

Microwave Pyrolysis of Plastic

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Abstract

Microwave heating has been used in the chemical industry for many years for diverse applications due to advantages, such as volumetric heating, high power density, and fast and easier temperature control. The motivation of this work is to develop a methodology to use potential benefits of microwave as a heat source in plastic pyrolysis. A goal was to use the available knowledge to design and build a rotating microwave reactor, to attain homogeneous temperature distribution in anticipation of producing pyrolysis products with uniform molecular hydrocarbon distribution. Also, it was sought to investigate the effect of process parameters on products' yield and composition, and examine their suitability as fuel and useful chemicals. A rotating microwave reactor was designed and fabricated using a coaxial transmission structure without the limitations of the commonly-used enclosed glass quartz reactor, which made the design appealing for any future industrial-sized microwave reactor.

The optimum pyrolysis operating condition led to the production of a suitable product for a fuel application. However these products cannot be used directly as phase change materials due to their low latent heat and very broad melting range. Fractionation of the products may also be used as phase change materials, capable of storing/releasing heat at suitable application temperature. The microwave heating provided a more uniform heating distribution, although it did not alter product composition in comparison to the conventional pyrolysis of plastic. In the small microwave reactor used in this project, microwave benefit was not evident with regards to its effect in creating uniform temperature distribution. The benefit may be clearer in an industrial-sized reactor in which heat penetration in conventional thermal reactor can be a serious problem.

The pyrolysed products (oil/waxes) were quantitatively analysed using GC/FID. The results of microwave pyrolysis showed 73% oil/wax yield, with silicon carbide used as a microwave absorbent.

Keywords: Microwave reactor; Plastic; Pyrolysis; Gas Chromatography/Flame Ionization Detector (GC/FID)

Introduction

Pyrolysis can be described as a chemical process and thermal decomposition of organic components in an oxygen-free atmosphere to yield char, oil and gas. Microwave heating has been used for pyrolysis of biomass [1-4], scrap tires [5,6], wood [7-9], rubber [10], oil shales [11], coffee hulls [12,13] and production of other chemicals [14-16]; however, there have been few studies of plastic wastes pyrolysis [17-19]. The benefits of the pyrolysis are recycling some of the stored energy within the waste plastics, consequently diminishing imports of crude oil considering limited supply of natural resources (crude oil and gas), fluctuation of high price and availability of crude oil. Pyrolysis of plastic is an endothermic process, so that it requires a supply of heat. Two types of the heat sources can be provided here i.e. conventional heat source and microwave. The conventional plastic thermal decomposition has been established to operate in industrial scale around the world. Using microwave as a heat source would open a new horizon in this topic. Microwave energy can be delivered directly to the reacting or processing species by using their dielectric properties or by adding absorbers to materials which allows more volumetric heating of materials [20,21]. The high heating rate can be several orders of magnitude greater than with conventional heating [20-22]. Microwave generator can respond quickly to changes in process parameters with a feedback loop of an automated process [20]. Microwave heating results from induced currents so the heating tends to be volumetric however the penetration of microwaves is influenced by the properties of the material. The field penetrates it losses power and therefore the field intensity will decrease suggesting that heating may not be uniform. Due to the fact that materials are heated volumetrically, materials with more uniform microstructure can be produced, if energy losses are minimised using insulation [16] and if the material has the proper dielectric loss factor. Advantages of

microwave technology may facilitate moving forward to produce clean, fast and high quality product. Further study should be performed to get a clear picture of microwave pyrolysis of plastic process at high temperature. Challenges such as controlling electromagnetic field and uniformity, temperature measurements may require more sophisticated approaches to be tackled [23]. Albeit in the light of present technique and instrument of temperature measurement, it is not easy to acquire a precise result of the temperature distribution from the interior of the medium at high frequency and high temperature. The only question remains to be answered is whether it is possible to achieve microwave pyrolysis of plastic with uniform heating in much less time comparing to conventional heating on a reasonable scale.

A few studies have outlined the scale up challenges in microwave process [23,24]. To the knowledge of the author there is currently no industrial scale application of microwave pyrolysis of plastic. The reason may be explained by the difficulties of combining the chemical and electrical engineering technologies to meet the requirements for a high temperature microwave processing of plastic degradation. Due to the complex nature of pyrolysis of plastic a very particular and detailed design with the help of a robust electromagnetic simulation model is needed in order to achieve this goal.

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Received December 22, 2012; Accepted February 12, 2013; Published February 14, 2013

Citation: Khaghanikavkani E, Farid MM, Holdem J, Williamson A (2013) Microwave Pyrolysis of Plastic. J Chem Eng Process Technol 4: 150 doi:10.4172/2157-7048.1000150

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Microwave application is very broad. Hence, many studies have been carried out with different designs to investigate the process outcome in various chemical applications. Microwave system that are dedicated to chemical syntheses consist of a microwave power source (generator), a section of transmission line (different type of transmission lines are parallel wires, waveguide, coaxial and micro-strip) that delivers microwaves from the generator into an applicator, and a microwave applicator (reactor). Industrial microwave heating systems typically use a variety of standardised waveguide components (different types of waveguide are circular, rectangular, elliptical or ridge cross section) with specific sizes and necessary functions. The physical size of waveguide determines frequency, bandwidth, power handling capacity and impedance of line. The most commonly used microwave applicators belong to three main types. There are travelling wave applicators, single mode cavity and multimode cavity, depending on physical size and design characteristic. The initial feature of a microwave cavity design is the ability to maximise the electric field intensity which increases the heating rate, operate the experiment at high temperatures over 800°C and allow the removal of pyrolysis products from the cavity [25]. Studies suggested that pyrolysis in a single mode reactor requires an order of magnitude lower input energy comparing to a multimode reactor [25]. This is simply because the electric field density in single mode reactor is high hence higher heating rate is achievable as the heating rate is proportional to the square of the electric field strength (heating rate $\propto E^2$). Despite the stronger field density in a single mode cavity, limitation on physical dimension of the sample due to highly non-uniform field configuration makes the industrial scale usage of a single mode cavity undesirable. The idea of passing the material through the high field intensity of a multimode cavity was a common approach in pilot scale microwave system [26,27].

In 2000 Esveld et al. [26] design a continuous pilot scale (10 to 100 kg/h) multimode microwave reactor and model the unit for dry and solvent free chemistry in esterification reaction (stearic acid with stearyl alcohol). In their study [26] the microwave absorption is improved by adding clay (50 wt%) to the sample. Temperature is measured using a fluoroptic probe within the sample. In the design 4.4 kW microwave power is fed to a multimode cavity by a diagonal slotted waveguide. The materials are closely packed in a Teflon coated glass fiber web conveyor moving at 17 cm/min. To avoid arcing and plasma formation the power is limited to lessen the field density which was 15 ± 4 kV/m measured by a Luxtron MEF probe.

Robinson et al. [27] develop a continuous microwave treatment system using a conveyor system for the remediation of contaminated drill cuttings at pilot scale (500 kg/h of material). In the oil industry drill cuttings are called the broken bits of solid material removed from oil or gas well borehole. The optimum geometry of the applicator is determined based on simulation outcome. Field uniformity is improved in the cavity with design of step positioning at an offset distance from the centre line of the incoming waveguide, a solution which yields both high and uniform power densities across the cavity geometry.

Generally, multimode applicators are commonly used for industrial applications due to greater size and operational flexibility. Acceptable results are achieved by using a multimode applicator for treatment of contaminated drill cutting under high microwave power between 10 to 15 kW [28]. Simulation of the field distribution and power density in microwave treatment of oil contaminated waste in Shang et al. study [29] also yield very useful information in respect of location of maximum electric field strength and dependency of dielectric loss factor and microwave absorption.

Simulation assessment is one of the efficient methods used to achieve the optimum design through a 3D visualisation of field distribution within the applicators and materials. This technique has been used in other studies using available electromagnetic design software [27,29] such as QuickWave-3D[®], COMSOL and Microwave Studio[®] in the market.

