for chemical composition analysis, while the physiochemical properties of liquid were tested using standard methods. The yield of the liquid products for microwave and thermal pyrolysis was correlated as a quadratic function of the variables. Response surface methodology (RSM) was used to study the effects of variables and identify optimal points. For both pyrolysis, yield decreased as particle size increased. The yield increased with increase in microwave power for microwave pyrolysis optimal being 50% microwave power and similar trend was observed with temperature for thermal pyrolysis optimal being 500°C. Yield increased with increase in reaction time for both processes. For microwave pyrolysis, the highest liquid yield of 39.1 wt % was at 50% power, 18 minutes reaction time, and particle size of 25 mm^2; optimal yield of 40.4 wt. % in thermal pyrolysis corresponded to temperature of 500°C, time of 80 min for 60 mm^2 size. RSM gave conditions for optima close to that of experimental results. The calorific value for liquid fuel from microwave and thermal pyrolysis were 48.99 and 47.31 MJ/kg respectively. GC-MS analysis showed that oil comprised of complex mixtures of organic compounds with Limonene, Toluene and Xylene as major components. Both processes gave similar maximum yield but microwave process was superior due to 77.5% and 71.6% reduction in time and energy respectively. The fuel properties meet requirements for heavy fuel. Microwave pyrolysis was recommended because of savings in time and energy requirements. Refining of liquid fuel and up scaling of microwave pyrolysis is recommended for further research.

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ABBREVIATIONS

ANOVA:	Analysis of Variance
CCD:	Central Composite Design
CCRD:	Central Composite Rotatable Design
DoE:	Design of Experiment
FTIR:	Fourier Transform Infrared
GC:	Gas Chromatography
GCV:	Gross Calorific Value
KRA:	Kenya Revenue Authority
MP:	Microwave Power
NEMA:	National Environment Management Authority
NMR:	Nuclear Magnetic Resonance
PAH:	Polycyclic Aromatic Hydrocarbons
RSM:	Response Surface Methodology
RTD:	Resistance Temperature Detector
SMP:	Specific Microwave Power
TPO:	Tyre Pyrolysis Oil
WATMA:	Waste Tyre Management Kenya
WPPO:	Waste Plastics Pyrolysis Oil
WTPO:	Waste Tyre Pyrolysis Oil

CHAPTER ONE

INTRODUCTION

1.1 Background and Motivation

In most countries, the use of motor vehicles is increasing and as a result, disposal of automobile tyres is inevitable. In 2007, WATMA was formed to manage the disposal of used tyres. It was championed by NEMA to set up funds to be used in proper disposal of tyres in an environmentally friendly way while coming up with requisite policy and the regulatory framework (Gernod Dilewski, 2012). The members of WATMA comprises of manufacturers, transport companies and various relevant government departments and ministries. According to WATMA, in 2012, close to 2 million wastes tyres were generated in Kenya and this figure is increasing every year since more vehicles are purchased (Gernod Dilewski, 2012). About 1.5 billion tyres are generated each year (Williams, 2013) and most of them after completing their value chain ends up being wastes. However, there is a growing interest in pyrolysis as a waste to energy technology to treat tyres to produce valuable products. The most used reactors are screw kiln, fixed-bed (batch), vacuum, rotary kiln, and fluidized-bed (Williams, 2013).

Tyres are non-biodegradable and in Kenya, there are no facilities to process the resulting wastes into useful products such as liquid fuel. If such wastes are not properly handled, they can result in major ecological hazards. Presently in Kenya, most of the used tyres are collected and burnt in an open space so as to recover steel strap which is then sold to metal dealers at a fee. This is not a good method of disposing such wastes because in the process of burning, toxic fumes are released to the atmosphere leading to air pollution. Some of the hazardous emissions include fluorides, sulphuric acid, hydrogen chloride, dioxins and mercury.

Apart from problems related to disposal of tyres and plastics, there is also the energy crisis in most developing countries. Most industries depend on energy from the grid and petroleum fuels for all industrial processes which may not be sufficient in some sub Saharan countries (Kunio Takase, 2019). There is a need to look for alternative sources of energy to supplement the energy from the national grid. Used tyres possess properties such as HHV, volatile content and higher carbon which makes it a good raw material for a thermochemical conversion processes (Osayi et al., 2018) . Therefore, they can be used in a waste to energy conversion process to generate Tyre Pyrolysis Oil (TPO) that can be used as a source of energy in internal combustion engines upon further purification to replace petroleum fuels. Furthermore, they can be used directly as a heavy fuel in boilers. According to Campuzano, (Campuzano et al., 2020) Tire pyrolysis oil (TPO) comprises a complex mixture of hydrocarbons (HC), and forms one of the useful fractions that can be obtained from the pyrolysis of used tires. As a result of its high energy content (HHV ~ 43 MJ/kg), the use of TPO as a fuel in combustion systems is a promising approach for recycling used tyres (Campuzano et al., 2020).

According to Mamun (Mamun et al., 2015), TPO has a calorific value close to 54 MJ/kg which is higher than that of petroleum diesel and gasoline at 44.8MJ/kg and 47.3MJ/kg respectively. It is slightly lower than that of methane at 55.5MJ/kg. Upon distillation, it can be used to replace petroleum diesel or even used as a blend.

Plate 1.1 Open Burning of Used Tyres

1.2 Policies Regarding Waste Tyre Management in Kenya

1.2.1 The Strategic Alliance on Waste Tyre Management in Kenya

The main objective of this alliance was to come up with used tyre management strategy in Kenya(Gernod Dilewski, 2012). The Kenyan Government and the relevant private sectors in collaboration with NEMA provided a regulatory framework in the implementation of the strategies. It was to be done in two phases where the first phase was to come up with a concept of collection and conversion of used tyres to useful products. Upon successful completion of the first phase, the second phase will be to implement a waste tyre management system.

The partnership between the relevant stakeholders was formed in May 2011. The feasibility studies for collection and recycling was conducted, analyzed and agreed upon in April 2012. The core group of the stakeholders is GIZ, NEMA, Sameer Africa, KRA and tread setters. The consultations with the ministries of Environment and Finance are ongoing through NEMA to seek financial support.

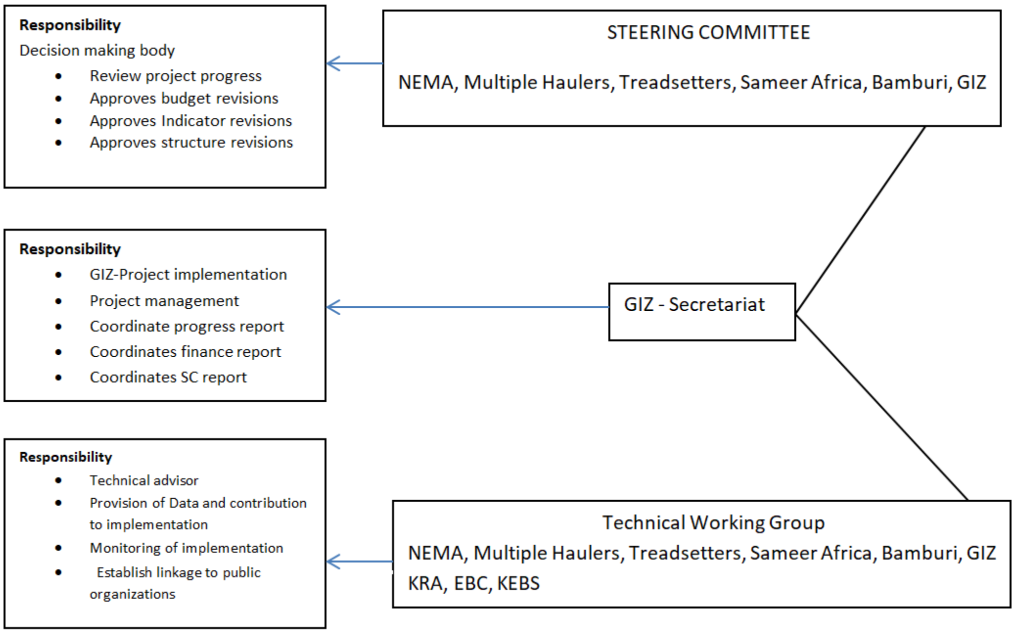


Figure 1.1: Waste Tyre management Association

1.2.2 The National Solid Waste Management Strategy

The strategy was formed in 2014 by NEMA (NEMA, 2014). The main objective of the strategy was to provide a way forward towards achieving a sustainable solid waste management in Kenya. The strategy was to address the following: the current situation of waste management in Kenya, the preferred state and finally, how to get to the preferred state.

The strategy categorized waste tyres as a waste stream that has reached their end of life because of wear and damage and hence cannot be reused or recycled. Apart from retreading, there are no formal methods for collecting and recycling used tyres in Kenya (NEMA, 2014). As a result, a higher percentage of used tyres are informally collected and burnt in open air to recover steel for recycling. This not only emits harmful gases that causes air pollution but also contaminates soil due to resulting residues. Currently in Kenya, there are only two facilities that uses used tyres to produce liquid fuel. NEMA has developed some relevant regulations regarding collection and disposal of used tyres and is awaiting gazettement.

1.3 Information about Waste Tyres in Kenya

The scope of this information includes amount and source of tyres, estimation of waste tyres, forecast and the final destination of waste tyres in Kenya.

Table 1.1: Tyre Imports into Kenya in 2011

Imported tyres per company [tons in 2011]	No. of Companies	Amount of tyres [tons in 2011]	Percentage of total weight
more than 1000	5	13,759	42%
250-1000	21	8,647	27%
100-250	36	5,176	16%
25-100	66	3,571	11%
0.001-25	462	1,390	4%
Total	590	32,543	100%

Table 1.2: Estimated amounts of waste tyres in Kenya in 2009

	Total Number of Vehicles in Kenya	Average Number of tires per Vehicle	Average Mass of tyre	Time period of use including retreading	Total Mass of Waste tyres	Total number of wastes tyres
	(no)	**(no)**	**(kg / tyre)**	**(years / tyre)**	**Tons**	**no**
Motor Cars	499,679	4	9	3.5	5,140	571,062
Utilities, Panel Vans, Pick-ups, etc.	219,901	4	25	2.5	8,796	351,842
Lorries, Trucks and Heavy Vans	91,431	10	50	2.5	18,286	365,724
Buses	26,558	8	50	2.5	4,249	84,986
Mini Buses/ Matatu	58,286	6	25	2.5	3,497	139,886
Trailers	27,039	6	50	2.5	3,245	64,894
Wheeled Tractors	25,091	4	60	5	1,204	20,073
Motor Cycles	239,104	2	5	4	598	119,552
Three Wheelers	13,856	3	6	3	83	13,856
Other Motor vehicles	20,138	4	15	3.5	345	23,015
Total	1,221,083				45,443	1,754,889

1.3.1 Source of tyres in Kenya

The major source of tyres in Kenya is tyre imports. According to Dilewski, (Gernod Dilewski, 2012) about 62% of all tyres in Kenya are imported. 20% of the tyres in Kenya are those that are imported with cars while those produced in Kenya and grey market account for 18%. Grey markets are those that are retreaded after being used for some time. To get the quantities of the tyres obtained from grey markets, tyre imports and those that are imported with tyres are subtracted from the total inventory of tyres.

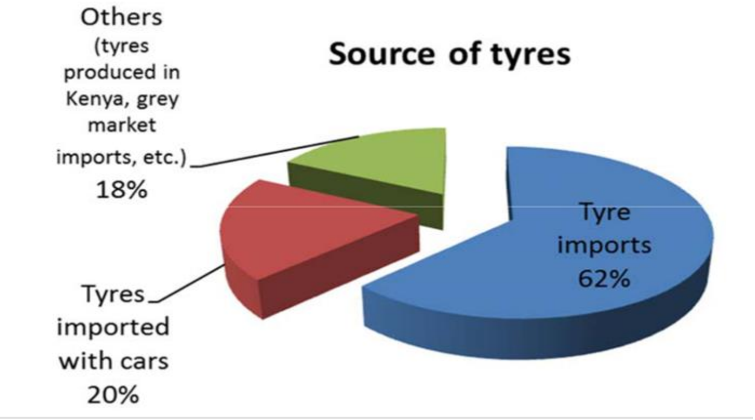


Figure 1.2: Sources of tyres in Kenya

1.3.2 Forecasted Quantities of Used Tyres in Kenya

The quantities of used tyres are expected to increase exponentially. This is due to the fact that the number of people and companies that buy new cars is increasing (Gernod Dilewski, 2012). In 2012, the total tones per annum (tons/a) of used tyres in Kenya were estimated at 51,000 tons/a and is expected to increase to about 130,000 tons/a in 2031 as shown in figure 1.3 below:

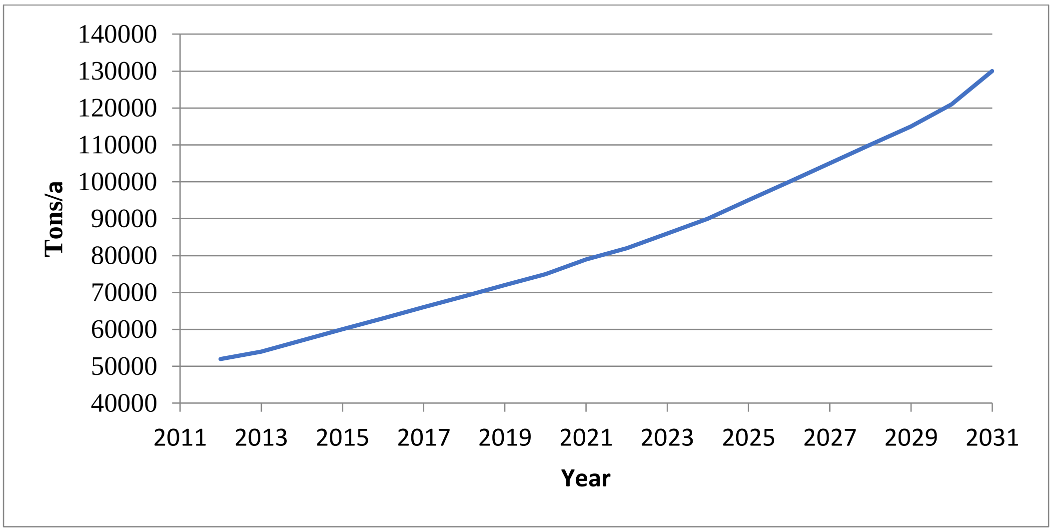


Figure 1.3: Forecast of used tyres in Kenya

1.4 Material Composition of Tyre

The materials used to make tyres are divided into two categories, Cords that are used to make the ply and the elastomer which encases the cords(Association, 2020)

Figure 1.4: Tyre profile

1.4.1 Cords

It contains steel, natural fibers such as cotton or silk or synthetic fibers such as nylon.

1.4.2 Elastomer

This is the part of the tyre that forms treads and it encloses the cords. It is made of various composites of rubber and the most common is styrene-butadiene copolymer. However, there are other compositions which include Silica and Carbon black.

1.5 Problem Statement/Gap

The cost of fossil fuels has been escalating thus rendering their use not only expensive but also unsustainable. Furthermore, the coal and petroleum deposits in the entire world will be exhausted due to continuous exploitations. According to some researches, the natural gas, oil and coal will be exhausted in 54, 53 and 110 years respectively (Singh, 2015). This makes it necessary to come up with an alternative source of energy.

Waste to energy conversion is one of the alternative sources of fuel. Used tyres pose a threat to environment especially in developing countries since the current methods of disposal pollutes environment. Liquid from used tyres has the potential of being used as a source of fuel to replace petroleum diesel (Mamun et al., 2015). It has been analysed by Ning (Ning et al., 2013), that waste to energy technologies through pyrolysis technique is a cheaper option and produces alternative source of fuel. The cost of producing Tyre pyrolysis oil according to Ning is USD 399.772 per cubic meter (Ning et al., 2013) while the current cost of petroleum diesel in Kenya is estimated at USD 1432 per cubic meter. It is not only a cheaper alternative but also sustainable due to availability of used tyres and appropriate conversion technologies. However, despite this cheaper alternative of used tyres, there is a gap of inefficient production of TPO in the conversion technologies. This has resulted in lower production of liquid fuel from used tyres and thus reducing the interests' in "Waste to Energy" technologies. It is important to know the combination of optimal values of variables that can produce optimal yields. Without the optimal values, there will be production of less liquid fuel yields which renders the whole process uneconomical. When the process is not optimized, it can lead to production of higher solid and gaseous products and reduced liquid products. The main objective of this study is to come up with optimal conditions for liquid fuel production from Microwave pyrolysis of used tyres using experimental and statistical approaches and compare with that of thermal pyrolysis.

1.6 Limitations of the Study

a) Laboratory prototype may vary with the large scale production in terms of power consumption, reaction time, capital cost and maintenance.

b) The particle size was limited to 25mm^2 being the smallest particle size but even smaller particle sizes can be achieved. On the other hand, sample size of the feedstock was limited to 100grams.

c) The feedstock (used tyres) was limited to one specific type of tyre which is Triangle 1000 R 20 10.00X20 Truck Tyre.

1.7 Objectives

1.7.1 General Objective

1. To carry out comparative study of pyrolysis of used tyres using microwave and thermal methods.

1.7.2 Specific Objectives

The specific objectives are:

1. To study the effects of operating variables on yield (solid, liquid, gas) for Microwave and Thermal pyrolysis.
2. To optimize the tyre pyrolysis oil yields of Microwave and Thermal pyrolysis using Central Composite Design.
3. To characterize tyre pyrolysis oil from Microwave and Thermal pyrolysis for fuel.

1.8 Justification

The increased use of automobile usage especially in areas with higher population has led to environmental problems one of them being disposal of waste tyres (Lam & Chase, 2012). There are several wastes that are being generated every year some are effectively collected and recovered to be used as a source of energy or chemical feedstock. However, some of the wastes such as used tyres are discarded or burned in ways that can easily pollute the environment. Improper disposal of tyres can result in environmental hazards. This is because tyres have undesirable species which includes soot, heavy metals and Polycyclic Aromatic Hydrocarbons (PAH) (Bekhiti et al., 2014). The associated environmental hazards from tyres are due to the fact that they are non-biodegradable and therefore last for many years in the environment causing pollution. Because of the difficulties that are associated with the contaminants present in the tyres, most of them are disposed by land filling.

Land filling has no recovery of the potential value of the tyres. Researches have been done on the possible values of Tyre pyrolysis oil from thermal pyrolysis and it has been found out that the liquid products have several uses (Murugan et al., 2008). Pyrolysis is the heating of a feedstock in absence of oxygen to produce liquid, solid and gaseous products (Biogreen, 2018). Pyrolysis is a technique that has been developed to be used as an alternative to convert the wastes to potential energy source. The process has the potential to recover the energy and chemical value of the used tyres since it generates potentially valuable products from the pyrolysis process.

The energy content of the oil and gaseous products can be analysed and the char produced has the potential of being used as a substitute for carbon black. The oil and gaseous products are of great interest because of their potential of being used directly as a heavy fuel. Besides, it can be used as a blend for diesel at the right proportions and be used either for cooking or in internal combustion engines.

