Laboratory scale equipment for microwave pyrolysis of waste petrochemical based products

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The decomposition of hydrocarbons by elevated temperatures in an inert atmosphere is a well understood process within chemistry. In recent years there has been a developing interest in the process to recycle waste hydrocarbon based materials. Conventionally furnaces have been used to heat the materials, however over the last decade small industrial facilities have been constructed that heat the waste using microwave energy.

Pyrolysis as a recycling process is economically justifiable if the value of the products recovered plus any reduction in taxes and tariffs are intrinsically more valuable than the installation and running costs of the facility. To understand the economic value of the end products of the waste materials a laboratory scale equipment has been developed.

The equipment consists of two 1 kW cw magnetrons connected to a microwave chamber which takes small samples of waste materials, typically in the range of 50 to 100 grams and heats the samples by means of the microwave energy in an inert atmosphere. The resulting hydrocarbon gases, liquids and solids are then collected and analysed.

This paper discusses the equipment details and shows results from the microwave pyrolysis of waste rubber and ashphaltene.

INTRODUCTION

Pyrolysis is the thermal decomposition of organic materials (hydrocarbon compounds) in a inert, oxygen free, atmosphere (1). In recent years it has been of interest in the production of char (fig 1) from biomass such as wood pellets.

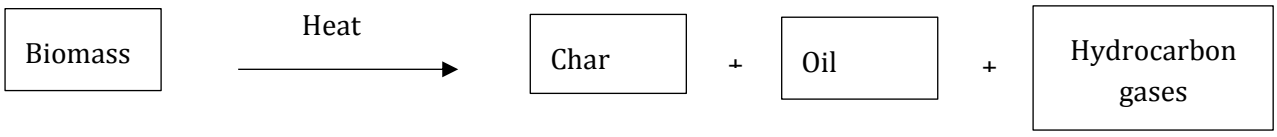


Fig 1 Production of char, oil & gases from biomass

The same process can be applied to any other hydrocarbon based chemicals, such as waste plastics or rubber.

The temperature required to initiate pyrolysis depends on the chemical bond in the compounds and is normally applied by heating the material in an electric or gas furnace with oxygen removed.

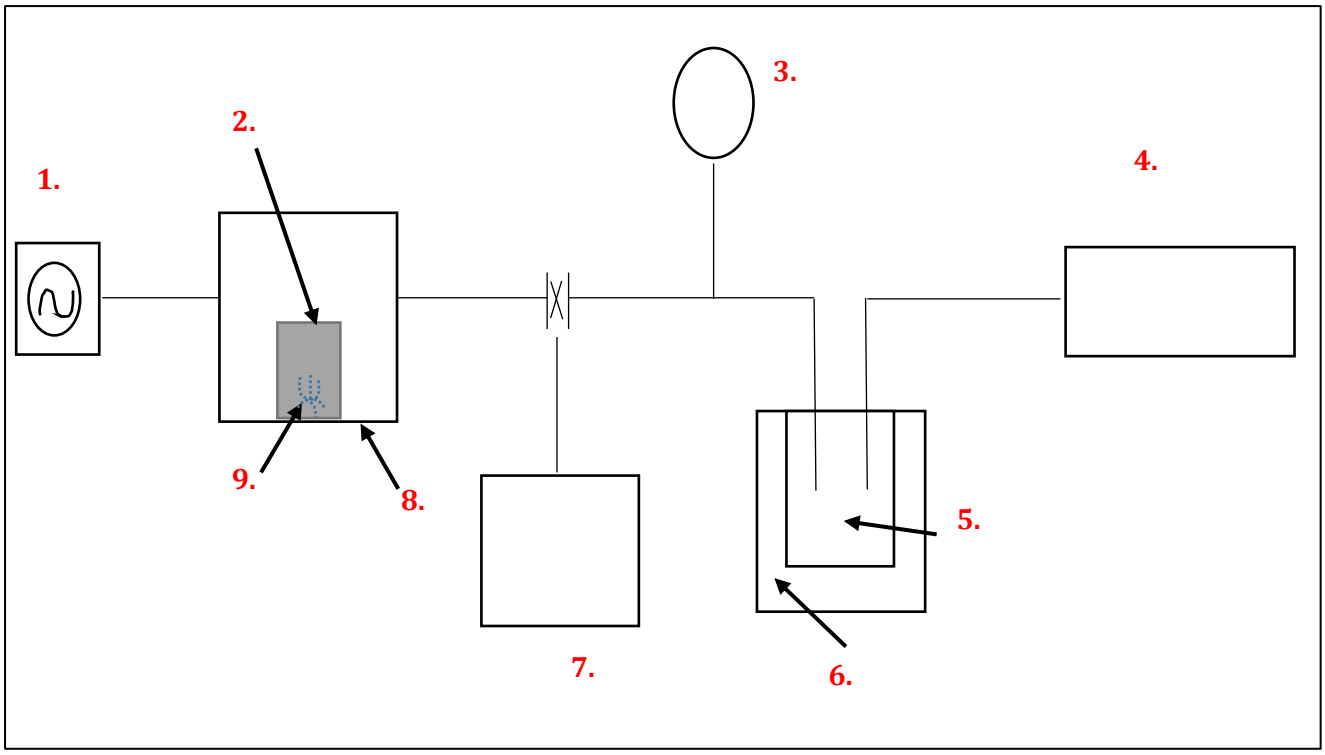
However, an alternative approach that is generating interest is to apply the heat by means of microwave energy.