This paper was devoted to the microwave pyrolysis of High-Density Polyethylene (HDPE) for the production of fuel and other useful chemicals. In this research the design for a microwave unit with the use of a coaxial structure based on the simulation model was suggested. This was followed by the electromagnetic simulation conducted on the microwave unit, providing some insight into microwave behaviour with regards to energy dissipation and electromagnetic wave distribution. The microwave adjustments-including the building, tuning and calibrating of the microwave-induced pyrolysis unit-were then discussed, together with setting up the unit for operation. The experiments were then described and the factors influencing various operating conditions on pyrolysis products were explained. After that, condensable products were analysed quantitatively using GC/FID and Differential Scanning Calorimetry (DSC) in order to characterise their properties.

Materials and Methods

In this study HDPE in a pellet form with diameter of 2 mm to 3 mm was used with carbon blocks in a cubic form with dimensions of 30 mm×30 mm×30 mm as a microwave absorbent. Alternatively silicon carbide powder was also utilised.

In this study Microwave Studio[®] (MWS) was utilised to predict microwave field distribution, intensity and absorbed microwave power inside the waveguide and applicator. This tool can also help to optimise the unit dimensions in order to minimise the reflected power. MWS is powerful and easy to use software for high frequency range however it does not permit the evaluation of temperature dependent thermal properties or chemical phenomena such as latent heat or heat of reaction.

Experimental Apparatus

An experimental apparatus was designed, built and used (Figure 1)

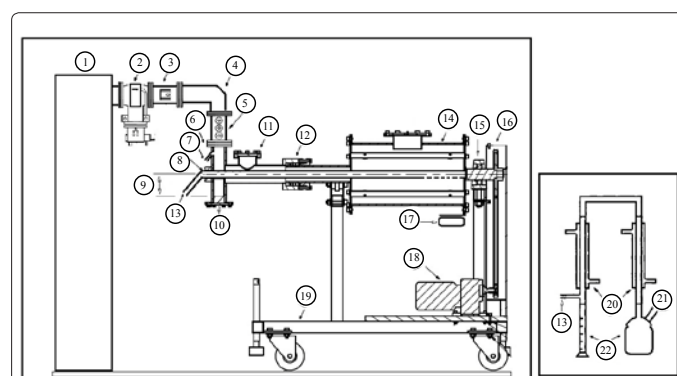


Figure 1: Schematic of the microwave pyrolysis of plastic apparatus in rotation mode

1) Generator, 2) Isolator (Circulator + Dummy load), 3) Directional coupler, 4) Bend, 5) 3-stub tuner, 6) Quartz window, 7) Nitrogen inlet, 8) Central conductor, 9) Step height, 10) Step, 11) Rupture disk, 12) Gland, 13) Product outlet in microwave unit and product inlet in condensation system, 14) Reactor (Applicator), 15) Bearing, 16) Gear transition, 17) Wireless temperature data logger, 18) Adjustable speed electrical machinery, 19) Stand, 20) Condensers, 21) Outlet of non-condensable gases, 22) Collection flasks.

based on the electromagnetic field simulation results which will be discussed below. The three main equipment components of the system were (a) the microwave generator (number 1), (b) the wave guide components (numbers 2 to 6), and (c) the applicator (number 14). The microwave generator was built by Keam Holdem Ltd. with a maximum of 6 kW output power operating at 2.45 GHz. The transmission line consists of the following parts:

1. An isolator consisting of a circulator and a dummy load, which was purchased from Richardson Electronics Pty Ltd.
2. A directional coupler with power readout devices, which was used to detect the reflected and forwarded power in the wave guide.
3. A three stub tuner, which was located close to the load in the waveguide section to tune the unit and minimise the reflected power in order to maximise the heating in the reactor.
4. The aluminium rectangular waveguide (WR340) with the dimension of 86.36 mm×43.18 mm which was constructed in the University of Auckland.
5. A quartz window (number 6 in Figure 1) was fixed between the flanges to keep the transmission line clean and protect the generator from the produced gases. Sealing around the glass quartz was very important and quite tricky, as it was necessary to prevent leakages of gases both into the generator and the atmosphere. To prevent any breakages, the quartz window was surrounded by a metal frame designed to protect it from cracking. Enough clearance was left for both the glass and metal frame expansion during the high temperature experiments. Two gaskets were placed above and beneath the metal frame to cease any leakages. And the gaskets edges were covered with copper shim to prevent arcing and provide good electrical contact.
6. A step was attached to the end of the waveguide flange as shown in figure 1 with number 10. In order to ensure good contact between the step and waveguide's walls a row of beryllium copper teeth (0.5 mm thicknesses) were designed. The teeth (long fingers) touched the wall in such a way that they eliminated gaps and unexpected resistance between the metals' surfaces, so as to provide an effective electrical connection between the step and the waveguide.
7. A gland was designed to connect the rotated and fixed sections in the unit. Pure expanded braided graphite ring packing suitable for high temperatures (Uni-Pac1 compression packing with 98% purity from Novus sealing limited) was used to provide proper gas sealing and a good electrical contact.

In technical terms the cylindrical cavity (reactor) is a multimode resonator, used for batch processing. The reactor used was made of stainless steel (304) to accommodate high temperature process. The reactor was sized for the multimode type of the applicator in which the dimensions (length 39 cm and diameter 22 cm) were somewhat greater than the wavelength (12.2 cm). Increasing the dimension with respect to wavelength increases the number of excited modes in the cavity which has proved to be helpful in providing better heating uniformity.

The reactor was designed to rotate around its horizontal axis and had four longitudinal baffles bolted to the interior reactor wall in order to help the mixing process. Initially, the reactor was not rotated, therefore, for simplification; gaseous products were taken out via the top flange

of the reactor. In this study 10 experiments were conducted. All the experiments from numbers 1 to 8 were conducted in stationary mode of the reactor. However, both experiments 9 and 10 were conducted while the reactor was rotated. In this design the central conductor of coaxial had six holes in order to collecting the products from the microwave reactor during the reactor rotation. The coaxial structure provided flexibility and allowed for the collecting of the products and nitrogen passes through the unit. The schematic of the microwave system is shown in figure 1.

The reactor was filled and discharged via the top flange with HDPE pellets and microwave absorbents of either carbon blocks or silicon carbide (SiC). The reactor was operated under inert atmospheric pressure using nitrogen. In all experiments, the pyrolysis reactor was heated up to temperatures between 400°C and 550°C. In this thermo chemical process, plastic was pyrolysed into gases with no residue. The waxy liquid fraction of the gases products was collected using two glass coil condensers supplied by Technical Glass Products Limited. The temperature of the first condenser (coil length 250 mm, body length 320 mm, joints NS 14/23) was set at 60°C while cold tap water (17°C) was used as the circulating water in the second condenser (coil length 150 mm, body length 220 mm, joints NS 14/23). A small amount of waxy product was also collected from the interior parts of the unit when it cooled down. Non-condensable gases were vented without analysis, as their analysis was not part of this investigation. These gases are usually used as a source of the energy needed for the pyrolysis process. The liquid portion of the pyrolysed products can be used as a fuel source and to produce valuable chemicals with a high calorific value of 46.5 MJ/kg [30].

The central conductor was a hollow carbon steel tube (SEA 1045) with a diameter of 25.5 mm. A coaxial transmission line was utilised in this design to transmit energy from the associated electric and magnetic fields transverse to the axis of the line; this is illustrated in figure 2. Following this figure, the electric field vectors are normal to the inner and outer surfaces of the conductors. It also shows the magnetic field as a series of concentric circular vectors between the two conductors.

