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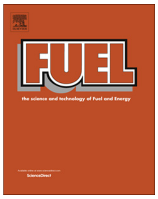

Full Length Article

Fuel production from pyrolysis of natural and synthetic rubbers

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HIGHLIGHTS

- Different rubber materials were pyrolysed to produce fuels.
- Product fuels were characterised by GC, FT-IR and GC–MS, respectively.
- Mass change of rubbers during pyrolysis was tested by thermogravimetric analysis.
- Heat change during rubber pyrolysis was tested by computer aided thermal analysis.
- Natural and synthetic rubber tyres and natural rubber mat were compared.

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ABSTRACT

Pyrolysis of rubber wastes is an efficient thermo-chemical means of generating high-value energy and fuels. In this work, a variety of technological tools were employed to characterise the pyrolysis of natural and synthetic rubbers, which included three tyres (natural rubber tyre, pneumatic tyre and synthetic rubber tyre) and one natural rubber mat. The composition of gas products was determined by gas chromatography (GC). H_2 and CO were the major gases released from the pyrolysis process although the evolution rates of gas species differed for the four samples. The organic compounds contained in the resulting pyrolysis oils were analysed by gas chromatography–mass spectrometry (GC–MS), and the results confirmed the existence of large amounts of hydrocarbons and nitrogen-containing compounds as well as some sulphur-containing compounds. The raw rubbers and solid char products were analysed by Fourier transform-infrared (FT-IR) spectrometry to determine the change in functional groups, which indicated the loss of organics after pyrolysis at different temperatures. Distinct mass and heat change behaviour of the selected rubber samples was also detected through thermogravimetric analysis and computer aided thermal analysis, respectively. The pyrolysis behaviour and fuel product properties of the rubbers were compared in this work. The obtained data will provide important reference information for energy and fuel generation from rubber pyrolysis.

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1. Introduction

Waste management is a global issue which impacts the sustainability of environmental and economic developments. Rubber waste in the forms of tyres, gloves or others have been a problematic material for treatment as it is highly resistant to biodegradation. Natural rubber (NR) consists of an elastic polymer (*cis*-1, 4 polyisoprene) from latex which is almost all from the rubber tree (*Hevea brasiliensis*). Due to the shortage of natural rubber and purpose of various applications, currently the majority of the rubber market share is held by vulcanized synthetic rubber (SR) which is generally comprised of cross-linked long-chain man-made polymers (e.g., tyrene/butadiene and isobutylene/isoprene) with sulphur atoms. These artificial polymers are synthesised from fossil petroleum and then primarily used to make tyres. For instance, *cis*-polybutadiene rubber (CBR), isobutylene–isoprene copolymer rubber (i.e., butyl rubber, abbr. BR) and styrene–butadiene copolymer rubber (SBR) are three of the most common synthetic rubbers used in tyre manufacturing. A tyre may contain synthetic rubber, natural rubber, carbon black, steel, fabric, plasticizers, lubricants, antioxidants, antiozonants, inorganic materials (e.g., calcium carbonate and silica), and other components.

As the most abundant rubber waste, approximately 4×10^9 end-of-life tyres (equivalent to 3.2×10^8 tonnes) are estimated to be disposed through landfills and stockpiles worldwide [1]. In Australia, an annual estimate of more than 2×10^5 tonnes of waste tyres are landfilled or stockpiled [2].

According to the hierarchy of sustainable waste management by US EPA, the priority decreases in the order: source reduction

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and reuse > recycling and composting > energy recovery > treatment and disposal [3]. Considering the difficulties for the reuse or recycling of rubber wastes, energy and fuel generation through several thermo-chemical conversion technologies is gaining increased interest. In the EU, incineration for production of heat and/or electricity is the most common treatment technology followed by reuse and export [4]. Additional advanced technologies have been applied to recover fuels or chemicals from rubber wastes, such as gasification [5] and pyrolysis [6].

Pyrolysis technology thermally decomposes rubber or other organic wastes (e.g., biomass), in an inert atmosphere at temperatures of around 350–550 °C or higher, to give fuel products containing liquid pyrolysis oil, solid char and pyrolytic gas. The resulting products can be further and selectively upgraded into value-added fuels or chemicals [7–10]. Many studies have been conducted on the pyrolysis of rubber materials. Choi [11] applied pyrolysis-GC to study the volatile pyrolysis products from 15 patterns of SBRs with different microstructures. It was found that butadiene, 4-vinylcyclohexene and styrene are the dominant products with the ratios affected by relative contents of styrene, 1,2, *cis*-1,4-, and *trans*-1,4-units. Danon et al. [12] identified the primary pyrolysis products of monomers and dimers from three different rubbers of NR, CBR and SBR using the instrument of combined thermogravimetric analyser and mass spectrometer (TGA-MS). However, in their research, the heat changes of the rubbers during pyrolysis, including specific and latent heats, and the properties of other products were not characterised. Lah et al. [13] modelled the pyrolysis kinetics of different tyre components of NR, BR, SBR and other fabric, oil and additives in scrap tyre rubber using a thermogravimetric analyser. Miranda et al. [14] also investigated the kinetics of waste tyre mainly consisting of NR, BR and SBR and determined the effect of temperature on the composition of pyrolysis oil products. It was observed that the lower temperature (<390 °C) favoured the formation of alkene products while the higher temperature favoured formation of aromatics. Williams [15] compared the waste tyre pyrolysis in various reactor types, such as fixed bed, moving screw bed, rotary kiln, vacuum, conical spouted bed, fluidised bed and drop tube reactor, and the oil, char and gas yields fell in the ranges of 5–63%, 22–49% and 3–73%, respectively.