Microwave pyrolysis is a relatively new process which is not only efficient but also characterized by minimum emissions released to the atmosphere (Kalla & R., 2016). It is characterized by a unique heating technique where the heating effect arises from the interaction of electromagnetic waves together with dipoles within the complex structure of the material. This mode of heating ensures that the heat is generated within the material instead of external sources. Such heating ensures that there is a more efficient heating process compared to conventional surface heating because there is even distribution of heat and easier control of the heating process (Kalla & R., 2016). Finally, higher temperatures and heating rates can be achieved through microwave heating. There is a need to optimise some of the parameters such as microwave power, particle size and reaction time in order to achieve the best output from microwave pyrolysis process. Optimization of the process can pave way to generation of high- value products.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

The literature review under this study covers the concept of microwave pyrolysis, the heating mechanisms of microwave heating and comparison of microwave pyrolysis to conventional heating. Besides, the literature review analyses the past research that has been done on both microwave and thermal pyrolysis in order to identify the existing gaps. The proximate and ultimate analysis of used tyres is also analyzed under literature review based on past research. Finally, a review of central composite design is explained in order to justify why central composite design was used for process optimization.

2.2 Microwave Heating Technology

2.2.1 Microwaves

Microwaves lies between radio frequencies and infrared in electromagnetic spectrum(CURNUTTE, 1980). The wavelengths of the microwaves are between 1 mm and 1 m and the corresponding frequencies are between 300 GHz and 300 MHz respectively (Vollmer, 2004). Electrical energy is the source of microwave energy with a conversion efficiency of 50% to 80%. Microwaves generates molecular disturbance either by rotation of dipolar species or migration of the ionic species (Haque, 1999). Materials that can absorb microwaves are termed as dielectrics or absorbers and hence microwave heating is also called dielectric heating.

2.2.2 Microwave pyrolysis

Microwave pyrolysis is the heating of feedstock at elevated temperatures using microwave irradiation in absence of oxygen (Zhang et al., 2017). Microwave pyrolysis has drawn interests in waste to energy conversions because of its numerous advantages compared to the conventional heating methods (Du et al., 2011). For conventional heating methods, the heat is transferred through convection mechanisms where it is transferred from high temperature gas to fuel particle surface. Furthermore, the useful heat is then transferred from the outside surface to the inner core through conduction. Because of the poor thermal conduction of tyres, a temperature gradient is formed from outside to the inside of the feedstock. On the other hand, in microwave heating, the microwaves penetrate the feedstock used and the energy is transferred into the inside of the feedstock particles. The heat energy constantly accumulates in the feedstock and transferred outwards as illustrated in figure 2.1 (Du et al., 2011).

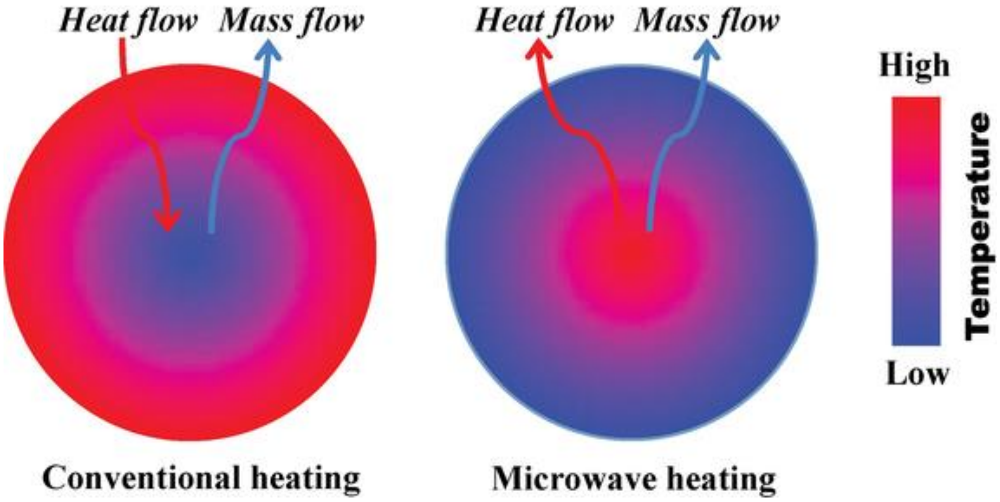


Figure 2.1: Temperature distribution, heat transfer, and mass transfer in the conventional heating and microwave heating

The differences between microwave heating and conventional heating methods are summarized in Table 2.1

Table 2.1: Comparison between microwave-assisted heating and conventional electrical heating

Microwave-assisted heating	Conventional electrical heating
Conversion of energy	Transfer of energy
In-core volumetric and uniform heating at molecular level	Superficial heating through conduction, convection, and radiation
Hot spot	No hot spot
Rapid and efficient	Slow, inefficient, limited
Higher electricity conversion efficiency	Lower electricity conversion efficiency
Selective	Non selective
Dependent on material's properties	Less dependent
Controlled heating	Heating Less controllable
Flexible Process	Less flexible Process
Equipment portable	Less portable
Lower contaminants	Higher contaminants
Lower thermal inertia and faster response	Higher thermal inertia and slower response

2.2.3 Microwave power verses Microwave Temperature

Microwave temperature is determined by microwave power. Microwave power is usually expressed as % of the total power rating of the microwave. The lower the power rating the lower the pyrolysis temperature and vice versa (Tian et al., 2011). Figure 2.2 indicates the temperature profile at various power levels and also effect of time on the pyrolysis process.

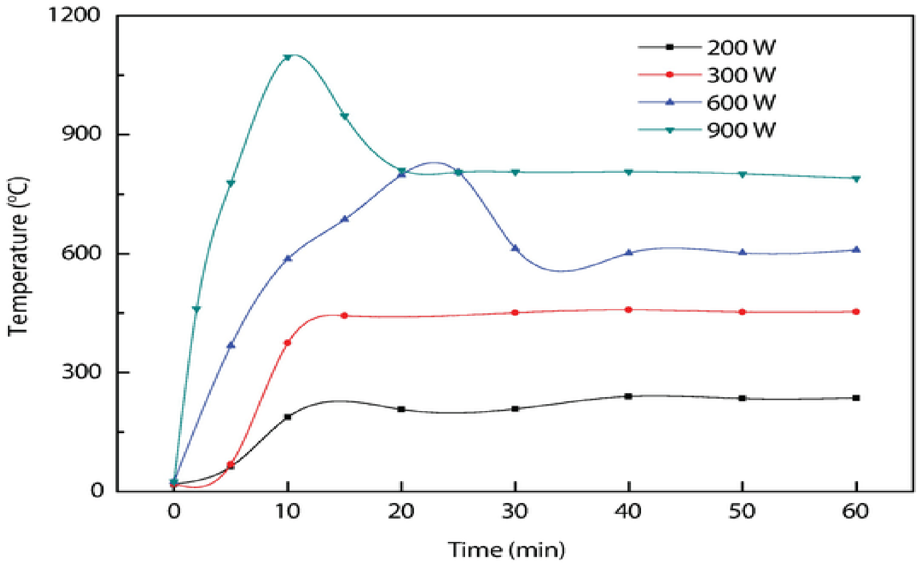


Figure 2.2: Temperature profiles of corn Stover during microwave-assisted pyrolysis at different microwave powers

From the figure, the higher the microwave power, the higher the pyrolysis temperature and the time taken to complete the pyrolysis process is between 10 minutes to about 20 minutes for a 50g corn sample. Therefore, Microwave power is a function of reaction temperature.

2.3 Fundamentals of Microwave Heating

The use of microwave heating technology in thermal treatment of feedstock has increased from the mid-nineties. The heating technique reduces the processing time and energy consumption used to convert the feedstock to useful products. Furthermore, it has a unique internal heating phenomenon compared to the conventional pyrolysis techniques (Kumar et al., 2016).

According to the interaction of microwave irradiation (N, 2010) (electric component of microwave field) with materials, there are three ways in which a material may be categorized:

1) Insulator or microwave-transparent material where microwaves pass through without any losses (e.g. quartz, teflon, etc.),
2) Conductor where the microwaves cannot penetrate and are reflected (e.g.metals), and
3) Absorber where the microwaves can be absorbed by the material (e.g. Water, oils, etc.). Microwave dielectrics are known as a material which absorbs microwave irradiation; thus, microwave heating is called dielectric heating.

2.3.1 Advantages of Microwave-Assisted Pyrolysis Technology over Conventional Heating

Compared to other pyrolysis techniques, microwave pyrolysis of used tyres has the following advantages (Haque, 1999):

1. The heating is fast, uniform and this makes heating of large size and non-homogeneous feedstock possible.
2. The products from microwave pyrolysis are clean compared to other heating techniques. This is because it does not require using feedstock powder and agitated fluidization.
3. The energy content of products of pyrolysis from microwave pyrolysis is higher compared to products from other pyrolysis processes. This is because microwave pyrolysis is free from contaminants and diluting agents such as atmospheric moisture.

2.4 Past Researches on Microwave Pyrolysis

There are some researches that have been done related to pyrolysis of tyres but there are still some areas that needs more research so as to utilize fully the energy potential of such products.

First, there is a research that has been conducted to find out the calorific value of pyro-oil which is a product of microwave pyrolysis (Mamun et al., 2015). The main objective of the research was to find out the calorific value of the oil products and to compare it with that of fossil fuels. In this research, a 195/65 R15 scrap tyre was utilized in the preparation of smaller chips. The scrap tyre was first cut into smaller pieces (chips) before being washed to remove impurities and dust. It was then dried in air to ensure that the required moisture content was achieved. They were finally ready for the pyrolysis process. Weighing was done before pyrolysis and a fixed bed system was used in this study. For the two different trials done, varying amount of tyre chips were used. Finally, the higher calorific value of the chips was determined using a bomb calorimeter.

A bomb calorimeter was used to determine the higher calorific value of the pyro-oil. A known mass of the pyro-oil produced which was less than one gram was used and after getting the temperature rise, the higher calorific value was obtained. The higher calorific value of the pyro-oil was found to be 53 MJ/kg. This value was found to be higher compared to fossil fuels. The Table 2.2 below compares it with other fossil fuels:

Table 2.2: Product yield from Microwave Pyrolysis

SER	EXPERIMENT NO.	1	2
1	Wight of tyre chips(gram)	162	103
2	Microwave Power (Watts)	900	900
3	Pyro-Oil Produced (gram)	48.87	38.7
4	Char Produced (gram)	85	49
5	Gas Produced (gram)	28.13	15.3

Table 2.3: Comparison of the Calorific Value of Different Fuels with That of Pyro-Oil

SER	Fuel Name	Calorific Value (MJ/kg)
1	Diesel	44.8
2	Gasoline	47.3
3	Methane	55.5
4	Pyro-Oil	53.8

It was then concluded that pyro-oil which is a product of microwave pyrolysis of used tires has a higher calorific value compared to other common fossil fuels. It is almost the same as that of methane.

Gap

The research does not study the effects of varying some parameters such as temperature, size of the feedstock (tyre chips), microwave power, and reaction time on the yield.

Another research was done to evaluate the performance and the emission characteristics of a single cylinder engine when diesel fuel blended with tyre pyrolysis oil (TPO) (Murugan et al., 2008). Analysis of combustion parameters which include cylinder peak pressure, heat release rate and the maximum rate of pressure rise were done. The TPO was obtained from waste tyres by subjecting them to vacuum pyrolysis. The blending proposition of TPO that were analysed was 10%, 30% and 50% mixture of TPO and diesel fuel.

The outcome of the study revealed that the brake thermal efficiency of the internal combustion engine that was fuelled by TPO and diesel fuel blend increased with increase in blend concentration. It was found that it was higher compared to pure diesel fuel.

Emissions were also analysed and it was found that for a blend of diesel and TPO, the HCs, CO and NO_x were higher at higher loads. This is because of longer ignition delay and aromatic content. There was also an increase in the cylinder peak pressure and from 71.4 bars to about 73.8 bars. The ignition delays were found to be longer as compared to the use of pure diesel fuel.

Gap

The research does not consider optimization of the microwave pyrolysis process. Furthermore, the study does not study the effects of varying some parameters such as temperature, size of the feedstock (Tyre chips), Microwave power, and reaction time on the yield.

In another research conducted in 2018, (Idris et al., 2019) , a microwave induced technique was used to conduct pyrolysis on used automobile tyres with the aid of activated carbon. The effect of temperature was studied on the yield, chemical composition, hydrocarbon fractions, fuel properties and energy yield. The activated carbon was used as an absorber so as to elevate the temperature to suitable values for production of the products of pyrolysis.

It was found out from the experiment that the optimal pyrolysis temperature was 500^{o}C and produces the highest yield of pyro-oil of 38.12 wt.%, calorific value of 42.39 MJkg^{-1} and energy yield of 40.55 wt.%. Furthermore, detailed analysis revealed that the products of pyrolysis has larger number of aromatic hydrocarbons and limonene (14.29 wt.%).

Gap

The research does not study the effects of varying some parameters such as particle size, microwave power and reaction time on yield.

Another research was conducted in 2017 by Alex Lu Chia Yang to study microwave pyrolysis of used tyre with and without activated carbon as catalyst and the oil performance in diesel engine (Yang & Ani, 2016). In this experiment, the scrap tyre was heated at temperatures between 400 and 600 degrees Celsius to produce liquid fuel. The experiments were carried out with and without activated carbon as microwave absorbent. The main objective was to study the effects of temperatures and activated carbon on the yields. Furthermore, the pyro-oil was characterized for the chemical composition, compound functional group and the calorific value. The set up was as shown in the figure 2.3 below:

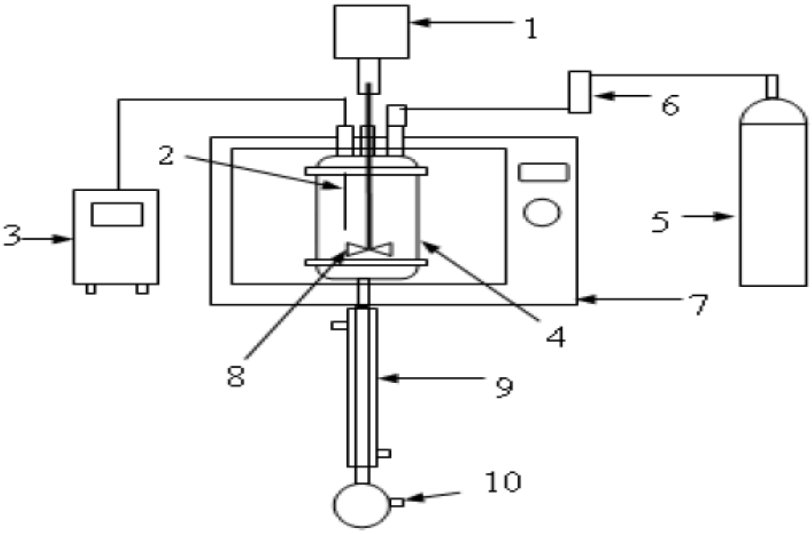


Figure 2.3: Setup of Microwave assisted pyrolysis system

Image source (Yang & Ani, 2016) (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

It was found out that the optimal temperature for the pyrolysis process was 500 degrees Celsius with the highest yield of pyrolytic oil at 54.39 wt.%. The high calorific value of the oil was in the range of 42-43 MJ/kg.

Gap

The research does not study the effects of varying some parameters such as particle size, microwave power and reaction time on yield. Besides, no optimization of yields was done on this particular research.

A research to study the effects of microwave power on microwave pyrolysis process was done by Song, (Song et al., 2017).The tyre powder was treated under different power levels and expressed as Specific Microwave Power (SMP) which is powers per 1 gram sample. The SMPs chosen were 9 W/g, 15W/g and 24 W/g. The microwave oven used was rated with an output power of 900W and SMPs of 9 W/g, 15W/g and 24 W/g corresponds to 270W (30%), 450W (50%) and 720W (80%) respectively. It was found that the highest yield of liquid fuel 45 (wt. %) was obtained at a SMP of 15W/g

(50%) while the highest gaseous yield of 18.5 (%wt.) was obtained at a SMP of 24 W/g (80%).

Gap

This research does not study the effects of other variables that affect the process and no process optimization was done.

2.5 Thermal Pyrolysis

Thermal pyrolysis is where the feedstock is heated using a furnace with absence of oxygen to produce liquid and gaseous products and solid residue. The thermal cracking of the long-chained hydrocarbons is achieved by high temperatures of more than 600°C. A reactor is connected to a condensing unit where the liquid products condense.

There is a research that was done in 2017 by Julius I. Osayi to study about the thermal pyrolytic conversion of used tyres to produce liquid fuel (Osayi et al., 2018). In his research, he focused on characterization and effects of the operating parameters on the yield. In an experimental set up, a chemical vapor deposition reactor was used for the pyrolysis process as shown in figure 2.4:

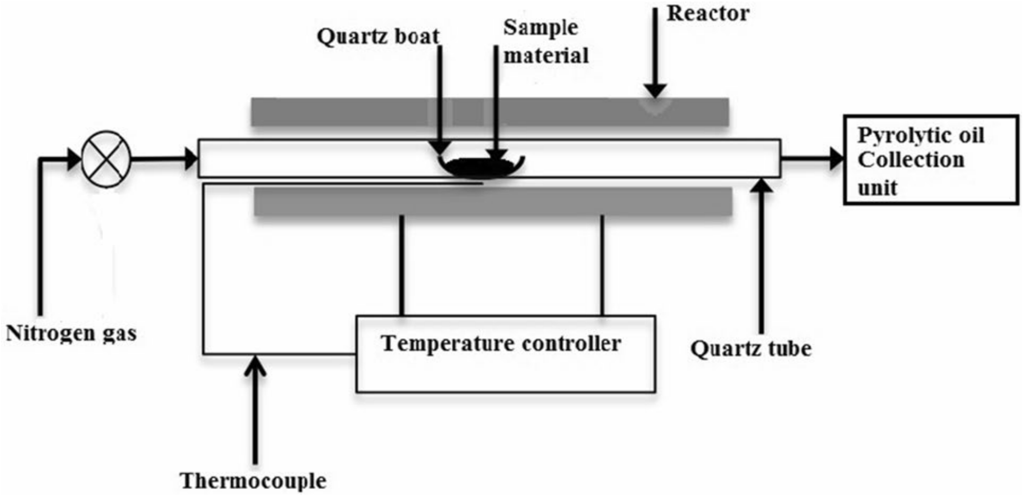


Figure 2.4: Schematic of the experimental setup for the Thermal pyrolysis of used Tyres

Image source: (Osayi et al., 2018)

In this research, the operating variables that were optimized were the operating temperature, heating rate and the particle size. The optimum yields (34.04%) for the liquid products were obtained at a temperature of 600 ^oC, heating rate of 15 ^oC/min and feed particle size of 6mm. furthermore, characterization of the liquid products were done using Fourier Transform infrared (FTIR), Nuclear Magnetic Resonance (NMR) and Gas chromatography. The results that were obtained indicated that the oil is a complex mixture which has aromatic and aliphatic compounds.

According to research by Durak H, (Durak et al., 2019) a cumin seed cake was transformed to liquid and solid products with and without catalyst. The pyrolysis was performed at temperatures of 300, 400 and 500^oC. The aim was to investigate the liquid yield and the effects of catalyst on the process. The highest liquid yield was 30% in presence of Al_2O_3 catalyst and less than 30% without catalyst. This clearly indicates that catalyst improves the yield of the liquid products. The High Heating Values (HHV) for the pyrolysis oil was found to be between 32.44 and 36.19 Mj/kg.