Simulation

The model structure for simulation of the microwave reactor discussed earlier was built using MWS as shown in figure 3. Microwave Studio® like other simulators follows three simple steps to perform calculation; firstly, model the geometry, secondly, run the solver to solve Maxwell's equations and lastly, plot the parameters. In this simulation the model's geometry was constructed by defining a cylindrical multimode applicator with the sample located inside the reactor, a coaxial transmission line including central conductor and outer conductor, waveguide structure, boundary conditions and excitation

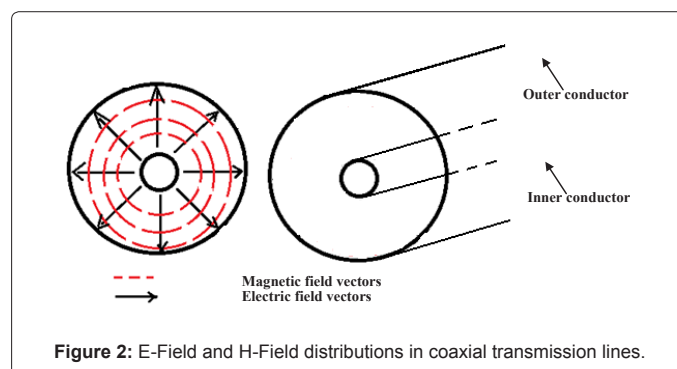


Figure 2: E-Field and H-Field distributions in coaxial transmission lines.

port. The material properties such as dielectric constant, loss tangent, density, thermal conductivity and specific heat capacity were assigned accordingly.

Physical properties

Electrical characteristic of the materials such as dielectric constant (real part) and dielectric loss factor (imaginary part) are terms used to specify the electromagnetic properties of material. Dielectric constant (ϵ') represents the stored energy and loss factor (ϵ'') represents the lost energy in dielectric materials. Dielectric loss tangent ($\tan \delta$) is a term to quantify lossiness which defines the ability of materials to absorb and convert electromagnetic energy into heat at a specified temperature and frequency. The lossiness is the ratio of energy lost to energy stored in the dielectric material. The dielectric loss factor of plastic is too low to achieve effective heating using microwave energy, therefore the ratio of absorbent (carbon or silicon carbide) to plastic (HDPE) should be high enough to be able to pyrolyse the plastic in a multimode applicator. Two different ratios of 10 (5 kg of absorbent to 0.5 kg plastic) and 3.3 (1.66 kg of absorbent to 0.5 kg plastic) were assumed. The thermal physical properties of the mixture were calculated using the weight factor, as shown in table 1, for one of the simulation case studies.

The dielectric properties of the mixture were calculated using equation 1 which is known as the Landau & Lifshitz, Looyenga mixture Equation [31].

$$(\epsilon_m)^{1/3} = v_1(\epsilon_1)^{1/3} + v_2(\epsilon_2)^{1/3} \quad (1)$$

Where ϵ_m represents the complex permittivity of the mixture, ϵ_1 is the permittivity of the medium in which particles of permittivity ϵ_2 are dispersed, and v_1 and v_2 are the volume fractions of the respective components, where $v_1 + v_2 = 1$.

By substituting dielectric properties of plastic ($\epsilon_1 = 1.95911 - 0.00027j$) and carbon blocks ($\epsilon_2 = 2.35469 - 0.09584j$) in equation 1 with 5 kg of carbon and 0.5 kg of HDPE, the mixture dielectric properties will be $\epsilon_m = 2.283 - 0.08j$ with a loss factor of 2.283, a dielectric constant of 0.08 and a tangent delta of 0.035.

Simulation results

The aim of conducting the simulations is to determine the correct

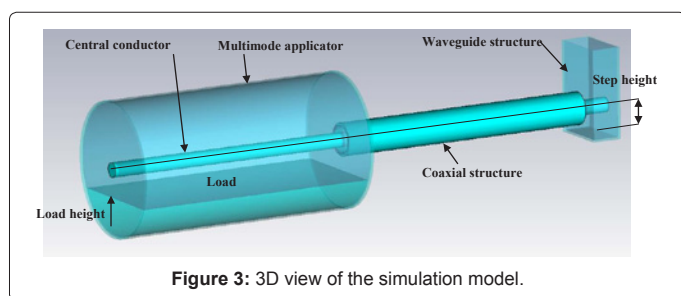


Figure 3: 3D view of the simulation model.

Property	HDPE (7 wt%)	Carbon (93 wt%)	property of the mixture	Mixture
Density (kg/m ³)	960	2185	$\rho_m = 0.93 \rho_c + 0.07 \rho_{HDPE}$	2099
Thermal Conductivity (W/m.K)	0.25	0.14	$\frac{1}{k_m} = \frac{0.93}{k_c} + \frac{0.07}{k_{HDPE}}$	0.1444
Heat capacity (J/kg.K)	2200	1000	$C_{pm} = 0.93 C_{pc} + 0.07 C_{pHDPE}$	1084

Table 1: The physical properties of the samples used in Microwave Studio® (5 kg of carbon + 0.5 kg of HDPE).

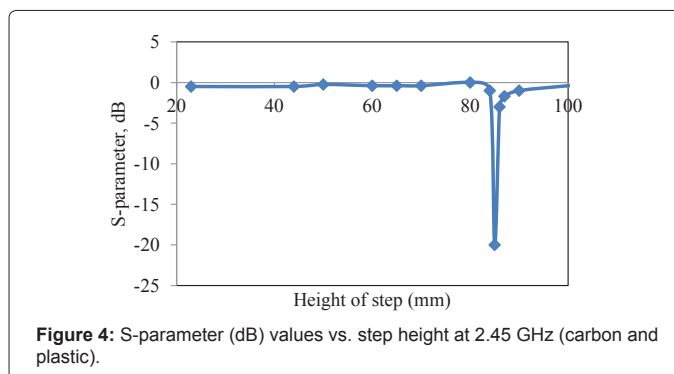


Figure 4: S-parameter (dB) values vs. step height at 2.45 GHz (carbon and plastic).

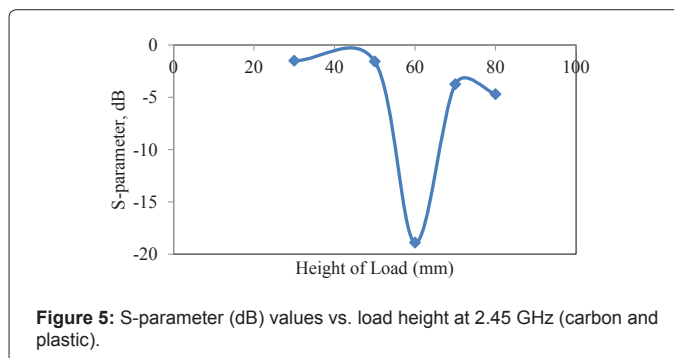


Figure 5: S-parameter (dB) values vs. load height at 2.45 GHz (carbon and plastic).

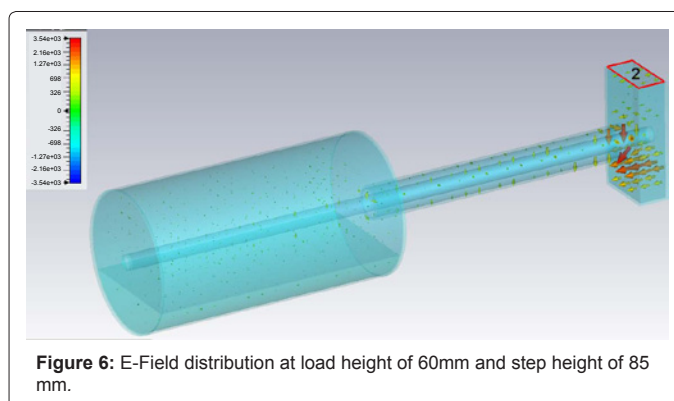


Figure 6: E-Field distribution at load height of 60mm and step height of 85 mm.

dimensions of the unit. Electromagnetic simulation is a useful theoretical way to design a microwave system and investigate the effect of material properties and system geometry [27,29]. This method was followed by optimising the step and load height. Step height indicates the distance between the wave guide flange to coaxial transition as shown in figure 3. The height of the step is one of key factors to minimise the reflected waves (maximise the S-parameter) and couple the impedance of sample with microwave source. S-parameter is a measure to identify how much energy is gained (or lost) in the system at given frequency and system impedance, and varies as a function of frequency. The simulation was run with different step heights. The sharp and high absorption in figure 4 designated the high sensitivity of the absorption in relation to the step height. It was evident that the lowest reflection (the highest absorption) was achievable at the step height of 85 mm. Afterwards the simulator was run to determine the optimised amount of sample. The best absorption took place at 60 mm load height as shown in figure 5.