The previous studies were mainly focused on the pyrolysis kinetics modelling of major components in lump waste tyres, characterisation of products (especially pyrolysis oil) from pyrolysis of a lump rubber tyre, or pyrolysis of NR products (e.g., latex gloves) [16,17]. Few studies have been performed on the comprehensive comparison on the pyrolysis characteristics and differences between NR tyres and mats, and between NR and SR tyres. In this work, rubber materials from different resources including three tyre types (i.e., NR tyre, pneumatic tyre and SR tyre) and one NR mat, were subjected to slow pyrolysis process wherein the product properties were characterised. Mass and heat changes of the selected rubber materials were also investigated. The work will facilitate further understanding of pyrolysis of different rubber materials to generate fuels, which is beneficial in meeting the increasing global fuel demand.

2. Experimental

2.1. Feedstock

All black rubber materials in the original forms were purchased from the distributor of J Blackwood & Son Pty Limited (Australia), including natural rubber tyre F50 coupling (abbr. NR–Tyre), natural rubber mat of 1200 mm × 1.59 mm (abbr. NR–Mat), pneumatic black rubber tyre (abbr. PR–Tyre, supplier: EHI Australia PTY LTD) and synthetic rubber tyre FRAS (fire resistant & anti-static) F40 (abbr. SR–Tyre). Rubber pieces were shredded from the tyres/mat and then ground with the assistance of liquid nitrogen, followed by sieving to obtain fine particles of below 212 μm. The particles were dried at 60 °C in a vacuum oven prior to the experiments. The proximate analysis was performed in a thermogravimetric analyser (model: TGA/DSC 1 STAR^e System, Mettler Toledo Ltd.) according to ASTM E1131. Table 1 lists the analysis results.

2.2. Characterisation of products from rubber pyrolysis

An infrared furnace with temperature-programming was used to generate gaseous (pyrolytic gas), liquid (mainly pyrolysis oils) and solid (chars) from the pyrolysis of different rubbers. The fine particles of each rubber type were packed in a quartz tube fixed-bed reactor at a heating rate of 10 °C/min in an inert helium atmosphere.

2.2.1. Pyrolytic gas

During the pyrolysis process from room temperature to 600 °C, the composition of released pyrolytic gas with temperature was analysed by an online micro gas chromatograph (Model: M200, MTI Analytical Instruments) with two thermal conductivity detectors. Ultra-high purity He was used as the carrier gas. The H_2 and CO peaks were determined by a molecular sieve 5A column at 60 °C with CO_2, CH_4, C_2H_4, and C_2H_6 peaks by a Poraplot U column at 40 °C. Chromatographic spectra were recorded at an interval of 100 s.

2.2.2. Pyrolysis oils

The condensable organic vapours (i.e., pyrolysis oils) from the rubber pyrolysis were continuously swept out of the reactor and condensed on cool quartz wool placed at the reactor exit until the temperature reached 500 °C. Subsequently, the quartz wool was washed using dichloromethane (DCM) solvent to extract the pyrolysis oil components which were then derivatised by N,O-bis (trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane (BSTFA + 1%TMCS). A GC–MS (gas chromatography–mass spectrometry, model: Agilent 7890B GC coupled with 5977A MS) system equipped with a HP-5MS column (60 m × 0.25 μm) was used to test the detailed components of pyrolysis oils. The GC oven was initially set to 40 °C for 2 min, then heated to 310 °C at 2 °C/min and kept at 310 °C for 30 min.

2.2.3. Chars

Rubber samples were heated to different target temperatures (300 and 500 °C) for char production. The functional groups of the raw rubber samples and the chars after pyrolysis were acquired by a Fourier transform-infrared (FT-IR) spectrometer (model: Nicolet 6700 FT-IR) with an attenuated total reflectance (ATR).

2.3. Thermal behaviour of rubbers

2.3.1. Mass loss during pyrolysis

The mass loss of rubbers presented as thermogravimetric (TG, wt.%) data during pyrolysis from room temperature to 600 °C at

Table 1
Proximate analyses of rubbers.

Proximate analysis	Results/wt.%			
	NR–Tyre	NR–Mat	PR–Tyre	SR–Tyre
Moisture	0.21	0.27	0.47	0.69
Volatiles	54.05	57.46	59.97	47.18
Ash	10.66	27.38	18.84	12.36
Fixed carbon [a]	35.08	14.89	20.72	39.77

[a] By difference.

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