Another research was done to investigate the effects of temperature and catalyst on the production of bio-oil and bio-char from avocado seeds by Durak & Aysu (Durak & Aysu, 2015). The reaction was performed in a fixed bed reactor using KOH, Al2O3 as a catalyst and another without catalyst. The temperature range was between 400^oC to 600^oC and the heating rate was 50^oC/min. Temperature and catalysts were determined to be the main factors that affect the thermal cracking of avocado seeds to liquid and gaseous products. In this research, the highest liquid fuel yield of about 37.5% was obtained using using 10 % KOH as a catalyst at pyrolysis temperature of 600 °C and heating rate of 50 °C/min.

Aysu & Durak (Aysu & Durak, 2015) did a research in 2015 to study the effects of temperature and catalyst on thermal pyrolysis of liquorice (Glycyrrhiza glabra L.). Experiment was performed with different catalysts (ZnO, $FeCl_3$, K_2CO_3, Al_2O_3, $Na_2B_4O_7.10H_2O$) and without a catalyst at three different reaction temperatures (350, 450, 550 °C). The experiment was performed in a tubular fixed bed reactor with a constant heating rate of 40 °C/min. The highest liquid yield of 34.35% was at a temperature of 550°C with $Na_2B_4O_7.10H_2O$ catalyst.

2.6 Proximate and Ultimate Analysis of Used Tyre

There is a research done to investigate the proximate and ultimate analysis of used tyre (Osayi et al., 2018). In this research, the calorific value, ultimate and proximate analysis was carried out on used tyre so as to know the physiochemical properties of the tyre sample. The gross calorific value of the tyre sample was obtained using DryCal modular 89 calorimeter. Proximate analysis was done using Perkin Elmer (Model STA 6000 Shelton USA) simultaneous analyser. The elemental composition of the sample was conducted using Thermo Scientific Flash 2000 elemental analyser. The results of proximate the analysis is as shown in Table 2.4 while that of ultimate analysis is as shown in table 2.5:

Table 2.4: Proximate analysis of Used Tyre

Proximate Analysis		
SER	**Component**	**% Composition**
1	Moisture Content	0.5
2	Ash	16
3	Fixed Carbon	27.1
4	Volatile Content	56.4

Table 2.5: Ultimate Analysis of Used Tyre

Ultimate Analysis		
SER	**Component**	**% Composition**
1	Carbon (C)	85
2	Hydrogen (H)	5.5
3	Nitrogen (N)	0.21
4	Sulphur (S)	1.2
5	Oxygen (O)	8.09

The Gross calorific value of used tyre was found to be 31.22 MJ/kg. This is greater than that of lignite coal and wood with GCVs of 15.0 MJ/kg and 21.7 MJ/kg respectively.

2.6.1 Physiochemical Properties and GC Analysis of Tyre Pyrolysis Oil

There are some researches that have been done to analyze the properties of Tyre pyrolysis oil and determine its suitability to be used as a fuel. According to Hariram, (Hariram et al., 2017), the tyre pyrolysis oil has almost similar properties as that of commercial petroleum diesel. Table 2.6 summarizes the properties and the methods used.

Table 2.6: Physiochemical Properties of Tyre Pyrolysis oil and its comparison with commercial diesel.

Properties	Units	Commercial Diesel	WTPO	WPPO	Testing Methods
Gross calorific value	MJ/kg	43.9	42.7	39.45	IS:1448 P:6
Kinematic viscosity, @ 40°C	cSt	2.1	3.4	2.2	IS:1448 P:25
Flash point	°C	52	46	44	IS:1448 P:20
Fire point	°C	57	53	47	IS:1448 P:20
Density @ 15°C	kg/m^3	832	936	837.5	IS:1448 P:16
Cetane index	-	46.4	55	68	IS:1448 P:9
Carbon residue	%	0.37	2.21	72.79	IS:1448 P:122
Sulphur content	%	0.047	0.99	0.034	IS:1448 P:33
Ash content	%	0.01	0.33	0.00027	IS:1448 P:126

From table 2.6, it can be concluded that the Tyre Pyrolysis Oil (TPO) has properties close to that of commercial diesel as reported by Hariram. The Gross calorific value of TPO is at 48.99 MJ/kg while that obtained by Hariram (Hariram et al., 2017) for WTPO was at 42.7MJ/kg. The Gross calorific value of commercial diesel is between 42-46 MJ/kg according to World Nuclear Association (World Nuclear Association, 2016) and 43.9 MJ/kg according to Hariram (Hariram et al., 2017). This implies that the TPO has calorific value higher than that of commercial diesel. Other parameters such as flash point, fire point, density and viscosity are close to that of petroleum diesel. Upon further purification, TPO can be used directly in internal combustion engines instead of diesel. The calorific value of Heavy Fuel Oil (HFO) is at 41MJ/kg(Kenya, 2020). The required standards for HFO sold in Kenya (Kenya, 2020) is that it must meet the following critical parameters: Maximum Kinematic viscosity @ 500C of 180 cst, sulphur content of 2.3% maximum and a minimum net calorific value of 41MJ/kg. Therefore, TPO qualifies to be used directly as an HFO.

GC/MS was performed on TPO to identify the components by FLaresgoiti (FLaresgoiti et al., 2004). It was found out that the TPO is a complex mixture of long chained hydrocarbons. It has complex components ranging from C_6-C_{24}. It was found out that TPO comprises of (53.4–74.8%) aromatics, nitrogenated components at (2.47–3.5%). Other components present include hydrocarbons such as Limonene, Toluene, benzene, and xylene.

2.7 Process Optimization

2.7.1 Response surface methodology (RSM)

This is a process that comprises of mathematical and statistical techniques that are used to generate and analyze models. The main concept of RSM is to determine the optimal response of processes (Farid, n.d.), In a manufacturing set up, the processes can be improved by incorporating the Design of experiments (DoE) in the early stages of the process development cycle (Guo & Mettas, 2011). Central Composite Design (CCD) was developed in 1951 by Box and Wilson and is one of the methods used in the optimization of the experimental conditions. It combines 2k full factorial or the 2k-p fractional experimental runs axial points (2k star) experimental runs on each of the k axis at a given distant from the central point. There is also at least one center point experimental run.

There are three types of central composite designs (Ait-Amir, 2015) as shown in Table 2.7.

Table 2.7: Types of CCD

Type of design	Description
Circumscribed (CCC)	The star points are outside the initial experimental domain (distance α). This design requires five levels per factor.
Face-centered (CCF)	α=±1, the star points are located on the faces of the experimental domain. This design requires three levels per factor.
Inscribed (CCI)	α=±1, this design is used when it is not possible to leave the experimental domain. The CCC design is then reduced to fit within this domain. This plan requires five levels per factor.

2.8 Summary of Literature Review

Used tyres can be used as a feedstock from pyrolysis of used tyres. Microwave pyrolysis is a latest technology which is efficient and saves energy. The published literature clearly indicates that the tyre pyrolysis oil from used tyres has calorific value slightly higher than that of diesel. Most of the published literature have analyzed the effects of variables on both thermal and microwave pyrolysis. Optimization of microwave power, particle size and reaction time has not been analyzed for microwave pyrolysis. Optimization of variables temperature, reaction time and particle size has not been analyzed in thermal pyrolysis. Limited research has been done to compare microwave pyrolysis with conventional methods under the same conditions. This research optimizes microwave pyrolysis and compares it with conventional methods under similar conditions.

CHAPTER THREE

MATERIALS AND METHODOLOGY

3.1 Introduction

This chapter includes the research station, equipment used, Feedstock and method used and the experimental and statistical procedures used. Tyre (Triangle 1000 R 20 10.00X20 Truck Tyre) was shredded into various sizes using a shredder and used as feedstock. Microwave pyrolysis was used to heat the feedstock under various conditions and the outcome analyzed. The variables considered on microwave pyrolysis were: particle sizes, reaction time and microwave power. Thermal pyrolysis was also done for comparison purposes to microwave pyrolysis. Optimization of the microwave pyrolysis process was done using Central Composite Rotatable Design (CCRD) and Response Surface Methodology (RSM) were done using Design Expert-13 software, trial version.

3.2 Research Station

The experiments of this research were carried out at the automotive laboratory, department of Mechanical, Production and Energy Engineering, Moi University, Eldoret- Kenya. Physiochemical Properties of liquid fuel and Gas Chromatography analysis was done at Lab Works East African Limited-Nairobi.

3.3 Research Equipment

The following equipment was used in this research:

a. Cutting Knife

A sharp knife was used to cut the feedstock (used tyres) into required sizes.

b. Microwave

Domestic microwave oven (SAMSUNG GE0103MB1) with an output power of 900W and 2450 MHZ was used to carry out the pyrolysis process. The microwave is as shown in plate 3.1

Plate 3.1: SAMSUNG GE0103MB1 Microwave oven

c. Liebig condenser

A 300mm Liebig condenser was used to condense the vapour from the reactor. The liebig condenser is connected to circulating water at an average temperature of 10^{o}C. A temperature gradient exists between the circulating cold water and the vapour from the reactor. As a result, heat transfer occurs and the temperature of the hot vapour is reduced resulting in condensation. The condensed liquid product is collected. The set-up is as illustrated in plate 3.2

Plate 3.2: 300mm Liebig condenser

d. 500mL round bottomed flask with a sprout

A borosilicate 500 ml round bottomed flask with a sprout was utilized as a reactor inside the microwave cavity. The round bottomed flask is as shown in plate 3.3

Plate 3.3: 500 mL Borosilicate Round Bottomed Flask

e. Laboratory Stand and Clamp

This was used to hold the liebig condenser in the required position once connected to the sprout of the reactor (round bottomed flask)

f. Rubber Corks

Two rubber corks were used in the experiment. The first one was used to seal the opening of the round bottomed flask to enable the fumes to escape only through the sprout. The other rubber cork was cut to connect the liebig condenser to the reactor as shown in plate 3.4.

Plate 3.4: Rubber corks

g. Digital Weighing Scale

A digital weighing scale (SCALTEC) with accuracy of 0.1 g was used in the research. It was mainly used to weigh the feedstock, liquid fuel and the solid residue. The weighing scale used is as shown in plate 3.5.

Plate 3.5: SCALTEC Digital Weighing Scale

h. Sample Bottles

50 ml plastic sample bottles were used to collect the liquid products of pyrolysis.

i. Laboratory Glassware

Various glassware used in experimental studies included test tubes and round bottomed flasks.

j. Furnace Clay

China Furnace clay was used to fabricate the thermal pyrolysis furnace. China clay was chosen because of its ability to withstand higher temperatures and besides, they are good insulators against heat loss.

k. Temperature Controller and Heating Elements

The heating element chosen was that made of tungsten as it can provide higher temperatures due to higher resistance. Temperature controller (Range 0-1300°C) was meant to control the heating temperature for thermal pyrolysis.

l. Thermocouple

RTD thermocouple probe was chosen with a range of up to 1300°C

3.4 Feedstock Preparation

A used Tyre (Triangle 1000 R 20 10.00X20 Truck Tyre) was used for the preparation of feedstock (tyre chips). The selection of used tyre was based on availability of that specific tyre and it was the most common type of tyre that could be easily found in Kenyan markets. Therefore, this research is limited to this specific type of tyre. It was shredded by a knife to achieve the required size unit of measure being the cross-sectional area. The sizes were estimated using an ordinary meter rule since smaller quantities were required. All sizes had a uniform thickness of 2mm. The feedstock was then screened to remove impurities and dust that were on the surface of the tyre material. They were then dried in the air to remove the moisture that could interfere with the pyrolysis process. To avoid the presence of water in the liquid fuel, low power microwave pre-treatment is recommended. Pre-heating to about 100°C vaporizes all the moisture present in the tyre chips. The feedstock is then ready for pyrolysis. 100 g samples of raw materials were weighed and clearly labeled. Plate 3.6 illustrates the preliminary stages of feedstock preparation.

Plate 3.6: Feedstock Preparation

3.5 Microwave Pyrolysis

An experimental investigation was done on the pyrolysis of used tyre using a microwave oven (SAMSUNG GE0103MB1) with an output power of 900W and 2450 MHZ. Modification was done on the microwave so as to suit the pyrolysis process. A 50mm hole was drilled at the ceiling of the microwave so as to hold the neck of the round bottomed flask. Besides, the hole was meant to ensure that the mouth of the round bottomed flask was out of the microwave cavity then sealed using a wooden cork. The 500 ml quartz glass round bottomed flask was utilized as a reactor. The microwave selected had 10 power levels and a timer and these features make it easier to monitor the effects of microwave power and the reaction time. The set-up is as shown in the figure 3.1 below:

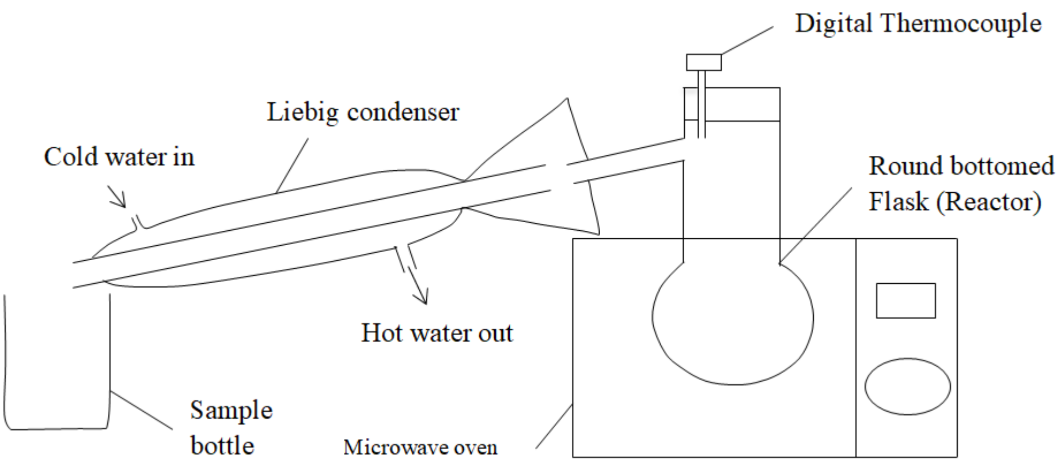


Figure 3.1: Microwave Pyrolysis set up

3.6 Thermal Pyrolysis

A heating furnace was fabricated using furnace clay. Furnace clay was chosen because of its ability to retain heat and to withstand higher heating temperatures. The furnace clays were arranged in such a way that they form a cavity similar to that of microwave. Two electrical heating coils each rated 300W were inserted on the furnace clays on the inner side of the heating cavity. The coils were then connected to a temperature controller to monitor the temperature of the heating cavity. A round bottomed flask connected to a liebig condenser was used as a reactor as shown in the plate 3.7. The feedstock is fed into the reactor and when the power is switched on, the feedstock is heated to the set temperatures. The fumes are formed at higher temperatures and they flow out of the reactor through the condenser where they are condensed and the liquid products are collected at the sample bottle.

Plate 3.7: Thermal Pyrolysis set up

3.7 Mass of the Products of Pyrolysis

This was performed in accordance with the law of conservation of mass using a weighing scale. The initial mass of the feedstock was recorded as $\mathbf{M_1}$. The mass of the liquid products was recorded as $\mathbf{M_2}$. The mass of the solid residue recorded as $\mathbf{M_3}$. Therefore, mass of gaseous products will be $\mathbf{\{M_1\text{-}(M_2+M_3)\}}$.

3.8 Effects of Operating Variables on Yield for Microwave Pyrolysis

This research mainly focused on particle size, microwave power and reaction time as independent variables for microwave pyrolysis. Reaction temperature, reaction time and particle size were the independent variables for thermal pyrolysis. The effects of each variable on the yield were determined by carrying out experiment.

3.8.1 Particle Size

Tyre (Triangle 1000 R 20 10.00X20 Truck Tyre) was shredded into various sizes using a shredder. The sizes used in this experiment were cubic with uniform thickness of 2mm. The cross-sectional area of various sizes was varied for the purpose of investigating the effect of particle size of the feedstock on the yield of microwave pyrolysis process. The cross-sectional areas considered were: 25, 50, 60, 100, 125 and 200mm^2. A Microwave power of 100% and reaction time of 20 minutes was used in all the sizes. 100 grams of each size were used and after 20 minutes, the microwave automatically stops and the liquid fuel collected is weighed. The solid residue was also weighed and the mass of the gaseous products are obtained using the following relation:

Mass of the gaseous products = 100g – mass of liquid fuel – Mass of solid residue

The results were recorded in form of a table as shown in Table 3.1:

Table 3.1: Effects of Particle Size on Yield for Microwave Pyrolysis

SER.	Particle Size (mm^2)	Oil Yield (% wt)	Gas Yield (% wt)	Volatile Yield (% wt)
1	25			
2	50			
3	100			
4	200			

3.8.2 Microwave power

The selected microwave (SAMSUNG GE0103MB1) with an output power of 900W and 2450 MHZ had 10 power levels ranging from 10% (90W) to 100% (900W). The other variables were held constant and microwave power varied. The particle size used was 25mm^2 and a reaction time of 25 minutes. For this experiment, the microwave power selected were 20%, 30%, 40%, 50%, 60%, 80% and 100%. The results were then tabulated in form of a table as shown in Table 3.2:

Table 3.2: Effects of Microwave Power on Microwave Pyrolysis Yield

SER.	Microwave Power (%)	Oil Yield (% wt.)	Gas Yield (%wt.)	Volatile Yield (% wt.)
1	20			
2	30			
3	40			
4	50			
5	60			
6	80			
7	100			

3.8.3 Reaction Time

The selected microwave has an automated timing system and stops when the set time elapses. The microwave power was set at 100% and particle size used was 25mm^2. The first step was to determine the time taken for the condensable products to condense at full power. The products were then monitored after every 5 minutes. Let the time taken for the first drop of liquid fuel be X mins. Therefore, the other time intervals considered are: (X+5) mins, (X+10) mins, (X+15) Mins, (X+20) mins and (X+25) mins the results were then tabulated as shown in the table 3.3:

X = Time taken for the first drop of liquid fuel to drop at full power in minutes.

Table 3.3: Effects of Reaction Time on Microwave Pyrolysis of used tires.