Figure 6 and 7 show 3D views of electric and magnetic field patterns. It is evident that the intensity of the fields was maximised in the

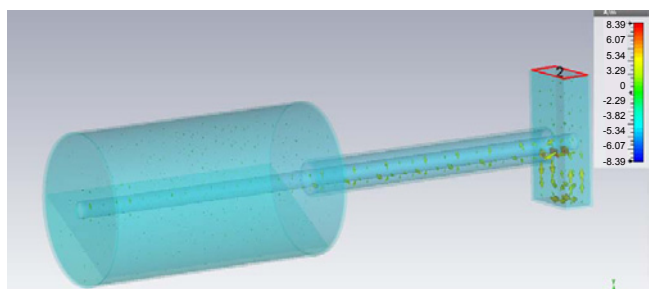


Figure 7: H-Field distribution at load height of 60 mm and step height of 85 mm.

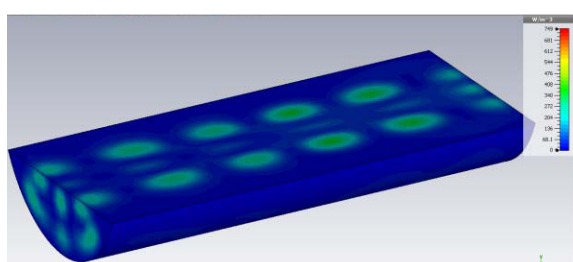


Figure 8: Power loss density within the sample in pyrolysis of HDPE using SiC as an absorbent.

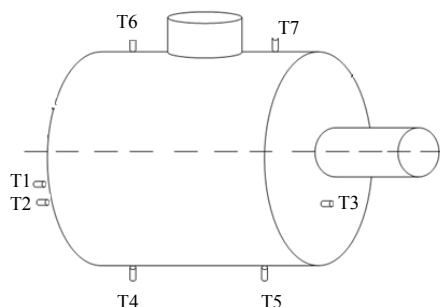


Figure 9: Location of the thermocouples on the reactor.

waveguide and decreased towards the kiln. Likewise the field intensity was maximised at the centre and decreased towards inner walls of the applicator. The field line patterns showed the existing intensity within the waveguide. Figure 8 illustrates the power loss density distribution in the sample in the stationary mode with scattered hot spots throughout the sample. This indicated a non uniformity in the heating process as the materials which were not in the hot spots will not be subjected to the same degree of microwave treatment [27]. It was expected that the scattered hot spots provide an efficient heating process due to a continuous mixing of material while the reactor rotated. Thereby, the located materials in the hot spots will be replaced by new cold materials repeatedly. This fact improves the heat transfer within the sample effectively and eliminates presence of the temperature gradient in the cavity. Furthermore, the changes of sample angle due to rotation have no sensible effect on power loss density variation within the sample based on the simulation outcome.

Operating procedure

The unit was assembled properly by placing the gaskets and quartz

window to isolate the waveguide and coaxial structure. Then it was calibrated and tuned after loading the reactor. The reactor was loaded with desirable amount of absorbent and then filled with a layer of plastic pellets on top. This arrangement of the sample loading allows the sample sink in the space between carbon blocks or absorbent pores in case of using SiC while it melts. Nitrogen was purged to provide the inert atmosphere in the unit before and during the experiment. Nitrogen was purged in the waveguide structure (from number 8 in Figure 7) for two reasons; firstly, to make sure of keeping the quartz window clean from any possible generated dust in the reactor and, secondly, to keep dust out of the waveguide and coaxial structure. The waveguide and coaxial both have much higher field intensity compared to the reactor due to their smaller dimensions. Afterwards access port holes were checked and sealed. Gas and microwave detectors were used to detect any gas and microwave leakage from the unit for safety matters. Heat accumulation can be identified by temperature rises with time very shortly after putting on the power. Plastic decomposition took place at above 300°C. Efficient coupling and controlled heating was achieved via tuning and power control. The three-stub tuner was used to maximise the amount of applied power absorbed by the sample with matching the impedance of the transmission line to the impedance of the load. Gaseous products were sent to the condenser system to condense the heavier components of the pyrolysed gases. The detail of tuning procedure and temperature measurement are explained below.

Calibration and tuning procedure

A network analyser (ENA_E5062A) was utilised to do the calibration and the initial tuning. The network analyser is an instrument that measures the electrical network parameters such as reflection (S-parameters) and transmission of electrical networks at high frequencies. Firstly, the unit was calibrated using three known standards, (a) zero-short, (b) offset-short, and (c) waveguide horn. Secondly, after loading the reactor with the desired amount of material, network analyser was connected to the waveguide just above the three-stub tuner. Then the calibration results based on standards were used to characterise the sample behaviour within the reactor using network analyser. The heights of the three-stub tuner's pins were adjusted in order to minimise the reflection at 2.45 GHz. Three markers were set at three frequencies, i.e. 2.44 GHz, 2.45 GHz and 2.46 GHz. These frequencies were set because 2.45 GHz is the nominal frequency and it is not expected that the operating frequency will drift by more than 10 MHz. This procedure allows us to provide efficient coupling at the starting point. Tuning the microwave unit is essential to achieve the best use of energy, protect the generator and also prevent high reflection and subsequent possible operating issues such as undesired overheating of the structure. However, during pyrolysis continuous tuning was required by adjusting the three-stub tuner pins height due to load phase changes. Changes of material volume within the cavity impact the electromagnetic field distribution and the power dissipation. It is worth noting that water has a high dielectric loss factor, therefore, relatively small variation in the sample's moisture contents would affect the final attained temperature during microwave heating [32]. For this reason calibration before each experiment was required to minimise the effect of daily fluctuation in moisture in the sample.

Temperature measuring system

Temperature measurement in microwave heating is highly controversial. Generally, three methods are suggested to do the measurements in microwave process. Choosing the right method depends on operating condition, material type, size and the

limitation of measurement technique. These methods are optical fibre thermocouple, infrared thermography (IR) and conventional thermocouple. Each one has some advantages and drawbacks which reveal the suitability of the chosen method. The detailed discussion of different aspects and available measurements methods are thoroughly discussed in [33]. The thermocouple type K with or without protected sheaths in microwave heating causes some errors [34] due to provoking problems such as field distortion, enhanced energy absorption (leading to thermal runaway), heat loss through conduction particularly when it comes to measurements in low loss materials. However, in other studies by Janny et al. [35] and Haque [36], thermocouple type C [35] with molybdenum sheath or type K thermocouple with an unground tip sheathed in Inconel 702 sheath [36] are suggested to perform the measurements within the sample, which is explained by accepting that microwaves cannot penetrate the metal shield. The majority of investigations agree to use fibre optic probes and pyrometer/IR sensors in microwave heating [20,34,37]. Optical pyrometers as a non-contact technique eliminate the interference in electrical field behaviour while they only record surface temperatures. The high temperature and strong agitation do not allow the use of fibre optics.