Recently several small scale industrial plants have been built, however these lend themselves to treat a single waste material which is well understood. There is a need to determine whether other mixed materials or less understood waste materials are suitable for microwave pyrolysis. We developed a laboratory scale equipment (fig 2) suitable to process small samples of materials to investigate their suitability for recycling by microwave pyrolysis.

Fig 2 Laboratory scale equipment for microwave pyrolysis

LABORATORY SYSTEM

A schematic of the system is shown in Fig 3.



1 – Magnetron & power supplies, 2 – Infra red thermometer, 3 – Pressure Gauge, 4 – Gas Collector, 5 – Liquid Collector, 6 – Condenser, 7 – Vacuum Pump, 8 – Cavity, 9 – Sample

Fig 3 Schematic of laboratory microwave pyrolysis equipment

The system consists of

- Vacuum pump to remove gases & allow an inert atmosphere. The waveguides, reaction chamber and condensing system all operate from vacuum to a pressure of 3 atmospheres. The oxygen is removed by nitrogen purging the system three times and then ensuring that the oxygen content is below 3% by volume. The system is then evacuated for a final time and the reaction can commence.
- Two 1 kW, 2450 MHz cw magnetrons powered by a line type high voltage supply. The output waveguides are designed with vacuum windows.
- Reaction chamber consisting of a microwave coaxial re-entrant cavity tuned to 2450 MHz.
- Stainless steel pipework to transport gases from the reaction chamber to the condenser.
- Condenser operating at -10°C to chill the gases and condense the oils.
- Liquid trap to collect the oils.
- Gas trap to collect the hydrocarbon gases.

- Sensors to monitor oxygen, sample temperature & pressure.

Below is an internal view of the system with some of the critical components labelled.