SER.	Reaction Time(min)	Oil yield (% wt.)	Gas Yield (% wt.)	Volatile yield (% wt.)
1	(X+5)			
2	(X+10)			
3	(X+15)			
4	(X+20)			
5	(X+25)			

3.8.4 Optimization of the liquid fuel yields of microwave Pyrolysis using Central Composite Design

This research was mainly focused on the liquid fuel yield. The three variables particle size, microwave power and reaction time were optimized using the CCD. The central composite design was performed as per the method of Liu (Liu et al., 2008). The central values, the step sizes and the range of variables were as follows: central microwave power of 50%, step of 10% and range of 40%-60%; central reaction time of 17.5 mins, step of 4.5 mins and range of 13 mins to 22 mins; and central particle size of 112.5 mm^2, step of 52.5 mm^2, and range of 60 mm^2 – 165mm^2 as shown in Table 3.4 below:

Table 3.4: The Central Composite Design

Factors	Units	-alpha	Low	Center Point	High	+alpha
Microwave power	%	30	40	50	60	70
Reaction time	Mins	10	13	17.5	22	25
Particle size	mm^2	25	60	112.5	165	200

The number of experimental obtained at each number of factors is given by the formula, $N=2^n +2xn +n_c$ where N is the number of Runs, n is the number of factors and n_c is the number of centre points. Where n is three, for a single replication, 15 experimental runs are obtained. However, 3 replicates were conducted for axial, centre and factorial points making the total number of runs to 45 as illustrated in table 3.5. For a full factorial rotatable design, $\alpha = [2^k]^{1/4} = [2^3]^{1/4} = 1.682$ (Antony, 2014). 0_1, 0_2 and 0_3 are the centre points while - α (1.682) and + α (1.682) are the axial points. To get the value for axial point, the following equation is applied:

Axial point = mean of both the upper and lower level ± α (range between the upper and lower level divided by 2). Therefore, axial point = X ± α (Range/2); Design expert 13 Trial Version was used to develop the runs for optimization (factorial, axial and centre point runs) for 3 replications for all points and translated to 45 experimental runs as shown in Table 3.5 below:

Table 3.5: Optimization table showing factorial, axial and centre point runs

Std	Run	X1:Microwave Power (%)	X2:Reaction Time (Mins)	X3:Particle Size (mm^2)	Liquid Fuel Yield (% wt.)
12	1	60	22	60	
39	2	50	17.5	25	
24	3	60	22	165	
5	4	60	13	60	
26	5	30	17.5	112.5	
30	6	70	17.5	112.5	
42	7	50	17.5	200	
15	8	40	13	165	
8	9	40	22	60	
41	10	50	17.5	200	
34	11	50	25	112.5	
44	12	50	17.5	112.5	
17	13	60	13	165	
35	14	50	25	112.5	
40	15	50	17.5	200	
3	16	40	13	60	
29	17	70	17.5	112.5	
7	18	40	22	60	
4	19	60	13	60	
16	20	60	13	165	
2	21	40	13	60	
27	22	30	17.5	112.5	
43	23	50	17.5	112.5	
33	24	50	10	112.5	
37	25	50	17.5	25	
31	26	50	10	112.5	
32	27	50	10	112.5	
20	28	40	22	165	
10	29	60	22	60	
21	30	40	22	165	
36	31	50	25	112.5	
9	32	40	22	60	
19	33	40	22	165	
45	34	50	17.5	112.5	
18	35	60	13	165	
11	36	60	22	60	
22	37	60	22	165	
6	38	60	13	60	
23	39	60	22	165	
38	40	50	17.5	25	
25	41	30	17.5	112.5	
28	42	70	17.5	112.5	
14	43	40	13	165	
13	44	40	13	165	
1	45	40	13	60	

3.9 Effects of Operating Variables on Yield for Thermal Pyrolysis

The variables considered under thermal pyrolysis were: reaction temperature, reaction time and particle size for comparison purposes with the microwave pyrolysis.

3.9.1 Reaction Temperature

The temperature of the furnace is set using an automated temperature controller. When the temperature set is attained, it automatically stops and maintains the temperature. The furnace was allowed to achieve the required temperature before feeding the feedstock into the reactor. The temperature considered are, 200°C, 300°C, 400°C, 500°C, 600°C and 700°C. The liquid products and solid products from each run were weighed and recorded for analysis as shown in table 3.6 below. In each experimental run, 100g of 25mm^2 feedstock was used and subjected to pyrolysis process.

Table 3.6: Effects of reaction temperature on thermal pyrolysis yield

SER.	Reaction Temperature (°C)	Oil Yield (% wt.)	Gas Yield (%wt.)	Volatile Yield (% wt.)
1	200			
2	300			
3	400			
4	500			
5	600			
6	700			

3.9.2 Reaction time

A stop watch was used to measure time and the first step was to determine the time taken for the condensable products to condense at set temperature. The temperature considered was 500°C. The sample size was 100 grams of 25mm^2 feedstock and the liquid fuel yield was monitored after every 10 minutes. The results were then tabulated as shown in the Table 3.7 below:

Table 3.7: Effects of Reaction Time on Thermal Pyrolysis of Used Tyres

SER	Time (Mins)	Liquid Fuel Yield (% wt)	Solid Yield (% wt.)	Gas Yield (% wt.)
1	10			
2	20			
3	30			
4	40			
5	50			
6	60			
7	70			
8	80			
9	100			
10	120			

3.9.3 Particle Size

Tyre (Triangle 1000 R 20 10.00X20 Truck Tyre) was shredded into various sizes using a knife. The sizes used in this experiment were cubic with uniform thickness of 2mm. The cross-sectional area of various sizes was varied for the purpose of investigating the effect of particle size of the feedstock on the yield of thermal pyrolysis process. The cross-sectional areas considered were: 25mm^2, 50mm^2, 100mm^2 and 200mm^2. The temperature considered was 500^{o}C. When there is no more liquid condensing and no more gaseous products, the heating furnace was stopped and liquid fuel collected are weighed. The solid residue was also weighed and the mass of the gaseous products are obtained using the following relation:

Mass of the gaseous products = 100g – mass of liquid fuel – Mass of solid residue

The results were recorded in form of a table as shown in Table 3.8:

Table 3.8: Effects of Particle Size on Yield for Thermal Pyrolysis

SER.	Particle Size (mm^2)	Oil Yield (% wt)	Gas Yield (% wt)	Volatile Yield (% wt)
1	25			
2	50			
3	100			
4	200			

3.9.4 Optimization of the liquid fuel yields of Thermal Pyrolysis using Central Composite Design

This research was mainly focused on the liquid fuel yield. The three variables reaction temperature, reaction time and particle size, were optimized using the CCD.

The central values, the step sizes and the range of variables were as follows: Central reaction temperature of 400°C, step of 100°C and Range of 300°C-500°C; Central reaction time of 60 mins, step of 20 mins and range of 40 mins - 80 mins; and central particle size of 112.5 mm^2, step of 52.5 mm^2, and range of 60 mm^2 – 165mm^2 as shown in Table 3.9 below:

Table 3.9: CCD Matrix

Factors	Units	-alpha	Low	Center Point	High	+alpha
Reaction Temperature	°C	231.8	300	400	500	568.18
Reaction Time	Mins	26.36	40	60	80	93.64
Particle Size	mm^2	24.21	60	112.5	165	200.79

Design Expert 13 Trial Version was used to develop the runs for optimization and the Factorial, Axial and Centre point runs were obtained as shown in Table 3.10. A total of 45 experimental runs were obtained using the software since 3 replications were done on the axial, factorial and centre point runs.

Table 3.10: Optimization table showing factorial, axial and centre point runs for thermal pyrolysis

Std	Run	X1:Reaction temperature (°C)	X2:Reaction time (Mins)	X3:Particle size (mm^2)	Liquid fuel yield (% wt.)
31	1	400	26	112.5	
38	2	400	60	25	
8	3	300	80	60	
5	4	500	40	60	
21	5	300	80	165	
6	6	500	40	60	
44	7	400	60	112.5	
1	8	300	40	60	
4	9	500	40	60	
9	10	300	80	60	
10	11	500	80	60	
11	12	500	80	60	
30	13	568	60	112.5	
41	14	400	60	200	
27	15	232	60	112.5	
23	16	500	80	165	
3	17	300	40	60	
29	18	568	60	112.5	
20	19	300	80	165	
24	20	500	80	165	
40	21	400	60	200	
28	22	568	60	112.5	
37	23	400	60	25	
7	24	300	80	60	
13	25	300	40	165	
36	26	400	94	112.5	
15	27	300	40	165	
45	28	400	60	112.5	
35	29	400	94	112.5	
34	30	400	94	112.5	
32	31	400	26	112.5	
19	32	300	80	165	
33	33	400	26	112.5	
22	34	500	80	165	
39	35	400	60	25	
25	36	232	60	112.5	
12	37	500	80	60	
16	38	500	40	165	
26	39	232	60	112.5	
43	40	400	60	112.5	
17	41	500	40	165	
2	42	300	40	60	
18	43	500	40	165	
42	44	400	60	200	
14	45	300	40	165	

3.10 Characterization of Tyre Pyrolysis Oil for Fuel

This was done at Lab works East Africa Limited-Nairobi using standard methods. The tests done on the tyre pyrolysis oil are the key physiochemical properties and Gas chromatography–mass spectrometry (GC-MS) analysis. The samples considered were from the optimal settings for both microwave and thermal pyrolysis. Specific pure standards of the components to be analyzed were first run in the GC. The retention time these standards were registered in the GC. The pyrolysis oil samples were diluted in an ethanol solvent before analysis and the Tyre Pyrolysis oil to Solvent ratio being 1:10. The GC detector then identified the components in the sample based on the stored information from the standards. It is the Peak Area that determined the concentration of a component in the sample. The retention time shows the identity of the component. The machine used for analysis was Thermo Scientific TM Trace Gold TG-1MS. The inert carrier gas used was Helium and FID detector was used. The temperature settings was an initial temperature of 100^{o}C and final Temperature of 380^{o}C

The physiochemical properties that were tested using either AOAC or ASTM D 2015 methods are:

- Viscosity (mm^{2}/s)
- Density (kg/L)
- Pour Point (^{o}C)
- Flash Point (^{o}C)
- Fire Point (^{o}C)
- Sulphur Content (%)
- Carbon Residue (%)
- Calorific Value (kcal/kg)

All the Physiochemical tests and GC-MS analysis were done using standard methods.

CHAPTER FOUR

RESULTS AND DISCUSSION

This chapter presents the results and discussion of the objectives as per the methodologies outlined in chapter 3.

4.1 Effects of Operating Variables and Optimization of Microwave Pyrolysis of Used Tyres

The operating variables for microwave pyrolysis considered were microwave power, reaction time and particle size.

4.1.1 Effects of Microwave Power on Yield

Microwave power (MP) was varied from 20% to 100% for a microwave with a power output of 900 Watts each run for 30 minutes using particle size of 25mm^2. The results indicated an increase in liquid fuel yield from 15.2 (wt. %) at a microwave power of 20% to an optimum point at 50% microwave power with a yield of 39.1 (wt. %). Then there is a continuous decrease in the liquid fuel yield to 19.1 (wt. %) at 100% microwave power. The solid char indicated a uniformly declining trend from 48.5 (wt. %) at 20% MP to 46.5 (wt. %). There was a slight increase to an optimum value of 48.7 (wt. %) at 50% MP and then a continuous uniform decline to 38.1 (wt. %) at 100% MP. Finally, the Gas yield indicated a declining trend from 36.3 (wt. %) at 20% MP to the lowest yield of 12.2 (wt. %) at 50% MP. There was an increasing trend to a maximum yield of 42.8 (wt. %) at 100% MP. The trend of the results is in agreement with published literature (Song et al., 2017). The rise of the liquid yield from MP of 20% to 50% is as a result of temperature elevation at higher MPs leading to higher oily products (Song et al., 2017). When the MP was further increased from 50% to 100%, there was a declining trend as a result of extremely higher temperatures leading to cracking of oil molecules into lighter gaseous molecules. This fact also substantiated the fact that there was a continuous increase in gaseous products from MP of 50% to 100%. The trend of solid residue was dictated by the trends of gaseous and liquid products as illustrated in figure 4.1

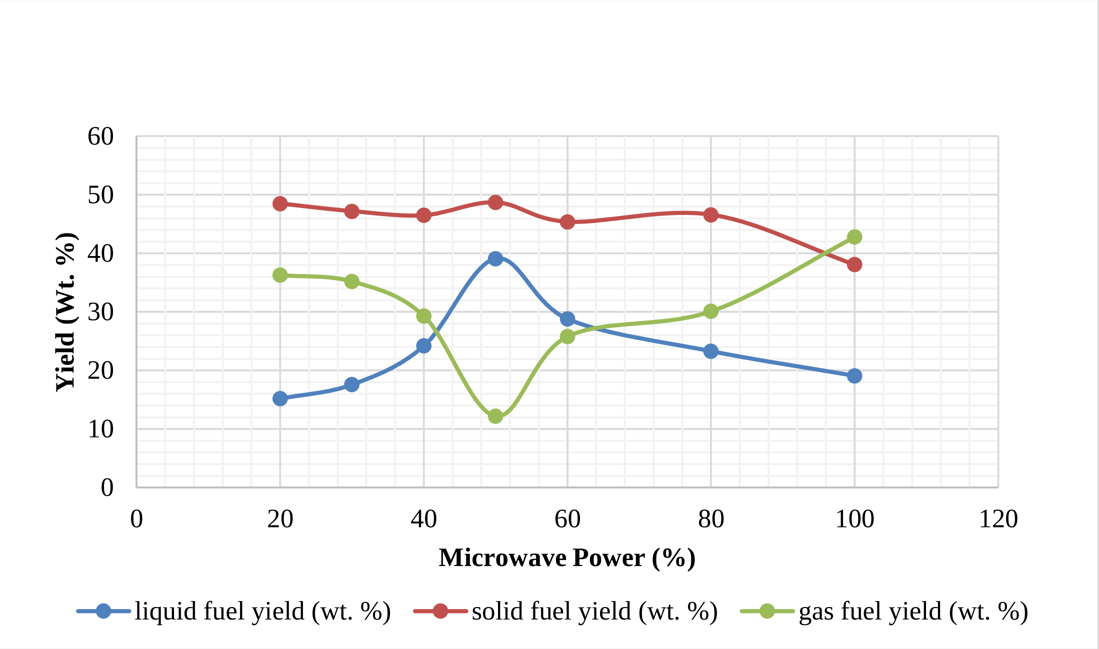


Figure 4.1: Effects of microwave power on liquid, solid and gas yield

4.1.2 Effects of reaction time on yield

The selected MP for the runs to investigate reaction time was constant at 50% because it is the optimal power for tyre pyrolysis oil production. The sample size for all runs was 100 grams of 25 mm^2. The first drop of liquid fuel was collected after 3 minutes. Therefore, the time considered were 8 mins, 13 mins, 18 mins, 23 mins and 28 mins. There was an increase in the liquid fuel yield from 8 mins to 19 mins from 15.3 (wt. %) to 35.0 (wt. %) respectively. Beyond 19 mins, there was no further increase in liquid fuel yield as the curve flattens as shown in table 4.2 and figure 4.2. Similarly, there was an increase in the gas yield from 12.4 (wt. %) to 31.5 (wt. %) from 8 mins to 20 mins respectively. Beyond 20 mins, there was no further increase in gas yield as indicated in figure 4.2. The solid products declined from 72.3 (wt. %) to 35.0 (wt. %) from 8 mins to 20 mins respectively then the yield became constant beyond 20 mins as depicted in figure 4.2. The trend for liquid fuel production over time was in agreement with published literature by Rius Alonso (Rius-Alonso et al., 2011). The increasing trend in liquid fuel and gas production from 8 mins to about 18 minutes is because the feedstock is allowed more time for the pyrolysis process to take place. At about 18 minutes, the pyrolysis process is complete and all products from the feedstock have been cracked leading to no more production of liquid and gaseous products.

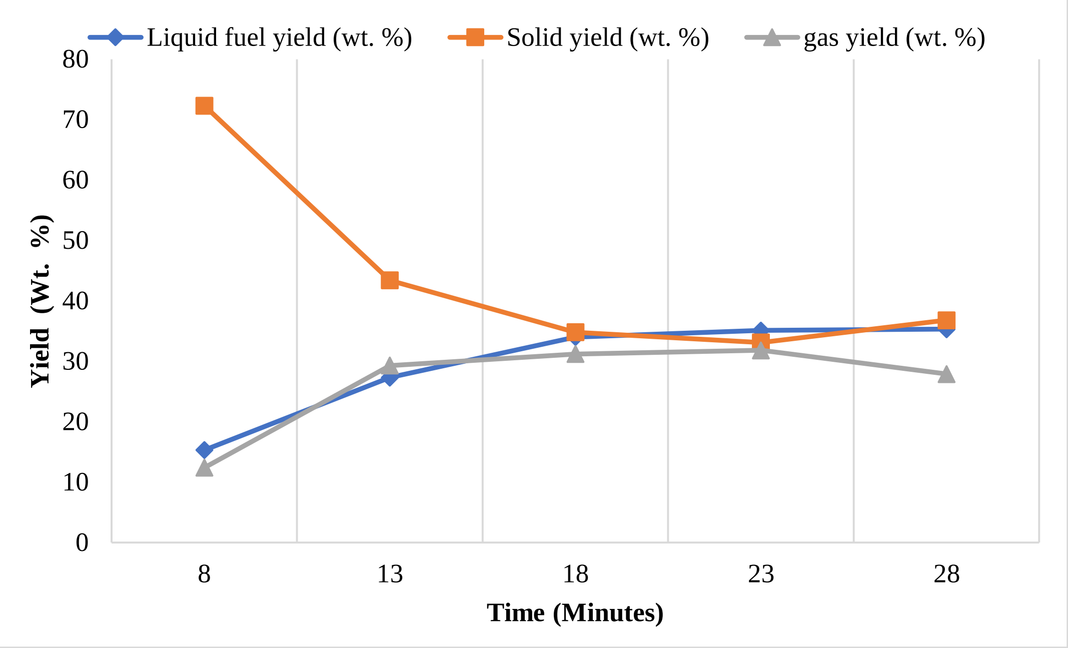


Figure 4.2: Effects of reaction time on yield

4.1.3 Effects of particle size on yield

The particle sizes considered were 25mm^2, 50mm^2, 100mm^2 and 200mm^2 and the sample size was 100 grams each subjected for 25 mins. On the other hand, the MP was kept constant at 100%. The results indicated that there was a continuous decline for liquid fuel yield from 24.1 (wt. %) to 13.1 (wt. %) for particle sizes of between 25mm^2 and 200mm^2 respectively. On the other hand, the gas products depicted a uniform increasing trend from 37.9 (wt. %) to 54.3 (wt. %) for particle sizes between 25mm^2 and 200mm^2 respectively. The decrease in liquid fuel is as a result of reduced exposed surface area for further cracking. The increase in gaseous products with the increase in particle size is as a result of incomplete cracking of tyre particles to form liquid fuel. Most of the components escape as gases, hence the trend. The trend is as shown in figure 4.3.