In the current study the temperature profile was logged continuously throughout the process using K type thermocouples inserted a certain short length (25 mm) inside the reactor. The reasons for choosing short length are, firstly, to eliminate the possibility of interference of the electromagnetic field by presence of K type thermocouple, and, secondly, over an extended specific length they could be burnt. The reason for overheating can be explained by induced currents at the thermocouple tip where currents are less but without the ability to get rid of heat on the stainless steel thermocouple sheet, because the stainless steel is a fairly poor conductor of both heat and current. The specific length of thermocouple in the reactor can be determined experimentally by trial and error. However, the temperature further inside the sample towards the central conductor is expected to be higher than the reactor wall as there is non-uniform electromagnetic field distribution in the reactor and field intensity increases towards central conductor. Figure 9 shows the mounted thermocouples in the reactor. A wireless data logger (WLS-TC) was utilised to monitor the temperature.

Analysis

All oil/wax products were analysed with the use of GC/FID and DSC for their compositions and thermal properties. In this study the reason for conducting DSC analysis is to determine the thermal characteristic (latent heat and melting range) of the products and investigate the suitability of the pyrolysed products for use as Phase Change Materials (PCMs). PCMs are capable of storing and releasing large amounts of energy during the phase changes. The scope of PCMs application is very wide and melting temperature of the material determines their suitability for the application. One of these applications is solar energy storage in building by encapsulating suitable PCMs. An ideal candidate for PCMs application should have proper melting ranges, large latent heat of melting, high thermal conductivity, high specific heat capacity, small volume change, and should be non-corrosive, non-toxic and exhibit little or no decomposition or super-cooling. Commercial paraffin waxes are cheap with moderate thermal energy storage density (200 kJ/kg) [38,39]. The paraffin waxes are straight chain hydrocarbons, similar to those products from pyrolysis plastic.

GC/FID

Oil/wax products were analysed using a Shimadzu GC-17A with flame ionisation detector, 0.53 mm tubular capillary column, 6 meters

length (MXT-500 Sim Dist Cap) and split injection (split ratio of 0.5) with a volume of 1.0 μ L. The injector and FID temperatures were 325°C and 395°C respectively and the oven was held at 50°C initially, then ramped to 380°C at 15°C/min. The column pressure was 3.6 kPa, which corresponded to a linear carrier gas (helium) velocity in the column of 25.5 cm/s. The total and column flows of helium were 5.7 and 3.17 ml/min respectively. Calibration was achieved using Restek Florida standard, which is comprised of even numbered n-alkanes from C₈ through to C₄₀. Alkenes and dienes could then be determined from their proximity to their corresponding n-alkane. Carbon disulphide was utilised as the solvent, due to its superior ability to dissolve heavier compounds. This was found by a preliminary solubility test performed on the collected samples from the unit before starting GC calibration. The GC would first be flushed once with pure solvent after each sample run to ensure all residues from the previous run was removed. Each sample was analysed 3 times to check for repeatability and consistency in the result.

DSC

The DSC (Shimadzu DSC-60) was calibrated using n-octadecane. Approximately 4.8-5 mg of sample was first melted at 80°C to achieve good contact with the aluminium pan surface, and then cooled beyond -135°C at low heating rate of 3°C/min. Measurements for all products (the first condenser, the second condenser and collected wax from the interior parts of the unit after cooling down) were then taken from -135°C to 130°C. The reason for starting from very low temperature was associated with the melting point of low carbon number of wax products i.e. -118.85°C for C₇ or -81.35°C for C₉. Therefore in order to get the correct base line in DSC curves, taking measurements from very low temperature is recommended.

Results and Discussion

The objective of the ten experiments conducted is to focus on the microwave pyrolysis reaction, through investigation of composition and yield under different operating procedures. Different nitrogen flow rates, absorbent type and mass ratios of plastic to absorbent, as well as the reactor in stationary and rotational mode with low and high rotation speed, were also investigated. The summary of the operating conditions is presented in table 2. The condensable gases were collected in waxy form via the condensation unit, and non-condensable gases were ventilated. The oil/wax products were then analysed using GC/FID and DSC to characterise their composition and heat flow.

Experiment 1 was designated as a trail run. In the first three experiments (Exp_2 to Exp_4) 0.5 kg HDPE was pyrolysed using 5

	Absorbent type & input mass	Mode	N ₂ Flow rate (L/min)	Power (kW)	Duration (min)	Total Oil/wax Yield
Exp_1	Carbon (5Kg)	stationary	0-5.12	3-5	66	63.75
Exp_2	Carbon (5Kg)	stationary	0	5	88	54.92
Exp_3	Carbon (5Kg)	stationary	1.28	5	86	62.02
Exp_4	Carbon (5Kg)	stationary	9.60	5	86	58.24
Exp_5	SiC (5kg)	stationary	6.40	5	71	43.40
Exp_6	SiC (5kg)	stationary	1.28	3-5	73	34.67
Exp_7	SiC (1.66)	Stationary (failed)	3.81	3-5	31	37.32
Exp_8	SiC (1.66)	stationary	3.81	3-5	100	39.27
Exp_9	SiC (1.66)	Rotation (4rpm)	3.81	3-5	78	46.36
Exp_10	SiC (1.66)	Rotation (8-18rpm)	2.56	3-5	60	73.36

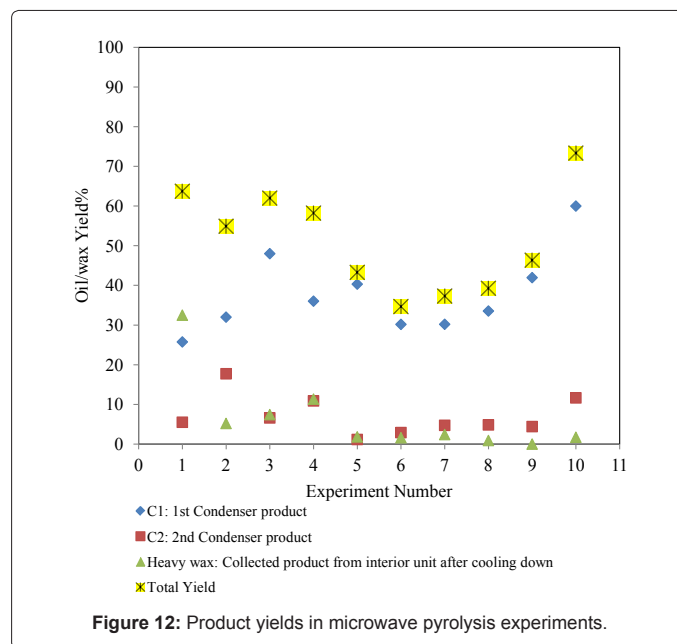
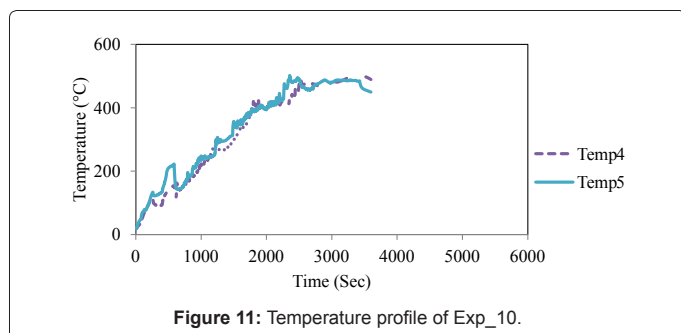
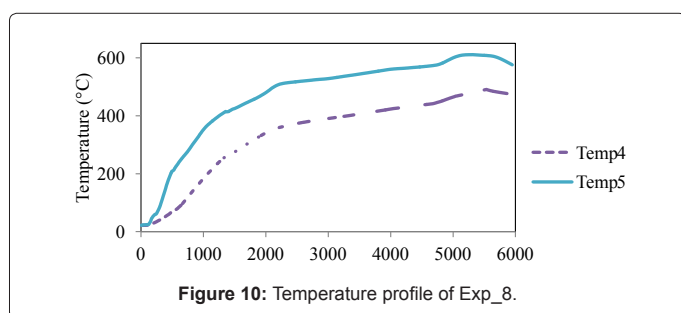
Table 2: Summary of experiment conditions.