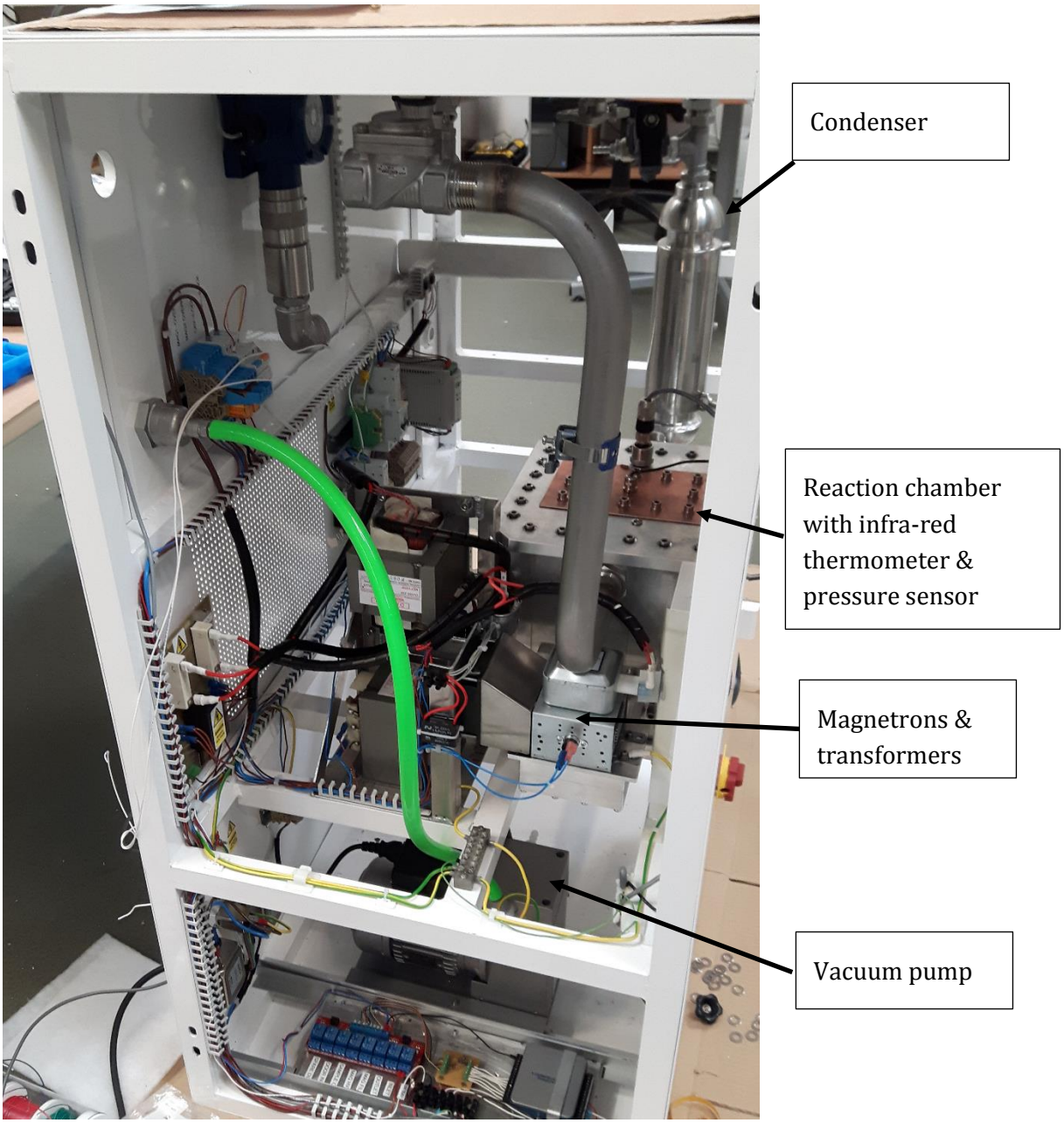


Fig 4 Internal view of laboratory system with some critical components labelled

The sample to undergo the pyrolysis reaction is placed in the middle of the reaction chamber and the process is then fully automated. The microwave power can be either applied for a defined time period or controlled by setting a temperature and the power will then be maintained by switching the microwave power on and off as required.

EXPERIMENTAL RESULTS

The equipment has been designed to pyrolyse any hydrocarbon materials, however it's capabilities were validated with waste rubber as this is one of the easiest materials to pyrolyse.

a) Pyrolysis of rubber

Below are the results for a pyrolysis reaction of 50g of rubber for 300s of applied microwave power.