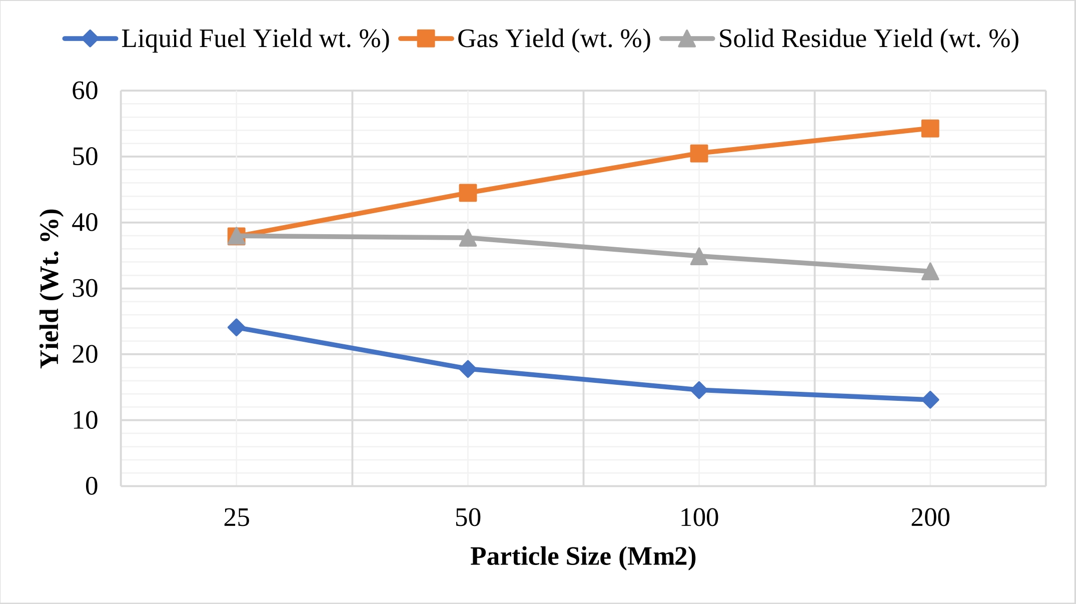


Figure 4.3: Effects of particle sizes on yield

4.1.4 Microwave Pyrolysis of Used Tyres Process Optimization

Central Composite Design (CCD) was used for optimization. The required runs were obtained using Design expert 13-trial version software. The results are as tabulated in table 4.1 for the 45 optimization runs (3 replications for axial, factorial and centre points). The predicted yield are obtained from equation 4.1

Table 4.1: Optimization Runs Results

Std	Run	X1:Microwave power (%)	X2:Reaction time (Mins)	X3:Particle size (mm^2)	Exp. yield (wt. %)	Predicted yield (wt. %)	Residual
12	1	60	22	60	35	35.02	-0.0158
39	2	50	17.5	25	37	37.02	-0.0244
24	3	60	22	165	30	30.01	-0.0117
5	4	60	13	60	29.2	29.17	0.0312
26	5	30	17.5	112.5	27	27.01	-0.0056
30	6	70	17.5	112.5	28.4	28.39	0.0111
42	7	50	17.5	200	32.1	32.1	-0.0009
15	8	40	13	165	26.6	26.59	0.0103
8	9	40	22	60	35.1	35.11	-0.0075
41	10	50	17.5	200	32.1	32.1	-0.0009
34	11	50	25	112.5	31.2	31.18	0.0204
44	12	50	17.5	112.5	35.4	35.4	0.0007
17	13	60	13	165	28.1	28.06	0.0353
35	14	50	25	112.5	31.2	31.18	0.0204
40	15	50	17.5	200	32.1	32.1	-0.0009
3	16	40	13	60	27.5	27.49	0.0062
29	17	70	17.5	112.5	28.4	28.39	0.0111
7	18	40	22	60	35.1	35.11	-0.0075
4	19	60	13	60	29.1	29.17	-0.0688
16	20	60	13	165	28	28.06	-0.0647
2	21	40	13	60	27.5	27.49	0.0062
27	22	30	17.5	112.5	27	27.01	-0.0056
43	23	50	17.5	112.5	35.4	35.4	0.0007
33	24	50	10	112.5	23.2	23.21	-0.0124
37	25	50	17.5	25	37.1	37.02	0.0756
31	26	50	10	112.5	23.2	23.21	-0.0124
32	27	50	10	112.5	23.2	23.21	-0.0124
20	28	40	22	165	30.3	30.3	-0.0034
10	29	60	22	60	35	35.02	-0.0158
21	30	40	22	165	30.3	30.3	-0.0034
36	31	50	25	112.5	31.2	31.18	0.0204
9	32	40	22	60	35.1	35.11	-0.0075
19	33	40	22	165	30.3	30.3	-0.0034
45	34	50	17.5	112.5	35.4	35.4	0.0007
18	35	60	13	165	28.1	28.06	0.0353
11	36	60	22	60	35	35.02	-0.0158
22	37	60	22	165	30	30.01	-0.0117
6	38	60	13	60	29.2	29.17	0.0312
23	39	60	22	165	30	30.01	-0.0117
38	40	50	17.5	25	37	37.02	-0.0244
25	41	30	17.5	112.5	27	27.01	-0.0056
28	42	70	17.5	112.5	28.4	28.39	0.0111
14	43	40	13	165	26.6	26.59	0.0103
13	44	40	13	165	26.6	26.59	0.0103
1	45	40	13	60	27.5	27.49	0.0062

The data in table 4.1 were tested for fit considering linear, two-factor interaction (2FI), quadratic and cubic polynomials. The results are as shown in table 4.2

The highest adjusted R^2 was for the cubic and quadratic models but the cubic model was aliased. Therefore, the quadratic model was considered. Putting into consideration the F value and the p-value, a quadratic model was suggested.

Table 4.2: Summary for model fit- sequential model sum of squares for microwave pyrolysis

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2		
Linear	< 0.0001	< 0.0001	0.4752	0.4249		
2FI	0.3143	< 0.0001	0.4837	0.4766		
Quadratic	**< 0.0001**	**0.3028**	**1**	**0.9999**	**Suggested**	
Cubic	0.2423	0.4338	1	0.9999	**Aliased**	
			Sequential Model Sum of Squares [Type I]			
Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Mean vs Total	41599.36	1	41599.36			
Linear vs Mean	326.79	3	108.93	14.28	< 0.0001	
2FI vs Linear	27.56	3	9.19	1.22	0.3143	
Quadratic vs 2FI	**285.15**	**3**	**95.05**	**1.37E+05**	**< 0.0001**	**Suggested**
Cubic vs Quadratic	0.0038	4	0.001	1.45	0.2423	Aliased
Residual	0.0204	31	0.0007			
Total	42238.88	45	938.64			

The selected quadric model was in the form

$Y=b_0 + \sum_{i=1}^{n} C_i X_i + \sum_{i=1}^{n} C_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} C_{ij} X_i X_j$, where Y is the liquid fuel yield, X_i are the coded variables. The Analysis of Variance (ANOVA) for the quadratic model is given by Table 4.3. As illustrated, the Model F-value of 102633.49 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case, X1, X2, X3, X1.X2, X2.X3, X1.X3, $(X1)^2$, $(X2)^2$ and $(X3)^2$ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The Lack of Fit F-value of 1.27 implies the Lack of Fit is not significant relative to the pure error. There is a 30.28% chance that a Lack of Fit F-value this large could occur due to noise. The p-value for lack-of-fit was greater than 0.05 and therefore, it was not significant. Significant lack of fit implies that the variation of the design points about predicted values is larger than the variation of the replicates about their mean values. Either the model doesn't predict well, or the runs replicate so well that their variance is small, or some combination of the two.

Table 4.3: ANOVA for Response Surface Quadratic model for Microwave Pyrolysis

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	639.49	9	71.05	1.03E+05	< 0.0001	significant
X1-Microwave power	5.74	1	5.74	8292.19	< 0.0001	
X2-Reaction time	232.32	1	232.32	3.36E+05	< 0.0001	
X3-Particle size	88.72	1	88.72	1.28E+05	< 0.0001	
X1.X2	4.68	1	4.68	6762.31	< 0.0001	
X1.X3	0.06	1	0.06	86.67	< 0.0001	
X2.X3	22.82	1	22.82	32954.52	< 0.0001	
(X1)^2	121.19	1	121.19	1.75E+05	< 0.0001	
(X2)^2	142.02	1	142.02	2.05E+05	< 0.0001	
(X3)^2	1.48	1	1.48	2133.74	< 0.0001	
Residual	0.0242	35	0.0007			
Lack of Fit	0.0042	5	0.0008	1.27	0.3028	not significant
Pure Error	0.02	30	0.0007			
Cor Total	639.52	44				

Table 4.4 gives the R-square Values for the model

Table 4.4: R-Square Values for Microwave Pyrolysis

PARAMETER	VALUE
Std. Dev.	0.0263
Mean	30.40
C.V. %	0.0865
R^2	1.0000
Adjusted R^2	1.0000
Predicted R^2	0.9999
Adeq Precision	1113.5527

As illustrated in table 4.4, the "**Predicted R²"** of 0.9999 is in reasonable agreement with the "Adjusted R²" of 1.0000 i.e. the difference was less than 0.2. **"Adeq Precision"** measures the signal to noise ratio. A ratio greater than 4 is desirable. In this particular case, the ratio of 1113.5527 indicates an adequate signal.

Yield,

$$Y=-8389211+2.14258\,X1+6.59046\,X2+0.073437\,X3-0.009815\,X1.X2-0.000095\,X1.X3-0.004127\,X2.X3-0.019255\,(X1)^2-0.145837\,(X2)^2-0.000109\,(X3)^2 \;\ldots\ldots\; (4.1)$$

Where:

X1 is microwave power in %

X2 is particle size in mm^2

X3 is the reaction time in minutes

The equation 4.1 can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the centre of the design space.

4.1.5 Response surface and contour plots

Eqn. 4.1 was used to plot response surface and contours for optimization of liquid fuel yield.

Fig. 4.4 is a plot for yield as a function of microwave power and reaction time. The optima lie close to a microwave power of 50% and reaction time of 17.5 Minutes.

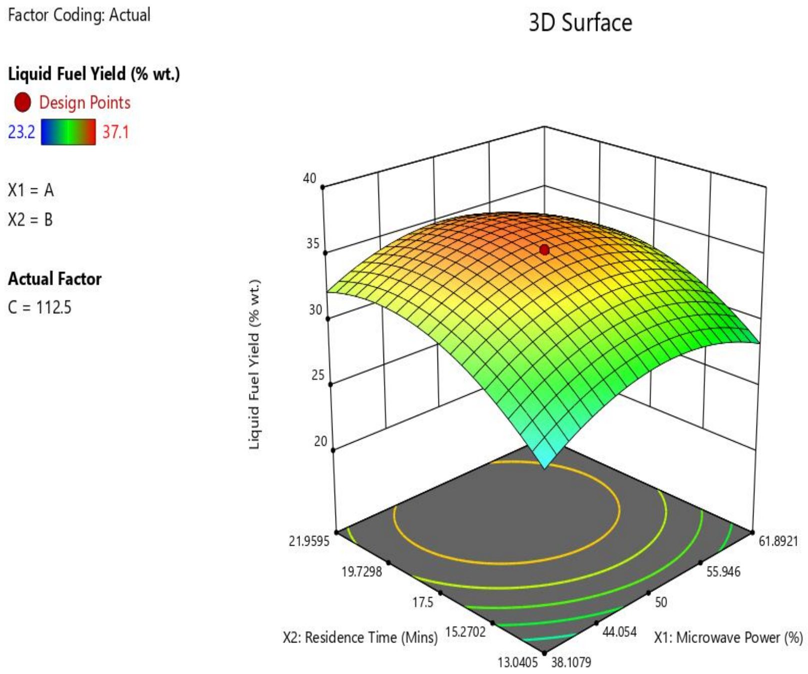


Figure 4.4: RSM plot on effect of microwave power and reaction time on yield

Fig. 4.5 gives a plot for yield as a function of microwave power and particle size. The optima lie close to a microwave power of 50% and particle size of 25mm^2.

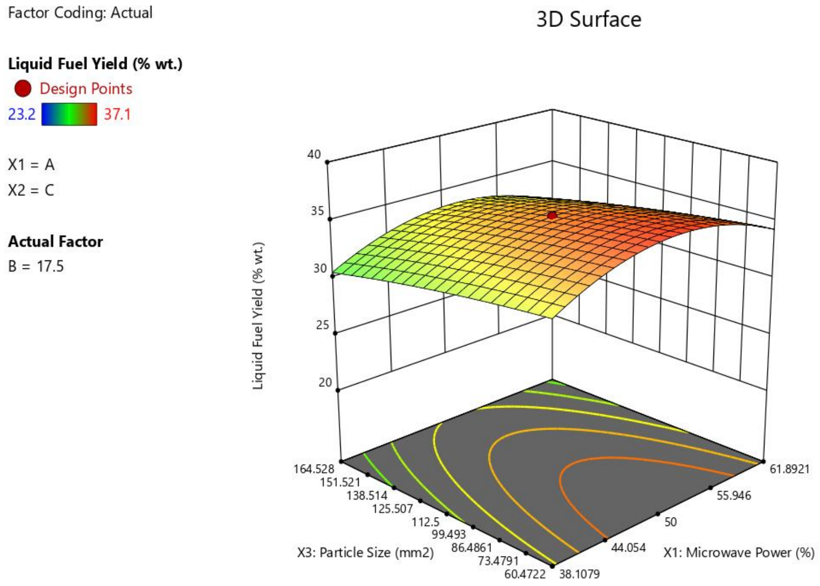


Figure 4.5: RSM plot on effect of microwave power and particle size on yield

Fig 4.6 gives a plot for yield as a function of particle size and reaction time. The optima lie close to a particle size of 25mm^2 and reaction time of 17.5 Minutes.

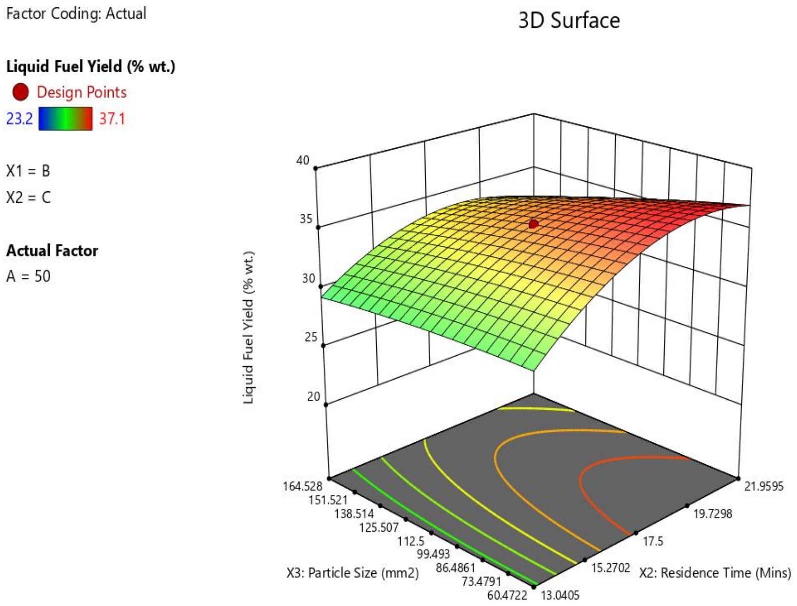


Figure 4.6: RSM plot on effect of particle size and reaction time on yield

4.2 Effects of Operating Variables and Optimization of Thermal Pyrolysis of Used Tyres

This section involves thermal pyrolysis of used tyres as explained in chapter 3. The studies include the effects of operating variables on the yield and optimization.

4.2.1 Effects of Temperature on Yield

An experimental run was done using particle size of 25mm^2. 100g sample was weighed and subjected to pyrolysis at different temperatures for 70 minutes. The temperature range was between 200^{o}C and 700^{o}C and for 25mm^2 particle size; there was an increase in liquid fuel yield from 18.4 (wt.%) at 200^{o}C to an optimum value of 38.0 (wt.%) at 500^{o}C. After 500^{o}C, there was a continuous decrease in the liquid fuel yield up to 30.6 (wt.%) at 700^{o}C as illustrated in Table 4.8 and Figure 4.7. On the other hand, there was a continuous decrease in gas yield from 200^{o}C to 400^{o}C from 13.3 (wt.%) to 8.5 (wt.%).

This was followed by a continuous increase up to 29.8 (wt.%) at 700°C as shown in Figure 4.7. There was a continuous decrease in the solid yield as illustrated in Figure 4.7 from 68.3 (wt.%) at 200°C to 39.6 (wt.%) at 700°C.

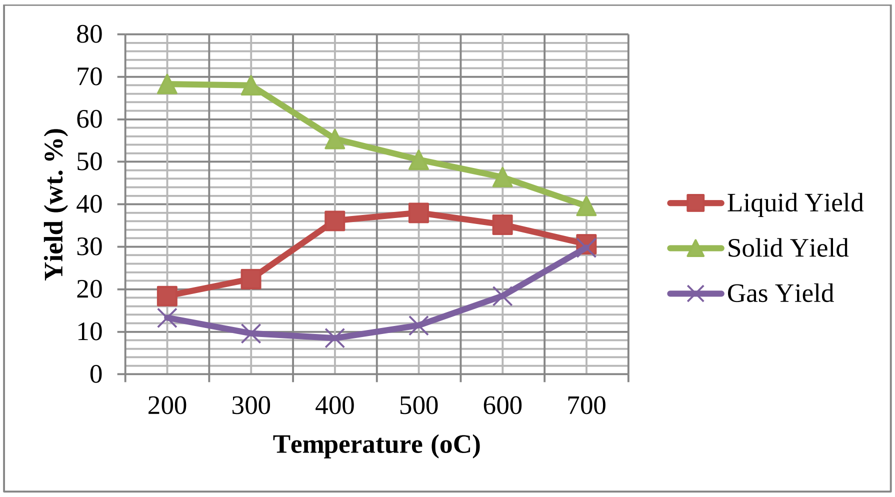


Figure 4.7: Effects of reaction temperature on yield

The results are in agreement with the published literature as reported by Hossain (Hossain & Rahman, 2015). The maximum yield was probably due to better cracking at 500 °C. The thermal cracking was not complete at lower temperatures hence the pyrolysis process was not complete. Beyond 500°C there was a decrease in the liquid fuel yield because of elevated temperatures that cracks further the liquid products to gaseous products. That explains why the gas products increase after 500°C. At lower temperatures, the pyrolysis process was partial and less oil and volatiles were produced and therefore, there was maximum retention of material in form of solid char (Hossain & Rahman, 2015).

4.2.2 Effects of reaction time on yield

A 100g Particle size of $25mm^2$ was used for this particular experiment while temperature was held constant at 500°C. The liquid and gaseous yield increased continuously up to about 60 mins where the yield remained constant. The liquid products increased from 5.2 (wt.%) at 10 mins to 38.2 (wt.%) at 60 mins. It then remained constant up to 120 mins. On the other hand, the gaseous products increased from 8.4 (wt.%) at 10 mins to 43.4 (wt.%) at 60 mins then remained constant. Finally, the solid char was maximum at the beginning with 86.4 (wt.%) at 10 mins to 18.4 (wt.%) then remained constant. The results are in agreement with the published literature by Shah (Shah et al., 2007). At 10 mins, the pyrolysis process is not yet complete and a few hydrocarbons in the tyre have been cracked to form liquid and gas. The cracking continues up to 60 mins which is the optimum time for the pyrolysis process. After 60 mins, the cracking of complex molecules in the tyre is complete and therefore, no more cracking takes place hence the constant trend as illustrated in figure 4.9.

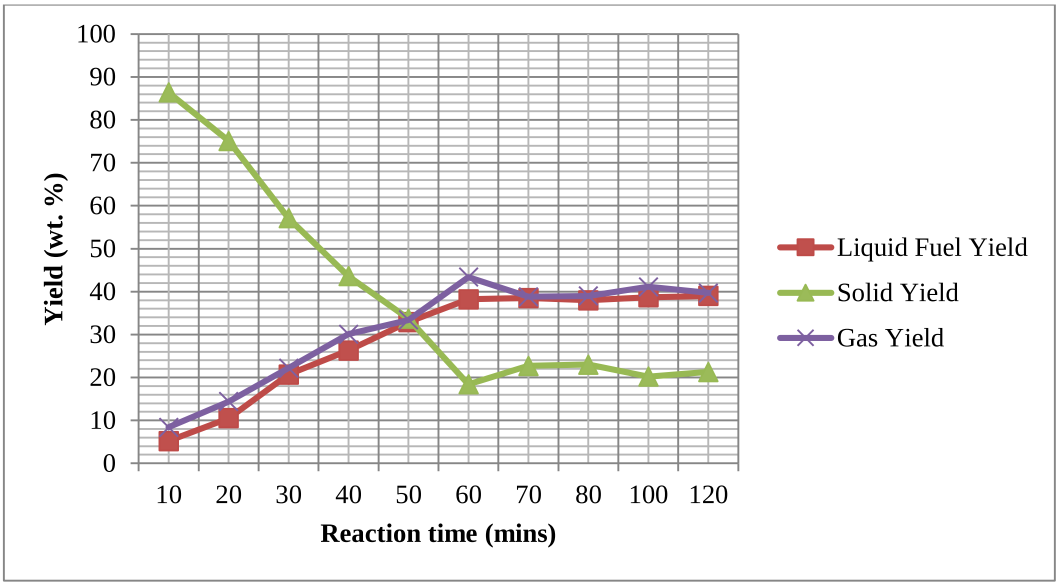


Figure 4.8: Effects of reaction time on yield

4.2.3 Effects of particle size on yield

To study the effects of particle size, 100 grams of different particle sizes were subjected to pyrolysis process for 75 minutes at 50% microwave power. The yield of the liquid fuel decreased continuously from 39.1 (wt.%) for 25mm^2 to 30.6 (wt.%) for 200mm^2. The solid residue had a similar trend from 46.2 (wt.%) for 25mm^2 to 32.2 (wt.%) for

200mm^2. On the other hand, the gaseous products increased with increase in particle size from 14.7 (wt.%) for 25mm^2 to 37.2 (wt.%) at 200mm^2. They are as illustrated in figure 4.9. The results are in agreement with the published literature (Hossain & Rahman, 2015).