kg carbon with different nitrogen flow rates and input microwave power of 5 kW. For the rest of the experiments (Exp_5 to Exp_10) silicon carbide was used as the absorbent according to the tabulated operating conditions. In the experiments with silicon carbide (6 to 10), the temperature was controlled by adjusting the power. The aim was to keep the temperature between 400°C and 500°C, as this temperature range is the optimum suggested range in the literature [40,41] to maximise the lighter liquid fraction. Hence, the input power was decreased when the average temperature in the reactor reached the proximity of 500°C. Experiment 7 was failed half way through the run due to leakage from the thermocouples; the run was stopped for safety concerns. The same operating conditions were repeated in experiment 8, however the products of the failed run (Exp_7) were still analysed and the results were reported. Also, in experiment 9 the rupture disk ruptured and it took about 25 minutes to remove the blockage and replace the aluminium foil. For this reason, during this period prior to commencing the product collection, the temperature profile showed a downward trend. However, the time wasted in experiment 9 has not been factored into the experiment duration. The reason for the rupture failure was the blockage of the condenser inlet by the travelled silicon carbide from the reactor during the reactor rotation. The effect of lower input power can be seen from the temperature profiles, with the recorded decline of the heating rates in experiments 7, 8 and 9. The reactor was rotated only in the last two experiments.

Figure 10 shows the temperature profile in experiment 8 (stationary mode) where the thermocouples 4 and 5 immersed in the sample. It is evident that there was a temperature gradient in the reactor. The temperature decreased from the microwave feeding location towards the far end of the reactor. In all stationary mode experiments (experiments 1 to 8) the temperature gradient was observed while very uniform temperature profile was monitored in experiment 10 when the reactor was rotated with high rpm (Figure 11).

Product yield

The yield of the products was calculated based on mass percentages



of collected oil/wax products relative to the initial mass of plastic feed. The main portion of the product was collected from the first condenser in the form of oil/wax and the second abundant product was collected from the second condenser in a liquid form. A waxy residue was collected from the interior part of the unit after it cooled down. A smaller amount of waxy residue was obtained when using SiC as an absorbent and a larger amount when carbon was used. This suggests the possibility of higher cracking in plastic using SiC, which is a more efficient microwave absorbent.

From the reported data in figure 12 and table 2, it is evident that the total yield of products in the experiments using carbon (experiments 1 to 4) was around 60%, while in the experiments with the SiC the yield was around 40% (more cracking to non-condensable gases). The sole exception to this was the last experiment, which had very different operating conditions, as will be explained later in this section. Pyrolysis is a very temperature dependent process, so temperature plays the most crucial role in product yield and composition. Other factors such as the heating rate and nitrogen flow rate are important because these parameters influence the residence time. The residence time effect on the pyrolysed product yield and composition were discussed in the context of conventional thermal pyrolysis in reference [42].

The high gas yield results (Exp_5 to Exp_9) can be attributed to the use of silicon carbide as a microwave absorbent and its electrical physical properties (high loss factor) and thermal physical properties (high thermal conductivity). As mentioned earlier, in the experiments with silicon carbide the temperature was kept between 400°C and 500°C by controlling the input microwave power. This approach was chosen based on the decomposition mechanism of polyethylene, which initially requires a high temperature for breaking the polymer chain to overcome the bound-dissociation energy of carbon double bonds. Later the heating rate was kept lower than a specific value to extend secondary reactions. Furthermore, the volumetric nature of the microwave heating, coupled with dielectric materials such as SiC, led to the pyrolysis temperature being reached in a very short time (mostly less than 7-10 minutes), consequently exposing the pyrolysed products to heat for longer periods than in

the experiments using carbon (over 35-40 minutes). This fact caused the severe decomposition of the polymer chain, resulting in a high portion of gaseous products.

The operating procedure in Exp_10 was very different from the rest of the runs. In this experiment the reactor was rotated with high rpm causing severe mixing and creating very uniform temperature distribution within the whole sample and, subsequently reaching the pyrolysis temperature of the whole sample at once. Having heating rate of 10°C/min and also, a high nitrogen flow rate led to shorten the pyrolysis duration and caused less cracking without significant secondary decomposition. This fact may justify the obtained high oil/wax yield in the Exp_10.

Composition of the products

Generally, the pyrolysis products of HDPE were hydrocarbons ranging from C₁ to C₆₀. In this study only condensable products, namely oil/wax, were analysed using GC/FID. The pyrolysis operations led to the production of hydrocarbons in the range of C₁₀-C₃₅ with a maximum concentration at C₁₉, which approximately represented the collected oil/wax from the first condenser. Figure 13 shows the total composition of the product from the first experiment. The second condenser collected a very light fraction mostly in the range of C₈ to C₁₅ (close to kerosene fraction which is within C₁₀-C₁₈ range) with a maximum concentration of C₉. A heavy fraction was detected in the collected waxy residue from the interior part of the unit with the bulk of hydrocarbon in the range of C₂₀-C₄₀ peaking at C₃₀. This product is named wax in figure 14a. The products of both condensers and also the residue waxes were analysed separately using GC/FID and afterwards the total compositions were calculated based on yield of each fraction. Figures 14a and 14b show the composition of the individual collected products and the GC chromatograms of used standard sample with the pyrolysis products from the first experiment. Similar trend was obtained in the rest of the experiments.

The objective of microwave experiments was to investigate if microwave pyrolysis leads to different product distribution and recognise the suitability of the products for use as fuel. As it is known

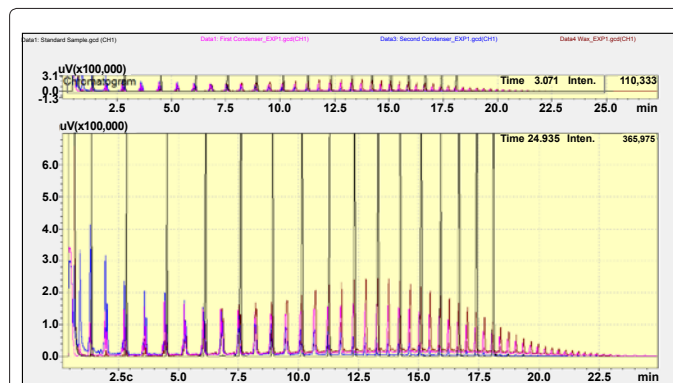


Figure 14b: GC chromatograms of used standard sample and pyrolysis products of Exp_1.

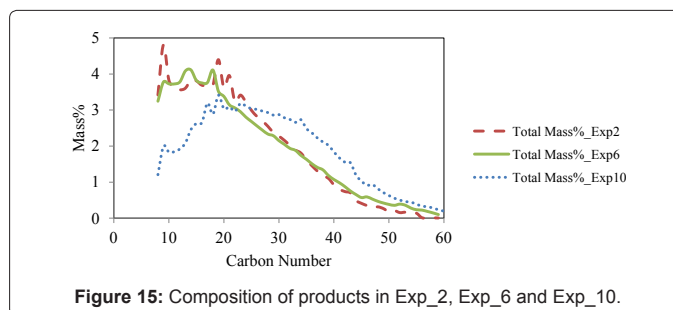


Figure 15: Composition of products in Exp_2, Exp_6 and Exp_10.