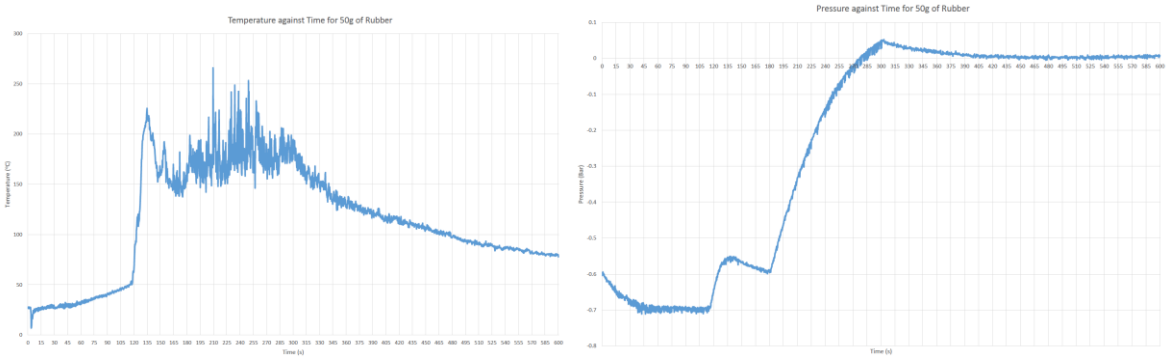


Fig 5 Temperature & pressure curves for microwave pyrolysis of a 50g rubber sample

At 0s the microwave power is applied and the sample temperature gradually increases until at 120 seconds the reaction starts. The reaction immediately starts generating gas and also results in a very steep thermal rise until equilibrium is reached. During this period the pressure gradually increases until it exceeds atmospheric pressure. After 300 seconds the microwave power is turned off and the sample gradually cools.

The gases then can be collected and the liquid allowed to condense in the liquid trap.

b) Pyrolysis of asphaltene

Asphaltene is a material produced during crude oil drilling and refining. It is a high temperature distillation residue of petroleum (>530 °C) and is approximately 1% by weight of crude oils (2). In some oil fields the amount of Asphaltene can be upto 10%.

The Asphaltene is a solid black material which clogs drill bits and precipitates to the bottom of oil refineries (3). It is currently a waste material that needs to be manually removed and treated as hazardous landfill waste. The higher temperature and complex properties make it more challenging than materials such as rubber. The equipment has been validated with Asphaltene and below are the results

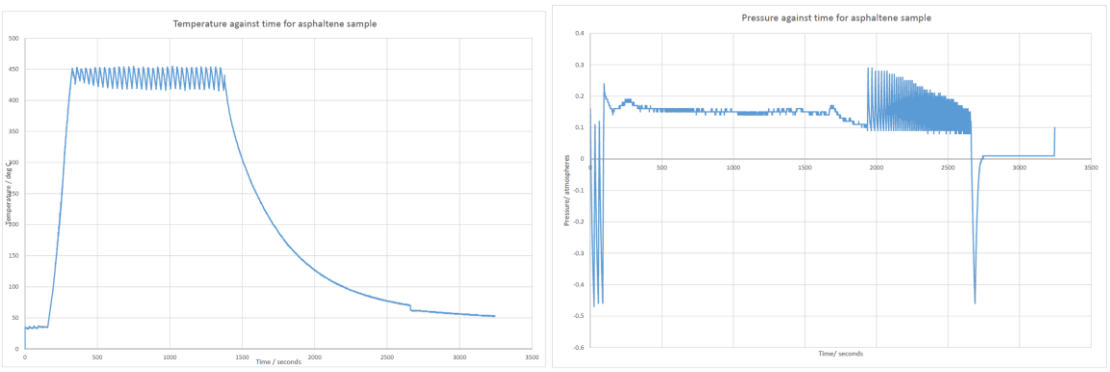


Fig 6 Temperature & pressure curves for microwave pyrolysis of a 10g Asphaltene sample

The Asphaltene material, unlike rubber, is not a good microwave absorber therefore silicon carbide was added to enable the energy to be coupled into the material until the reaction starts. In addition to ensure that the oils do not condense into the microwave chamber an inert transport gas was used to remove the gases from the chamber. The photograph below shows the oils collected in the liquid trap after this reaction.

Fig 7 Oil collected in liquid trap from microwave pyrolysis of 10g Asphaltene

CONCLUSION

A laboratory scale apparatus for the microwave pyrolysis of hydrocarbons has been built and demonstrated. It has successfully pyrolisied rubber and asphaltene. Producing char, oils and hydrocarbon gases.

Further work is intended with this technology particularly regarding analysis of the microwave absorption properties of various petrochemical waste materials and scaling the system up such that it can handle 1 kilogram samples.

REFERENCES

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