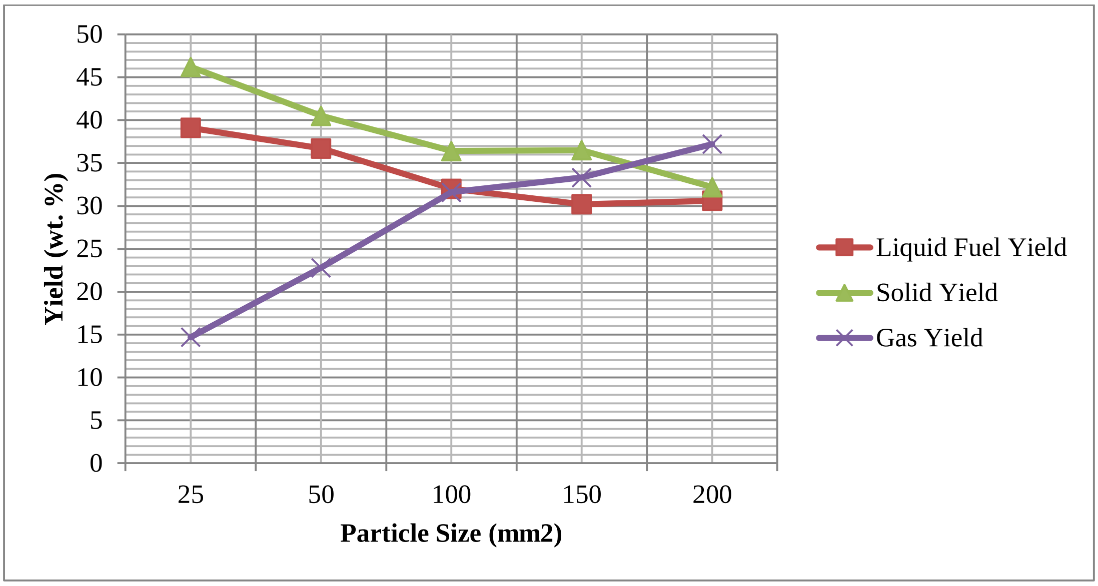


Figure 4.9: Effects of particle size on yield

4.2.4 Thermal Pyrolysis Process Optimization

Central Composite Design (CCD) was used for optimization. The required runs were obtained using Design Expert 13-Trial Version software. The results are as tabulated in table 4.5 for the 45 optimization runs comprising of 3 replications of the axial, factorial and centre point runs. The predicted yields are obtained from equation 4.2

Table 4.5: Optimization Runs for Thermal Pyrolysis Process

Std	Run	X1:Reaction temperature (°C)	X2:Reaction time (mins)	X3:Particle size (mm^2)	Exp. Liquid fuel yield (wt. %)	Predicted liquid yield (wt. %)	Residual
31	1	400	26	112.5	28.5	28.5	-0.0012
38	2	400	60	25	39.3	39.31	-0.0126
8	3	300	80	60	34.3	34.3	0.0041
5	4	500	40	60	35.6	35.59	0.0078
21	5	300	80	165	31.5	31.5	-0.0041
6	6	500	40	60	35.6	35.59	0.0078
44	7	400	60	112.5	37.4	37.4	0.0009
1	8	300	40	60	30.8	30.8	0.0029
4	9	500	40	60	35.6	35.59	0.0078
9	10	300	80	60	34.3	34.3	0.0041
10	11	500	80	60	40.4	40.39	0.0091
11	12	500	80	60	40.4	40.39	0.0091
30	13	568	60	112.5	36.7	36.71	-0.0085
41	14	400	60	200	33.5	33.49	0.0071
27	15	232	60	112.5	27.9	27.9	0.0032
23	16	500	80	165	37.2	37.2	0.0009
3	17	300	40	60	30.8	30.8	0.0029
29	18	568	60	112.5	36.7	36.71	-0.0085
20	19	300	80	165	31.5	31.5	-0.0041
24	20	500	80	165	37.2	37.2	0.0009
40	21	400	60	200	33.5	33.49	0.0071
28	22	568	60	112.5	36.7	36.71	-0.0085
37	23	400	60	25	39.3	39.31	-0.0126
7	24	300	80	60	34.3	34.3	0.0041
13	25	300	40	165	27	27.01	-0.0053
36	26	400	94	112.5	36.4	36.4	-0.0041
15	27	300	40	165	27.1	27.01	0.0947
45	28	400	60	112.5	37.4	37.4	0.0009
35	29	400	94	112.5	36.4	36.4	-0.0041
34	30	400	94	112.5	36.4	36.4	-0.0041
32	31	400	26	112.5	28.5	28.5	-0.0012
19	32	300	80	165	31.5	31.5	-0.0041
33	33	400	26	112.5	28.5	28.5	-0.0012
22	34	500	80	165	37.2	37.2	0.0009
39	35	400	60	25	39.3	39.31	-0.0126
25	36	232	60	112.5	27.9	27.9	0.0032
12	37	500	80	60	40.4	40.39	0.0091
16	38	500	40	165	31.4	31.4	-0.0004
26	39	232	60	112.5	27.9	27.9	0.0032
43	40	400	60	112.5	37.4	37.4	0.0009
17	41	500	40	165	31.4	31.4	-0.0004
2	42	300	40	60	30.8	30.8	0.0029
18	43	500	40	165	31.4	31.4	-0.0004
42	44	400	60	200	33.5	33.49	0.0071
14	45	300	40	165	26.9	27.01	-0.1053

The data in table 4.5 were tested for fit considering linear, two-factor interaction (2FI), quadratic and cubic polynomials. The results are as shown in table 4.6, the highest adjusted R^2 was for quadratic model and it was not aliased. Putting into consideration the F value and the p-value, a quadratic model was suggested.

Table 4.6: Summary for model fit- sequential model sum of squares for Thermal pyrolysis

Source	**Sequential p-value**	**Lack of Fit p-value**	**Adjusted R^2**	**Predicted R^2**		
Linear	< 0.0001	< 0.0001	0.8404	0.8283		
2FI	0.6758	< 0.0001	0.8345	0.8338		
Quadratic	**< 0.0001**	**0.794**	**1**	**1**	**Suggested**	
Cubic	0.7215	0.573	1	0.9999	Aliased	
Sequential Model Sum of Squares [Type I]						
Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Mean vs Total	51592.48	1	51592.48			
Linear vs Mean	628.84	3	209.61	78.25	< 0.0001	
2FI vs Linear	4.28	3	1.43	0.513	0.6758	
Quadratic vs 2FI	**105.53**	**3**	**35.18**	**57067.7**	**< 0.0001**	**Suggested**
Cubic vs Quadratic	0.0014	4	0.0003	0.5203	0.7215	Aliased
Residual	0.0202	31	0.0007			
Total	52331.15	45	1162.91			

The selected quadric model was in the form:

$Y = b_0 + \sum_{i=1}^{n} C_i X_i + \sum_{i=1}^{n} C_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} C_{ij} X_i X_j$, where Y is the liquid fuel Yield, X_i are the coded variables. The Analysis of Variance (ANOVA) for the quadratic model is given by Table 4.7. As illustrated, the Model F-value of 133149.35 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case, X1, X2, X3, (X1.X2), (X1.X3), (X2.X3), $(X1)^2$, $(X2)^2$, $(X3)^2$ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The Lack of Fit F-value of 0.47 implies the Lack of Fit is not significant relative to the pure error. There is 79.4 % chance that a Lack of Fit F-value this large could occur due to noise. The p-value for lack-of-fit was greater than 0.05 and therefore, it was not significant.

Table 4.7: ANOVA for Response Surface Quadratic Model for Thermal pyrolysis

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	738.65	9	82.07	1.331E+05	< 0.0001	significant
X1-Reaction Temperature	281.53	1	281.53	4.567E+05	< 0.0001	
X2-Reaction Time	223.35	1	223.35	3.624E+05	< 0.0001	
X3-Particle Size	123.96	1	123.96	2.011E+05	< 0.0001	
X1.X2	2.53	1	2.53	4112.66	< 0.0001	
X1.X3	0.2400	1	0.2400	389.36	< 0.0001	
X2.X3	1.50	1	1.50	2433.53	< 0.0001	
$(X1)^2$	58.96	1	58.96	95654.80	< 0.0001	
$(X2)^2$	55.28	1	55.28	89690.31	< 0.0001	
$(X3)^2$	2.26	1	2.26	3668.04	< 0.0001	
Residual	0.0216	35	0.0006			
Lack of Fit	0.0016	5	0.0003	0.4721	0.7940	not significant
Pure Error	0.0200	30	0.0007			
Cor Total	738.67	44				

Table 4.8 gives the R-square Values for the model;

Table 4.8: R-Square Values for Thermal Pyrolysis

PARAMETER	VALUE
Std. Dev.	0.0248
Mean	33.86
C.V. %	0.0733
R^2	1.0000
Adjusted R^2	1.0000
Predicted R^2	1.0000
Adeq Precision	1143.7156

As illustrated in table 4.8, the “**Predicted R^2”** of 1.0000 is in reasonable agreement with the “Adjusted R^2” of 1.0000; i.e. the difference was less than 0.2. **“Adeq Precision”** measures the signal to noise ratio. A ratio greater than 4 is desirable. In this particular case, the ratio of 1143.7156 indicates an adequate signal.

The quadratic equation is given by:

$$Y = -17.61552 + 0.163074\ X1 + 0.537908\ X2 - 0.010641\ X3 + 0.000162\ X1.X2 - 0.000019\ X1.X3 + 0.000238\ X2.X3 - 0.000181\ (X1)^2 - 0.004279\ (X2)^2 - 0.000130\ (X3)^2 \ldots\ldots\ldots\ldots\ldots \quad (4.2)$$

Where Y is the liquid fuel yield in (wt.%)

X1 is the reaction temperature in ^{o}C

X2 is the reaction time in Mins

X3 is the particle size in mm^2

The equation 4.2 can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the centre of the design space.

4.2.5 Response Surface and Contour Plots for Thermal Pyrolysis

Eqn. 4.2 was used to plot response surface and contours for optimization of liquid fuel yield. Fig. 4.11 is a plot for yield as a function of reaction temperature and reaction time. The optima lie close to a reaction temperature of 500^{o}C and reaction time of 80 Minutes.

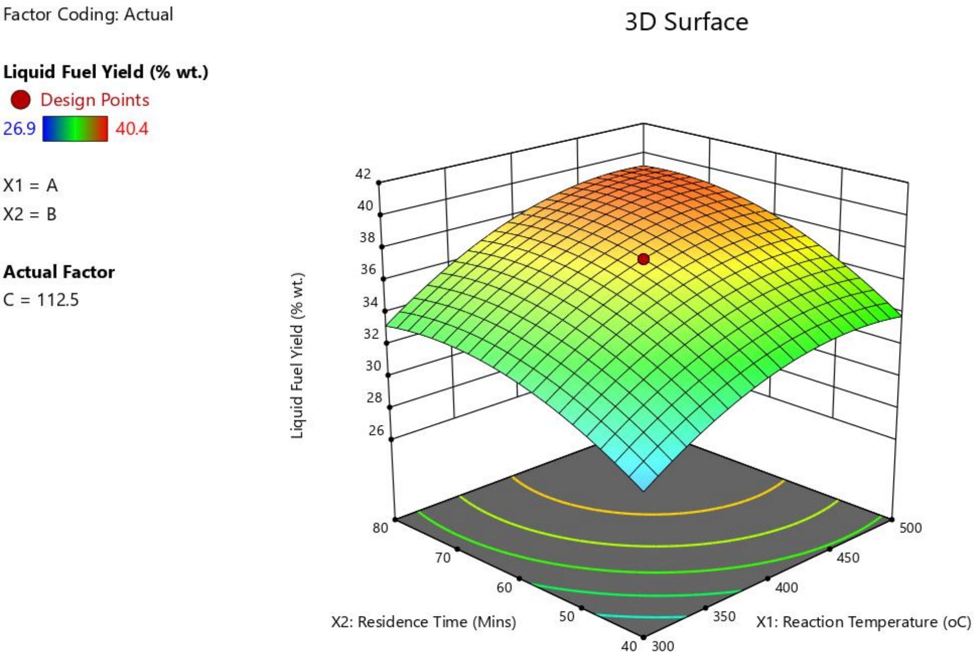


Figure 4.10: RSM plot on effect of reaction temperature and reaction time on yield

Fig. 4.11 gives a plot for yield as a function of reaction temperature and particle size. The optima lie close to a reaction temperature of 500^{o}C and particle Size of 60mm^2.

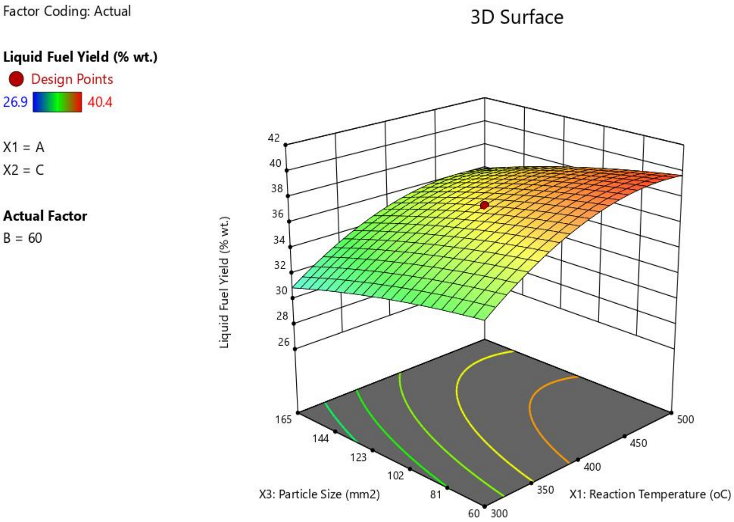


Figure 4.11: RSM plot on effect of Reaction Temperature and Particle Size on Yield

Fig 4.12 gives a plot for Yield as a function of particle size and reaction time. The optima lie close to a particle size of 60mm^2 and reaction time of 80 minutes.

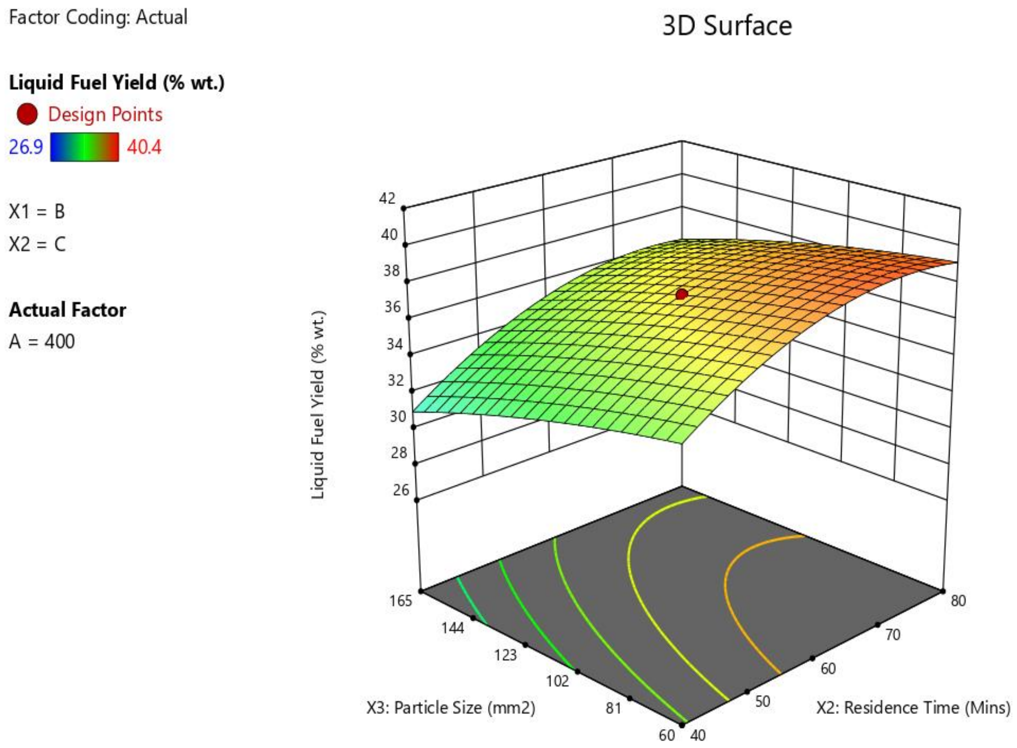


Figure 4.12: RSM plot on effect of particle size and reaction time on yield

4.3 Characterization of Tyre Pyrolysis Oil for Liquid Fuel

4.3.1 Physiochemical Properties

The physiochemical properties were conducted using standard methods at Lab Works East Africa Ltd. Table 4.9 summarizes the results of the physiochemical properties tests for both microwave and thermal pyrolysis. On the other hand, table 4.10 compares the results with published literature concerning a research done by Hariram (Hariram et al., 2017). The research compares properties of waste tyre pyrolysis oil, waste plastics pyrolysis oil and that of commercial diesel.