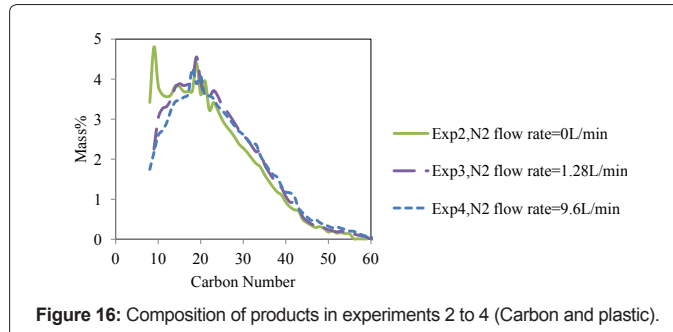


Figure 16: Composition of products in experiments 2 to 4 (Carbon and plastic).

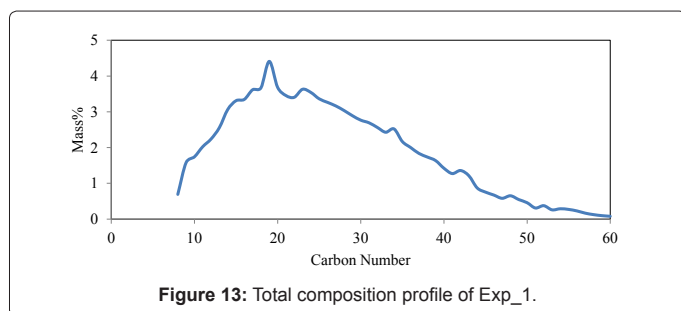


Figure 13: Total composition profile of Exp_1.

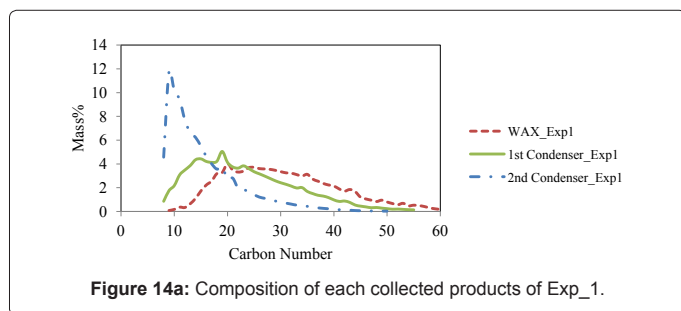


Figure 14a: Composition of each collected products of Exp_1.

the approximate carbon range of refinery fuels are [43]: Petrol C₅-C₁₂ (~C₈), Kerosene C₁₀-C₁₈ (~C₁₅), Diesel C₁₅-C₂₅ (~C₂₀). This goal was achieved in the conducted experiments using both SiC and carbon. Based on analysed compositions the lightest fractions were detected in Exp_2 using carbon and in Exp_6 using SiC with the bulk production in the C₈-C₂₀ range because of using a low nitrogen flow rate. Findings from this study show that the pyrolysis products with potential feedstock usage [44] were placed in the range of hydrocarbon series with the following application [43]; fuel for cars (~C₈), raw material for chemical and plastic (~C₁₀), fuel for aeroplane (~C₁₅), fuel for cars and lorries (~C₂₀), fuel for power station or lubricant or grease (~C₃₅), road surfing (~C₄₀). The heaviest product composition was obtained in the last experiment (Exp_10). The rest of the runs (Exp_1 to Exp_9) produced hydrocarbon ranging between the two lightest fractions (red or green line in Figure 15) and heaviest fractions (blue line in Figure 15).

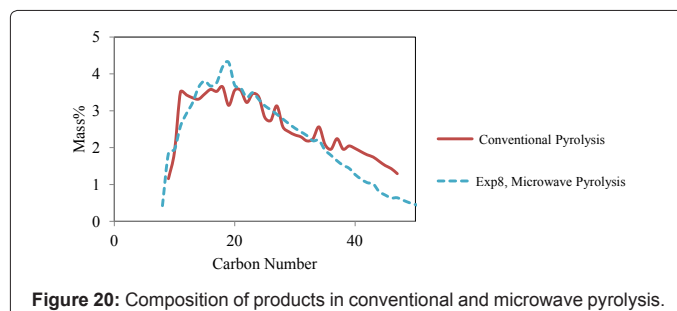
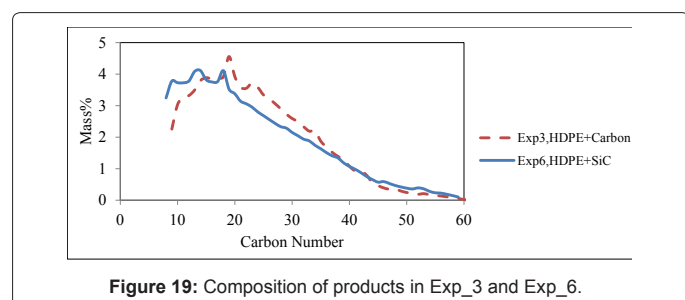
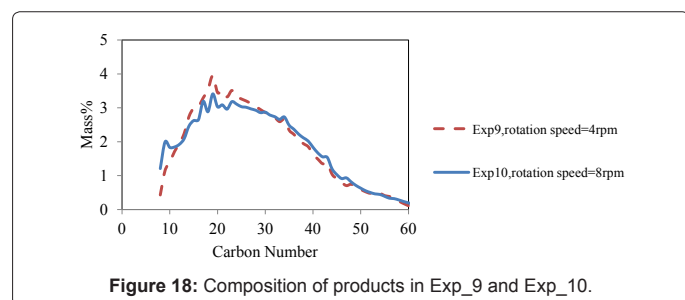
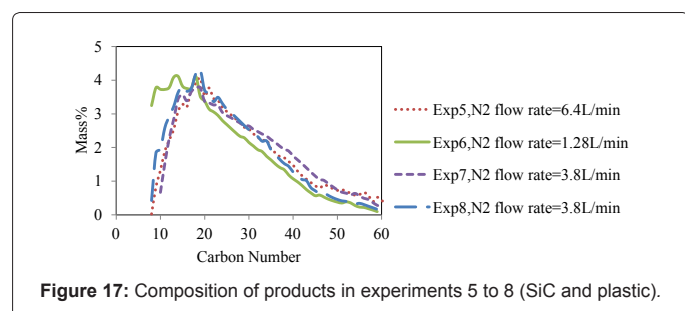
Effect of nitrogen flow rate

The change in residence time of the pyrolysed gases was due to

alteration of nitrogen flow rate which made a significant shift in carbon number distribution. This fact was observed regardless of the type of the microwave absorbent (Figures 16 and 17). Clearly lower nitrogen flow rate led to higher residence time for the pyrolysed gases and caused more cracking of the polymer chain as a result of secondary reactions. Therefore the proportion of low carbon number products (C_8 - C_{20}) was greater in the experiments with low nitrogen flow rate. A similar result was observed in conventional pyrolysis of plastic in reference [42]. The influence of nitrogen flow rate can be evidently seen in Exp_2 for carbon absorbent and in Exp_6 for SiC absorbent.

Effect of rotation

The general bulk composition in Exp_9 and Exp_10 lay within the C_{10} - C_{35} range similar to other experiments however variation in products distribution was observed with changes in rotation speed. The rotation of the reactor helped to achieve a uniform heating throughout the sample. Rotation enhanced the heat transfer and this fact made the temperature control easier. Furthermore, the rotation speed changed the residence time of the reaction; the faster it rotated the shorter time period for cracking was observed. The rotation speed was set constantly on 4 rpm in Exp_9 while higher speed was applied in Exp_10 starting at 8 rpm (in 12 minutes) and increased to 18 rpm after 40 minutes. According to the presented results in figure 18 increasing the rotation speed improved the heat transfer, shortened the decomposition time due to the whole plastic being heated at once and produced heavier



fraction. And as expected this fact increased the oil/wax yield tremendously from 46% to 73%.

Effect of absorbent type

In comparison between the absorbent type it was found out that under the experiment conditions mentioned above for Exp_3 and Exp_6, carbon distribution was shifted to the lighter fraction when silicon carbide powder was used instead of carbon block (Figure 19). In both experiments the same nitrogen flow rate and mass of load were used but a lower input power was set in Exp_6. Occupied volume in the reactor due to density differences of the absorbents could be responsible for dissimilar microwave dissipations and field patterns. The geometry of the material, position and shape of the material set a new boundary condition for the field configuration. This fact is particularly true when the dielectric loss factor is high. Moreover, SiC is recommended as an absorbent over carbon due to its high density, making it less dusty and with a higher thermal conductivity and dielectric loss factor. Minimum arcing was seen when using SiC.

Comparison of microwave and conventional heating

One of the main objectives in this study was to investigate the variation of composition under microwave heating compared to conventional heating. Figure 20 shows the comparison between the compositions of products acquired in this study (using a reactor with the volume of the 14825 cm³) with the compositions of conventional pyrolysis presented in literature (using a reactor with the volume of the 352 cm³) [42]. A very similar product distribution was obtained in both pyrolysis methods. This result is in agreement with the reported results for microwave pyrolysis of HDPE using a semi batch microwave unit (with a maximum power output of 5 kW controlled by 4 magnetron) by Ludlow and Chase [17].

In this study microwave volumetric heating did not alter the product composition, however progressive improvement in heating uniformity was observed. It was found out that pyrolysis of plastic using microwaves provides a clean and efficient heating process with an easier and faster heating control on a reasonable scale (0.5 kg of plastic). Enhancement in heating uniformity as a result of volumetric microwave heating could result in a faster pyrolysis process, while in conventional thermal pyrolysis a large surface area is required for heat transfer.

DSC results

As mentioned earlier DSC analysis was conducted to measure the latent heat and melting range of pyrolysed products and investigate their suitability as useful chemicals such as PCM. Based on GC analyses, composition of the HDPE oil/waxes cover a very diverse hydrocarbon ranges from C_9 to C_{60} suggesting a wide heat flow patterns with endothermic behaviour as shown in figure 21. All measured latent heats and melting ranges of the product came out from the first condenser

and collected waxes from the unit are summarised in figures 22 and 23. The latent heat of the second condenser product was reported only for experiments 5, 6, 8, 9, 10 and not for experiments 1 to 4 and 7. This is because for these products the DSC curve was very shallow in depth and no clear boarder for start and end points were recognised in curves, so accurate latent heats of melting could not be obtained.

According to GC/FID analyses, collected wax residue has the heaviest compounds. This may explain the higher latent heat measured with the average value of 130 J/g in figure 22 and the higher melting points with the average value of 60°C in figure 23, compared to the first condenser products with their average latent heat value of 112 J/g and 53°C melting point. Furthermore, the lowest latent heat was measured for the second condenser liquid product with an average latent heat value of 66 J/g. The measured values in this study were found below the theoretical values for the corresponding pure standard hydrocarbons. This may be associated with a high level of un-saturation in molecular structure and a very wide range of carbon numbers-as high as 160°C.

In this study it was observed that the latent heat of oil/wax products

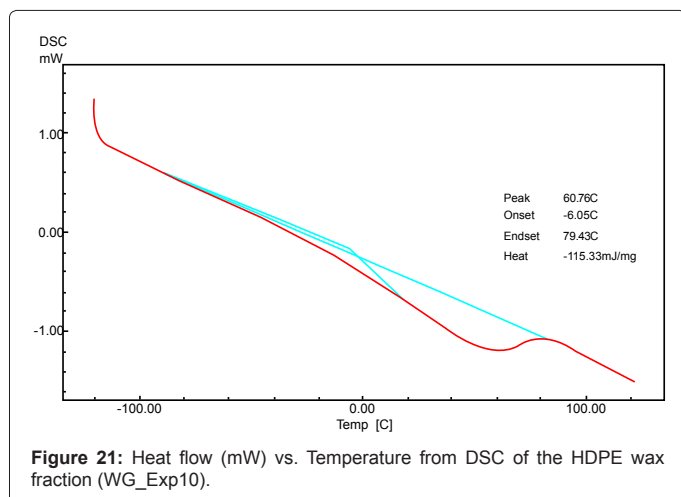


Figure 21: Heat flow (mW) vs. Temperature from DSC of the HDPE wax fraction (WG_Exp10).

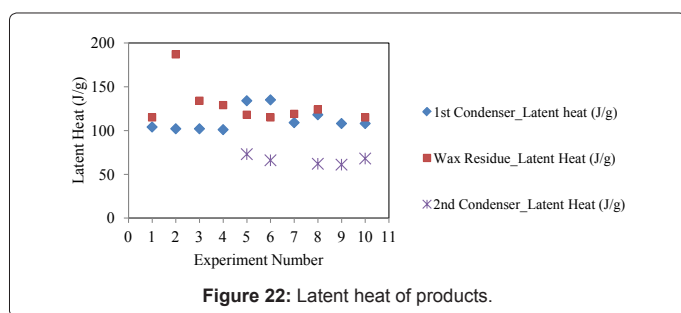


Figure 22: Latent heat of products.

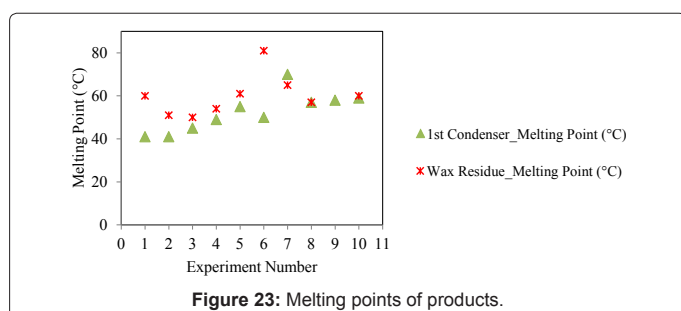


Figure 23: Melting points of products.

cannot be directly used as a PCMs application due to their low measured latent heat and very wide melting range. Hydrogenation is one of the effective methods that can be used to improve the latent heat of pyrolysed plastic products.

Conclusions

In this paper the design and performance of a microwave reactor were presented. Experiments were conducted and the results were analysed. The following items highlight the main achievements:

1. As a strong electromagnetic software Microwave Studio[®] software (MWS) provided very valuable information, furthering our understanding of electromagnetic field patterns and power loss density inside the microwave unit. The dimensions of the transmission line were optimised based on solving Maxwell's Equations using MWS.
2. The microwave pyrolysis of plastic on a reasonable scale is feasible using the proposed new design.
3. In a stationary microwave reactor, the total yield of the pyrolysis oil/wax products was around 60% and 40%, using carbon and silicon carbide as microwave absorbents, respectively. The oil/wax yield was increased to 73% with the use of silicon carbide in the rotating reactor. This is probably due to shorter residence time as a result of temperature distribution within the sample, which indicated that the whole plastic was being heated uniformly at the same time.
4. The microwave pyrolysis of plastic produced a very wide range of hydrocarbons-up to C₆₀. The large portion of the produced hydrocarbons is in the range of C₈ to C₃₅. Under the optimised pyrolysis operating condition the lower carbon numbers (C₈ to C₂₀) can be maximised and ultimately utilised as fuel.
5. Microwave heating (with reactor volume of 14825 cm³) did not alter the product composition in comparison to the conventional pyrolysis of plastic (with reactor volume of 352 cm³); however it provided very uniform heating distribution on a reasonable scale. This indicates that the microwave pyrolysis of plastic can be achieved much faster than thermal pyrolysis. Although both thermal and microwave reactors behaved the same here, this will not be the case in an industrial scale pyrolysis reactor, which would require a large heat transfer area, unlike in the microwave reactor.
6. Silicon carbide is recommended as a microwave absorbent over carbon due to its electrical and thermal physical properties, such as its higher loss factor making it able absorb microwaves more efficiently, its higher density meaning it creates less dust in the reactor, and higher thermal conductivity to aid in the heat transfer within the sample mixture.

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