Table 4.9: Physiochemical Properties of TPO

Parameter	Method	Results	
		Microwave Pyrolysis	Thermal Pyrolysis
Viscosity @ 40ºC (mm^2/s	AOAC	3.07 mm^2/s=3.07 cSt	3 mm^2/s=3 cSt
Ash content (%)	AOAC	1.9	1.5
Density @ 15ºC (Kg/L)	AOAC	0.91	0.91
Pour Point (ºC)	ASTM D2015	6.7	6.5
Flash Point (ºC)	ASTM D2015	61	59
Fire Point (ºC)	ASTM D2015	65	62
Sulphur Content (%)	AOAC	0.08	0.08
Carbon Residue (%)	ASTM D2015	0.75	0.68
Calorific Value	ASTM D2015	11700 kcal/kg=48.99 MJ/kg	11300 kcal/kg=47.31 MJ/kg

Table 4.10: Physiochemical Properties of TPO compared to that of Petroleum Diesel

Properties	Units	Commercial Diesel	WTPO	WPPO	Testing Methods
Gross calorific value	MJ/kg	43.9	42.7	39.45	IS:1448 P:6
Kinematic viscosity, @ 40°C	cSt	2.1	3.4	2.2	IS:1448 P:25
Flash point	°C	5 2	4 6	4 4	IS:1448 P:20
Fire point	°C	5 7	5 3	4 7	IS:1448 P:20
Density @ 15°C	kg/m^3	832	936	837.5	IS:1448 P:16
Cetane index	-	46.4	5 5	6 8	IS:1448 P:9
Carbon residue	%	0.37	2.21	72.79	IS:1448 P:122
Sulphur content	%	0.047	0.99	0.034	IS:1448 P:33
Ash content	%	0.01	0.33	0.00027	IS:1448 P:126

Source: (Hariram et al., 2017)

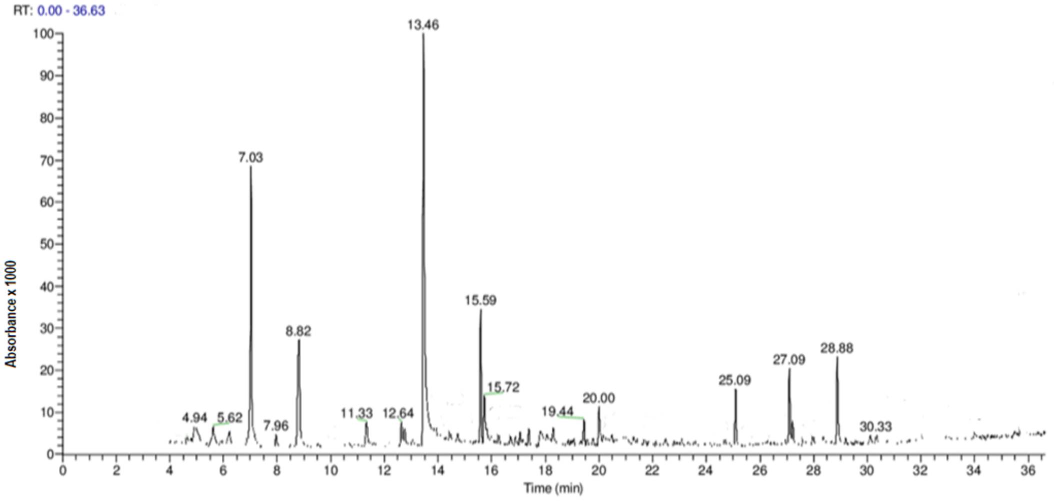
From the results, most of the parameters are close to that of commercial diesel as indicated on tables 4.9 and table 4.10. The gross calorific value of TPO is at 48.99 MJ/kg while that obtained by Hariram (Hariram et al., 2017) for WTPO was at 42.7MJ/kg. The gross calorific value of commercial diesel is between 42-46 MJ/kg according to World Nuclear Association (World Nuclear Association, 2016) and 43.9 MJ/kg according to Hariram (Hariram et al., 2017). This implies that the TPO has calorific value higher than that of commercial diesel. Other parameters such as flash point, fire point, density and viscosity are close to that of petroleum diesel. Upon further purification, TPO can be used directly in internal combustion engines instead of diesel. The calorific value of Heavy Fuel Oil (HFO) is at 41MJ/kg (Kenya, 2020). The required standards for HFO sold in Kenya (Kenya, 2020) is that it must meet the following critical parameters: Maximum Kinematic viscosity @ 500C of 180 cst, sulphur content of 2.3% maximum and a minimum net calorific value of 41MJ/kg. Therefore, TPO qualifies to be used directly as an HFO.

4.3.2 GC-MS Analysis

The TPO Optimal samples about 1 Litre each was collected in the laboratory for Microwave pyrolysis and thermal pyrolysis process. Specific pure standards of the components to be analyzed were first run in the GC. The retention time these standards were registered in the GC. The pyrolysis oil samples were diluted in an ethanol solvent before analysis and the Tyre Pyrolysis oil to Solvent ratio being 1:10. The GC detector then identified the components in the sample based on the stored information from the standards. It is the Peak Area that determined the concentration of a component in the sample. The retention time shows the identity of the component. The machine used for analysis was Thermo Scientific TM Trace Gold TG-1MS. The inert carrier gas used was Helium and FID detector was used. The temperature settings was an initial temperature of 100^oC and final Temperature of 380^oC. The results of GC analysis are as illustrated in table below: From the analysis, it is evidenced that the TPO from both processes comprises of a complex mixture of hydrocarbons with major components being Limonene, Toluene and Xylene. The proportions of tested components are as illustrated in tables 4.11 and 4.12 for microwave and thermal pyrolysis respectively. The results are in agreement with the published literature by Flaresgoiti (FLaresgoiti et al., 2004).

Table 4.11: GC analysis of optimal Liquid fuel from microwave pyrolysis

Retention Time (RT) (Mins)	Components	Molecular Formula	Peak Area %
13.46	D-Limonene	$C_{10}H_{16}$	20.23
7.03	Toluene	C_7H_8	10.65
8.82	m-Xylene	C_8H_{10}	7.31
27.09	1-methylnaphthalene	$C_{11}H_{10}$	5.8
15.59	Naphthalene	$C_{10}H_8$	5.42
28.88	Estradiol, 3-deoxy	$C_{18}H_{24}O$	3.5
15.72	3,4-Dihydro-1-methylnaphthalene	$C_{11}H_{12}$	2.74
25.09	Naphthalene,2,7 –dimethyl	$C_{12}H_{12}$	2.5
20	Naphthalene 1,7-dimethyl	$C_{12}H_{12}$	1.96
11.33	o-Xylene	C_8H_{10}	1.4
19.44	Naphthalene, 1,2-dihydro	$C_{10}H_{10}$	1.2
12.64	Cyclohexane, 1-methyl-5-(1-methylethenyl)-(R)	$C_{10}H_{16}$	1.1
5.62	Benzene	C_6H_6	0.92
4.94	Ethylenzene	C_8H_8	0.6
30.33	Fluoranthene	$C_{16}H_{10}$	0.45
7.96	2,4-dimethyl-quinolin	$C_{11}H_{11}N$	0.41

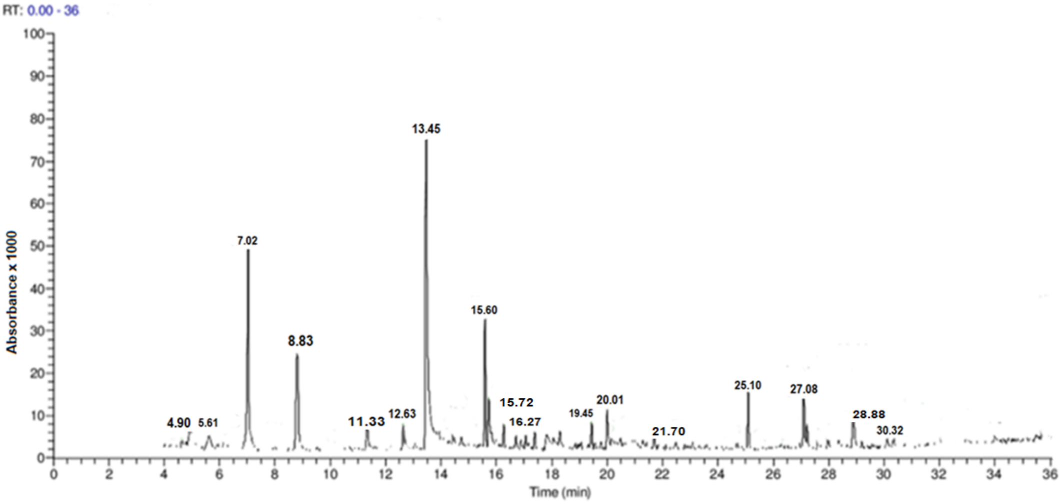


The y-axis is the absorbance levels and does not necessarily reflect the concentration of the components. The concentration of the test components is expressed by the Peak Area. The x-axis is the Retention Time.

Figure 4.13: Chromatogram for TPO from microwave pyrolysis

Table 4.12: GC analysis of optimal Liquid fuel from Thermal pyrolysis

Retention Time (RT) (Mins)	Components	Molecular Formula	Peak Area %
13.45	D-Limonene	$C_{10}H_{16}$	14.55
7.02	Toluene	C_7H_8	7.45
8.83	m-Xylene	C_8H_{10}	6.82
15.60	Naphthalene	$C_{10}H_8$	5.40
27.08	1-methylnaphthalene	$C_{11}H_{10}$	4.45
15.72	3,4-Dihydro-1-methylnaphthalene	$C_{11}H_{12}$	3.20
28.88	Estradiol, 3-deoxy	$C_{18}H_{24}O$	2.50
25.10	Naphthalene,2,7-dimethyl	$C_{12}H_{12}$	2.32
20.01	Naphthalene 1,7-dimethyl	$C_{12}H_{12}$	2.05
16.27	Fuorene	$C_{13}H_{10}$	2.00
19.45	Naphthalene, 1,2-dihydro	$C_{10}H_{10}$	1.75
11.33	o-Xylene	C_8H_{10}	1.45
21.70	Acenaphthylene	$C_{12}H_8$	1.01
5.61	Benzene	C_6H_6	1.00
12.63	Cyclohexane, 1-methyl-5-(1-methylethenyl)-(R)	$C_{10}H_{16}$	1.00
4.90	Ethylenzene	C_8H_8	0.68
30.32	Fluoranthene	$C_{16}H_{10}$	0.62



The y-axis is the absorbance levels and does not necessarily reflect the concentration of the components. The concentration of the test components is expressed by the Peak Area. The x-axis is the Retention Time.

Figure 4.14: Chromatogram for TPO from Thermal Pyrolysis

4.4 Energy Needs for Optimal Microwave and Thermal Pyrolysis of Used Tyres

4.4.1 Microwave Pyrolysis

A 37.1 g of liquid fuel with a density of 0.91 kg/L was produced in 17.5 minutes using 50% Power level for a microwave oven ratted 900W. A magnetic stirrer rated 50W was used and the magnetron efficiency of a microwave oven was taken to be 62% according to Devine (Devine & Leadbeater, 2011).Therefore, the energy requirements can be summarized as follows:

Table 4.13: Microwave pyrolysis energy requirement

Microwave energy consumption (Power x Time)	**(50/100)x(900/0.62)x (17.5/60) =211.7Wh**
Stirrer energy consumption (Power x time)	50x(17.5/60)=14.6Wh
Total energy consumption	**226.3Wh**
Total energy consumption per liter of Tyre pyrolysis oil	226.3/(37.1/1000)x(1/0.91)=5550.75Wh
Total energy consumption per kg of Tyre pyrolysis oil	5550.75/0.91 = 6.0997kWh

4.4.2 Thermal Pyrolysis

A 40.4 g of Tyre pyrolysis oil was produced using two heating elements each having a power rating of 300W. The density of the liquid produced was 0.91kg/L and was produced after 80 minutes. Electric stirrer rated 50W was used. The energy requirement can be summarized as shown in the table below:

Table 4.14: Thermal pyrolysis energy requirement

Heating element energy consumption (Power x Time)	**(2x300) x (80/60) =800Wh**
Stirrer energy consumption (Power x time)	50x(80/60) =66.67Wh
Total energy consumption	866.67Wh
Total energy consumption per liter of Tyre pyrolysis oil	866.67/(40.4/1000)x(1/0.91)=19521.53Wh
Total energy consumption per kg of Tyre pyrolysis oil	1951.53/0.91 =21452.23Wh=**21.452kWh**

From tables 4.13 and 4.14, it can be concluded that the Microwave pyrolysis requires less energy compared to Thermal pyrolysis process to produce 1 kg of liquid fuel. The energy requirement is reduced by (21.452-6.1)/21.452 x100 % =71.6% compared to thermal pyrolysis.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary and Conclusions

The main aim of this study was to perform microwave pyrolysis and compare with that of thermal pyrolysis. The effects of selected variables were studied for both experiments and the variables were: microwave power, reaction time and particle sizes for microwave pyrolysis and reaction temperature, reaction time and particle size for Thermal pyrolysis. Besides, optimization of liquid fuel yield was performed using Central composite Design in both experiments. Finally, characterization of liquid fuel was done at Lab Works East Africa Ltd where Physiochemical properties of the TPO were tested and GC-MS analysis performed on optimal samples to identify the key components of the liquid products. The following Conclusions were drawn from the study:

a. The microwave power affects the yield of microwave pyrolysis as explained in Chapter 4. At lower power levels, the liquid fuel yield is lower and as the power is increased, the liquid fuel yield increases up to an optimal point at 50% power level. Beyond the optimal point, the liquid fuel yield decreases uniformly. The gas yield is relatively higher at lower power levels and decreases continuously up to the lowest value at 50% power level. As Microwave power is further increased, the gas yield increases up to the optimal gas yield at 100% power level. The solid residue is dependent on the trends of liquid and gas yield but shows a declining trend as power level is increased.

b. The Reaction time was found to affect the microwave pyrolysis process and at lower reaction time, the quantities solid residue is higher while the liquid and gas products are lower as explained in chapter 4. As the Reaction time is increased, the liquid and gas yields increase while the solid residue decreases. Beyond 18 minutes, the yields of liquid, gas and solid residue remains constant for 100 grams sample indicating completion of pyrolysis process.

c. The particle size affects microwave pyrolysis process as explained in chapter four. The yield of solid and liquid products decreases with an increase in particle sizes while the yield of gaseous products increases with an increase in particle size.
d. The optimal yield of liquid fuel for microwave pyrolysis was achieved at a microwave power of 50%, particle size of $25mm^2$ and reaction time of 17.5 minutes. $25mm^2$ was the smallest particle sizes for this specific study and even smaller particle sizes can be achieved and is likely to give better results.
e. The reaction temperature affects the yield of thermal pyrolysis process as illustrated in chapter four. The liquid fuel yield increases with increase in temperature up to an optimal yield at about 500^oC beyond which the liquid fuel yield decreases. On the other hand, the gas yield decreases with increase in temperature up to 500^oc beyond which the gas yield starts to increase. The solid residue is dependent on the liquid and gas yields and it portrays a decreasing trend as temperature rises.
f. Reaction time affects thermal pyrolysis as outlined in chapter four. The liquid and gas yields increase with increase in reaction time while the solid residue decreases uniformly. At about 60 minutes and beyond, the solid, liquid and gaseous products remain constant and that is a clear indication that the pyrolysis process is completed.

g. Particle size affects the thermal pyrolysis process as outlined in chapter four. Liquid and solid products yields reduce with increase in particle sizes while the gaseous products increases with increase in particle size.

h. The optimal yield of liquid fuel for Thermal pyrolysis was achieved at a reaction temperature of 500^oC, particle size of $60mm^2$ and reaction time of 80 minutes. 60 mm^2 was among the smallest particle sizes for this specific study and even smaller particle sizes can be achieved and is likely to give better results.

i. Both microwave pyrolysis and Thermal pyrolysis gives almost the same results in terms of optimal liquid fuel yield. However, Microwave Pyrolysis is preferred because it uses less time to complete pyrolysis process for a similar amount of feedstock. It reduces reaction time from 80 minutes to about 18 minutes. The percentage reduction in reaction time can be calculated as ((80-18)/80)x100=77.5%

j. Microwave pyrolysis reduces the energy requirement by 71.6% compared to thermal pyrolysis as illustrated in chapter 4.

k. The Tyre Pyrolysis Oil from both Microwave and Thermal pyrolysis have almost the same physiochemical properties as commercial diesel as illustrated in Chapter 4. It was found that the calorific value of the Oil from microwave pyrolysis is slightly higher (48.99 MJ/kg) compared to that of thermal pyrolysis (47.31 MJ/kg). Both have calorific value slightly higher than that of petroleum diesel at 43.9 MJ/kg according to Hariram (Hariram et al., 2017)

l. The GC-MS analysis on liquid fuel indicated that they comprise of complex mixtures of organic compounds with larger proportions of Limonene, Toluene and Xylene.

5.2 Recommendations

This study recognizes and appreciates the advances that have been made in wastes to energy conversions and recommends the following for further research:

a. The use of appropriate catalyst in both Thermal and Microwave pyrolysis to further reduce the reaction time for better efficiency.

b. The liquid fuel produced is not refined despite the fact that it has a higher calorific value compared to petroleum diesel. Research is still needed in refining the liquid fuel so as to be used directly in the internal combustion engines. Furthermore, more research is needed on the composition and possible use of gaseous products and the solid residue.

c. Further research is recommended on the up scaling of microwave pyrolysis. Most of the researches done are mainly focusing on using prototypes.

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APPENDICES

Appendix A: Response Surface Methodology {Central Composite Design (CCD)}

Source: (Olawoye, n.d.)

Response surface methodology, or **RSM**, is a collection of mathematical and statistical techniques useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response. For example, suppose a chemical engineer wishes to find the levels of temperature (X1) and pressure (X2) that maximize the yield (Y) of a process.

This optimize response can either be maximum, minimum or targeted values. Maximum is the case of maximum enzyme yield when the factors such as temperature, pH and time of fermentation are being optimized. It can also be minimum in the case of microbial contamination and can be targeted when increase or decrease in the parameter will be regarded as defects such as the strength of material.

Response surface methodology can basically be categorized into 4 groups namely:

(1) Box-Behnken Design
(2) Central Composite Design
(3) Dohlert design
(4) Mixture response surface methodology

In Box-Behnken Design: This is design as an alternative to time consuming and labour intensive factorial design. In this design, the minimum number of factors (continuous or numerical factors) it can accommodate is 3 and it has 3 levels of factors namely:

- Upper level
- Lower level

- Centre point

Doehlert Design: This design can be likening to central composite design but the difference is that the number of levels in Doehlert design is irregular in that the first factor had 3 levels; the second factor is made up of five levels while the third factors had 7 levels. Therefore, as the factor increases the levels increase by +2. The advantage of Doehlert design is its flexibility over other type of experimental design.

Mixture response surface methodology: This type of RSM is used when the totality of the component is equal to 1 or 100. In this type of RSM, two or more components are used in the production or formulation of products. Mixture RSM is usually used by chemical engineers, pharmaceutical industry and the food scientists. Example of a mixture response is the production of bread using two flours or production of active drugs using two active compounds or two drug excipient substances. Like other RSM design, it is based on quadratic or second-order polynomial model.

Central Composite Design (CCD) is a response surface design which apart from the 3 level factors has axial or star point. The axial or star point usually denoted as (a) increases the number of levels to 5 levels thereby giving the experimental design flexibility.

Its advantages over Box-Behnken is that it allows the experimental designer to know what effect the factors had on response is the experimental designer goes beyond or below the chosen levels of factors. It also provides high quality predictions of linear and quadratic interaction effects of parameters affecting the process.

However, in Central Composite Design the minimum numbers of factors it can accommodate is two (i.e. numerical or continuous factors). The number of experimental obtained at each number of factors is given by the formula.

$N = 2^n + 2xn + nc$

Where N is the number of runs

n is the number of factors

nc is the number of centre points the designer desired for example, if we have 2 factors then;

N = 2^2 + 2^2 + nc (which can be set between 2 to 6)

Therefore, N = 4 + 4 +2 (if the number of centre point is assumed to be 2)

N = 10 runs. So in an experiment where the experimenter wants to look at the effect of temperature and pressure on methanol yield. The Central Composite Design will be like this:

Factors		-a (1.1414)	-1 (lower	0 (Center)	1 (Upper Level)	+a (1.414)
		(axial point)	level)			(axial point)
X1	(Temperature)	53.79	60	75	90	96.21
X2	(Pressure)	3.965	5	7.5	10	11.035

To get value for the axial point we need to first get the value for *a*

Alpha (a) = $2^{k/4}$ where K is the number of factor

For the example above where the number of factors is two

Therefore, a = $2^{2/4}$ = $2^{1/2}$ = V2 which is equal to 1.414

To get value for the axial point, we apply this equation.

Axial point = mean of both the upper or lower level ± a (range between the upper and lower level divided by 2)

Axial point = X ± a (Range/2)

For the above example

Axial point for temperature = 75 ± 1.414 ((90-60)/2)

= 75 ± 1.414 (30/2)

= 75 ± 1.414 (15)

= 75 ± 21.21
For the upper axial point (i.e. + a) = 75+21.21 = 96.21

For the lower axial point (i.e. - *a)* = 75-21.21 = 53.79

Applying the same formula for the calculation of axial points for

pressure we have Axial point = 7.5 ± 1.414 (5/2)

Upper axial point = 7.5 + 1.414 (2.5) = 11.035

Lower axial point = 7.5 - 1.414 (2.5) = 3.965

The combination of the experimental run for the coded value will be:

*Note that the number of experimental run for the discussed example is 10 runs
Therefore, the experimental runs are shown in table A2

Table A2: Showing the experimental design for both the coded and actual value.

Experiment Runs	X1 (Temperature °C)	X2 (Pressure bar)	Yield
1	-1 (70)	-1 (5)	
2	1 (90)	-1 (5)	
3	-1 (70)	1 (10)	
4	1 (90)	1 (10)	
5	- a (96.21)	0 (7.5)	
6	a (53.79)	0 (7.5)	
7	0 (75)	- a (11.035)	
8	0 (75)	a (3.965)	
9	0 (75)	0 (7.5)	
10	0 (75)	0 (7.5)	

*Note: Runs 1 to 4 is known as factorial runs, runs 5 to 8 is kwon as axial runs while runs 9 and 10 is known as centre point.

The meaning of the table above is that when carrying out the experiment for example for runs 1, you set the temperature and pressure of the equipment you are using to 70 °C and 5 bar respectively and you find your response which is percentage oil yield

Appendix B: Pictures

B.1: Feedstock Preparation and Pre-processing

B.2 : Weighing of pre-processed tyres

B.3: Microwave Pyrolysis Set Up

B.3: Thermal Pyrolysis Set Up

B.4: Liquid Products from Pyrolysis of Used Tyres

B.5: Weighing Liquid Products

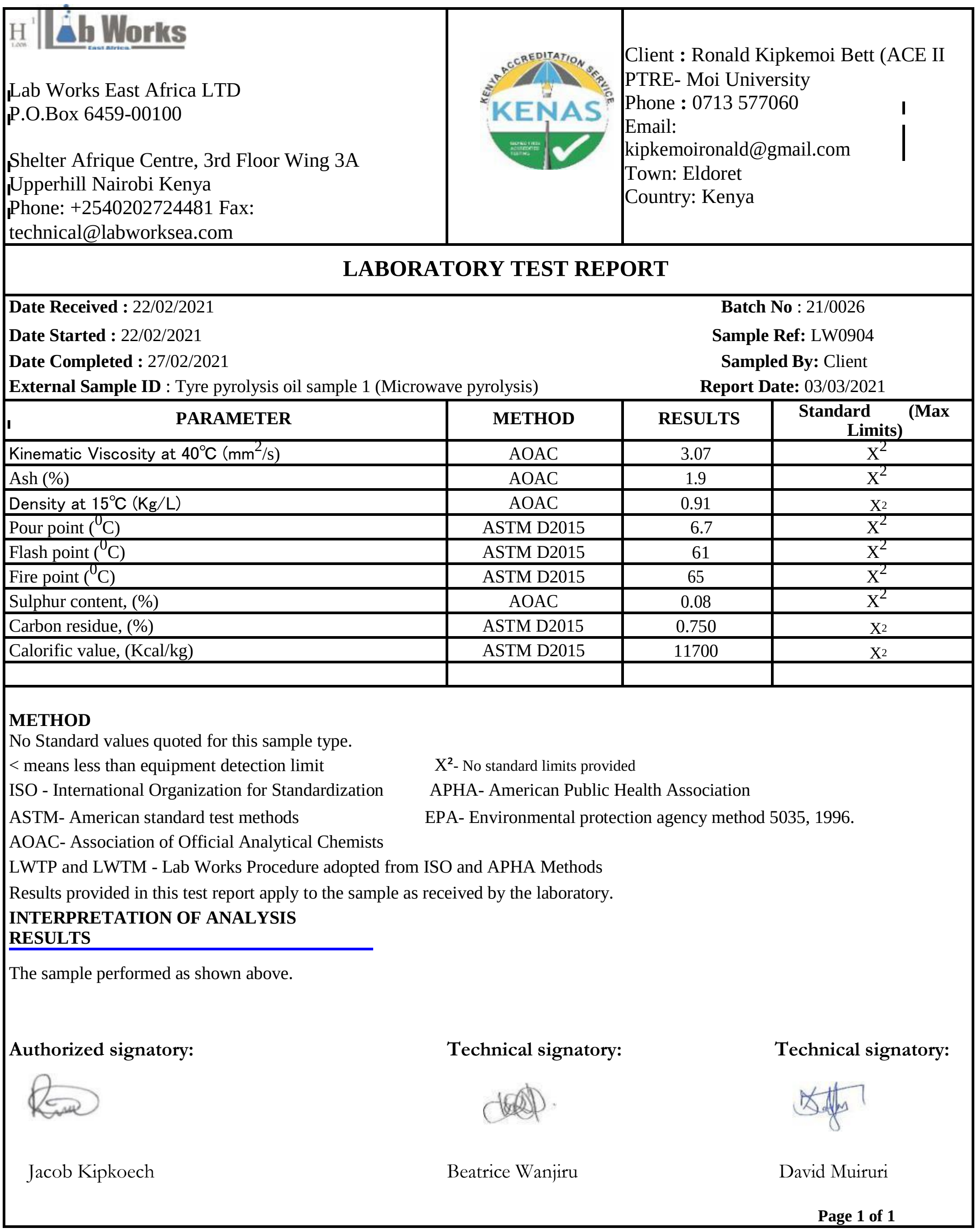
B.6: Solid Residue

B.7: Tyre Pyrolysis Oil for analysis

B.8: Oil from Microwave and thermal pyrolysis ready for analysis

Appendix C: Analysis Reports

Table C.1: Analysis of Optimal Microwave Pyrolysis Tyre Oil



Lab Works East Africa LTD
P.O.Box 6459-00100

Shelter Afrique Centre, 3rd Floor Wing 3A
Upperhill Nairobi Kenya
Phone: +2540202724481 Fax:
technical@labworksea.com

Client : Ronald Kipkemoi Bett (ACE II PTRE- Moi University
Phone : 0713 577060
Email:
kipkemoironald@gmail.com
Town: Eldoret
Country: Kenya

LABORATORY TEST REPORT

Date Received : 22/02/2021
Date Started : 22/02/2021
Date Completed : 27/02/2021
External Sample ID : Tyre pyrolysis oil sample 1 (Microwave pyrolysis)

Batch No : 21/0026
Sample Ref: LW0904
Sampled By: Client
Report Date: 03/03/2021

PARAMETER	METHOD	RESULTS	Standard (Max Limits)
Kinematic Viscosity at 40℃ (mm^2/s)	AOAC	3.07	X^2
Ash (%)	AOAC	1.9	X^2
Density at 15℃ (Kg/L)	AOAC	0.91	X^2
Pour point (^0C)	ASTM D2015	6.7	X^2
Flash point (^0C)	ASTM D2015	61	X^2
Fire point (^0C)	ASTM D2015	65	X^2
Sulphur content, (%)	AOAC	0.08	X^2
Carbon residue, (%)	ASTM D2015	0.750	X^2
Calorific value, (Kcal/kg)	ASTM D2015	11700	X^2

METHOD
No Standard values quoted for this sample type.
< means less than equipment detection limit
X^2- No standard limits provided
ISO - International Organization for Standardization
APHA- American Public Health Association
ASTM- American standard test methods
EPA- Environmental protection agency method 5035, 1996.
AOAC- Association of Official Analytical Chemists
LWTP and LWTM - Lab Works Procedure adopted from ISO and APHA Methods
Results provided in this test report apply to the sample as received by the laboratory.

INTERPRETATION OF ANALYSIS RESULTS

The sample performed as shown above.

Authorized signatory:
Jacob Kipkoech

Technical signatory:
Beatrice Wanjiru

Technical signatory:
David Muiruri

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Table C.2: Analysis of Optimal Thermal Pyrolysis Tyre Oil

Lab Works East Africa LTD P.O.Box 6459-00100 Shelter Afrique Centre, 3rd Floor Wing 3A Upperhill Nairobi Kenya Phone: +2540202724481 Fax: technical@labworksea.com		**Client :** Ronald Kipkemoi Bett (ACE II PTRE- Moi University **Phone :** 0713 577060 Email: kipkemoironald@gmail.com Town: Eldoret Country: Kenya

LABORATORY TEST REPORT

Date Received : 22/02/2021
Date Started : 22/02/2021
Date Completed : 27/02/2021
External Sample ID : Tyre pyrolysis oil sample 2 (Thermal pyrolysis)

Batch No : 21/0026
Sample Ref: LW0905
Sampled By: Client
Report Date: 03/03/2021

PARAMETER	METHOD	RESULTS	Standard (Max Limits)
Kinematic Viscosity at 40℃ (mm^2/s)	AOAC	3	X^2
Ash (%)	AOAC	1.5	X^2
Density at 15℃ (Kg/L)	AOAC	0.91	X_2
Pour point (^{0}C)	ASTM D2015	6.5	X^2
Flash point (^{0}C)	ASTM D2015	59	X^2
Fire point (^{0}C)	ASTM D2015	62	X^2
Sulphur content, (%)	AOAC	0.08	X^2
Carbon residue, (%)	ASTM D2015	0.680	X_2
Calorific value, (Kcal/kg)	ASTM D2015	11300	X_2

METHOD

No Standard values quoted for this sample type.

< means less than equipment detection limit

X^2- No standard limits provided

ISO - International Organization for Standardization

APHA- American Public Health Association

ASTM- American standard test methods

EPA- Environmental protection agaency method 5035, 1996.

AOAC- Association of Official Analytical Chemists

LWTP and LWTM - Lab Works Procedure adopted from ISO and APHA Methods

Results provided in this test report apply to the sample as received by the laboratory.

INTERPRETATION OF ANALYSIS RESULTS

The sample performed as shown above.

Authorized signatory: Jacob Kipkoech

Technical signatory: Beatrice Wanjiru

Technical signatory: David Muiruri

Page 1 of 1

Lab Works East Africa Ltd P.O. Box 6459-00100 Shelter Afrique Centre, 3rd Floor Wing 3A Upper hill Nairobi Kenya Phone: +2540202724481 Fax: technical@labworksea.com		Client: Ronald Kipkemoi Bett (ACE II PTRE- Moi University Phone : 0713 577060 Email: kipkemoironald@gmail.com Town: Eldoret Country: Kenya

LABORATORY TEST REPORT		
GC ANALYSIS		
Date Received: 22/02/2021		Batch No : 21/0026
Date Started: 22/02/2021		Sample Ref: LW0904 & LW0905
Date Completed: 27/02/2021 External Sample IDs: Tyre pyrolysis oil sample 1&2 (Microwave & Thermal pyrolysis)		Sampled By: Client Report Date: 16/03/2021

Table C3: Components detected in sample 1 (Microwave Pyrolysis)

Retention Time (RT) (Mins)	Components	Molecular Formula	Peak Area % for Microwave Pyrolysis
4.94	Ethylenzene	C_8H_8	0.6
5.62	Benzene	C_6H_6	0.92
7.03	Toluene	C7H8	10.65
7.96	2,4-dimethyl-quinolin	C11H11N	0.41
8.82	m-Xylene	C8H10	7.31
11.33	o-Xylene	C8H10	1.40
12.64	Cyclohexane, 1-methyl-5-(1-methylethenyl)-(R)	C10H16	1.10
13.46	D-Limonene	C10H16	20.23
15.59	Naphthalene	C10H8	5.42
15.72	3,4-Dihydro-1-methylnaphthalene	C11H12	2.74
19.44	Naphthalene, 1,2-dihydro	C10H10	1.2
20.00	Naphthalene 1,7-dimethyl	C12H12	1.96
25.09	Naphthalene,2,7 –dimethyl	C12H12	2.50
27.09	1-methylnaphthalene	C11H10	5.80
28.88	Estradiol, 3-deoxy	C18H24O	3.50
30.33	Fluoranthene	C16H10	0.45

Table C4: Components detected in sample 2 (Thermal Pyrolysis)

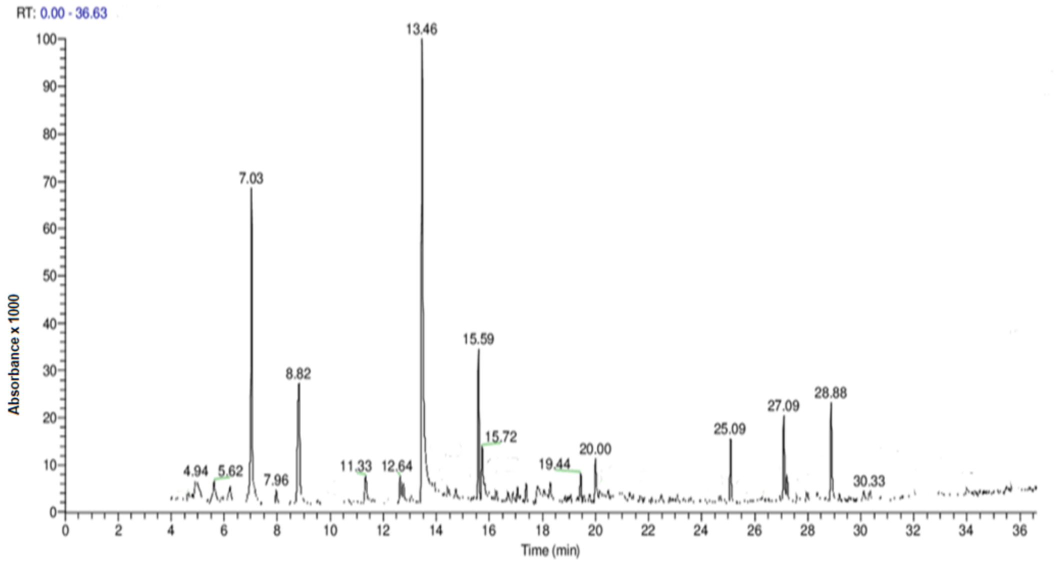
Retention Time (RT) (Mins)	**Components**	**Molecular Formula**	**Peak Area % for Thermal Pyrolysis**
4.90	Ethylenzene	C_8H_8	0.68
5.61	Benzene	C_6H_6	1.00
7.02	Toluene	C_7H_8	7.45
8.83	m-Xylene	C_8H_{10}	6.82
11.33	o-Xylene	C_8H_{10}	1.45
12.63	Cyclohexane, 1-methyl-5-(1-methylethenyl)-(R)	$C_{10}H_{16}$	1.0
13.45	D-Limonene	$C_{10}H_{16}$	14.55
15.60	Naphthalene	$C_{10}H_8$	5.40
15.72	3,4-Dihydro-1-methylnaphthalene	$C_{11}H_{12}$	3.20
16.27	Fluorene	$C_{13}H_{10}$	2.0
19.45	Naphthalene, 1,2-dihydro	$C_{10}H_{10}$	1.75
20.01	Naphthalene 1,7-dimethyl	$C_{12}H_{12}$	2.05
21.70	Acenaphthylene	$C_{12}H_8$	1.01
25.10	Naphthalene,2,7 –dimethyl	$C_{12}H_{12}$	2.32
27.08	1-methylnaphthalene	$C_{11}H_{10}$	4.45
28.88	Estradiol, 3-deoxy	$C_{18}H_{24}O$	2.50
30.32	Fluoranthene	$C_{16}H_{10}$	0.62

Reading and interpretation of GC Results

Specific pure standards of the components to be analyzed were first run in the GC. The retention time these standards were registered in the GC. The pyrolysis oil samples were diluted in an ethanol solvent before analysis and the Tyre Pyrolysis oil to Solvent ratio being 1:10. The GC detector then identified the components in the sample based on the stored information from the standards. It is the Peak Area that determined the concentration of a component in the sample.

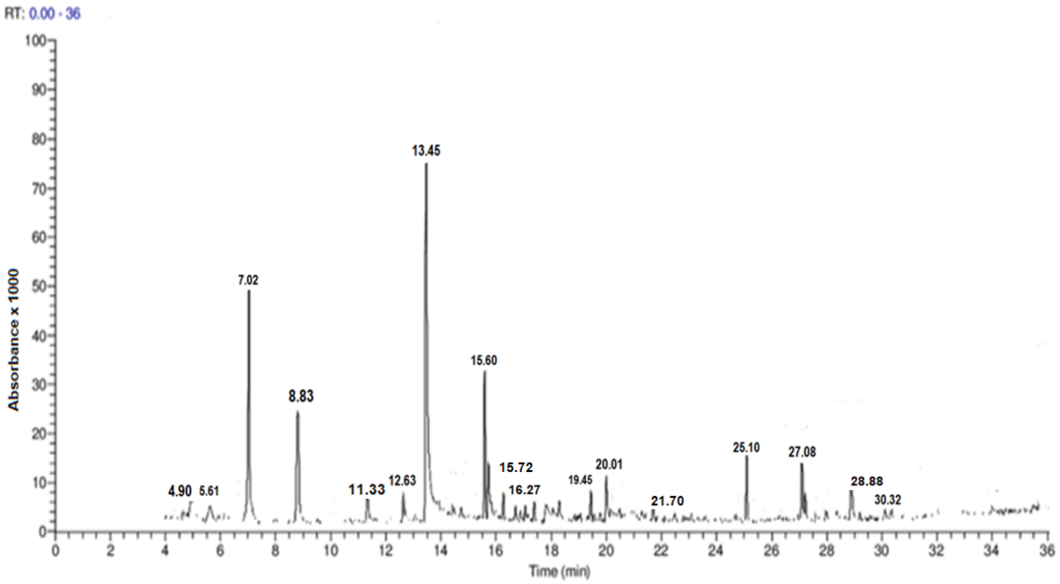
The retention time shows the identity of the component.
The machine used for analysis was Thermo Scientific TM Trace Gold TG-1MS.
NOTE:
RT- Retention Time
Initial temp: 100 ^{O}C
Final temp: 380 ^{O}C
Carrier Gas: Helium
Detector: FID
Column: 19091J-411E (5in cage HP-5 15m x 0.32mm x 0.25um

Chromatogram 1: Microwave Pyrolysis Tyre Oil



The y-axis is the absorbance levels and does not necessarily reflect the concentration of the components. The concentration of the test components is expressed by the Peak Area. The x-axis is the Retention Time.

Chromatogram 2: Thermal Pyrolysis Tyre Oil



The y-axis is the absorbance levels and does not necessarily reflect the concentration of the components. The concentration of the test components is expressed by the Peak Area. The x-axis is the Retention Time.

Authorized signatory:	Technical signatory:	Technical signatory:
Jacob Kipkoech	Beatrice Wanjiru	David Muiruri