



Towards the thermal utilisation of non-tyre rubbers – Macroscopic and chemical changes while approaching the process temperature



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HIGHLIGHTS

- Non-tyre rubbers may build sticky surfaces at the temperature of the fuel feeding path.
- The extensive fragmentation starts at about 240 °C.
- The release of volatile products occurs in two distinct temperature ranges.
- Most of the chlorine- and nitrogen-containing compounds are released below 400 °C.
- The duration of the complete volatile release is several minutes under FBC conditions.

ARTICLE INFO

Article history:

Received 29 November 2014

Received in revised form 14 April 2015

Accepted 16 April 2015

Available online 24 April 2015

Keywords:

Waste derived fuel

Non-tyre rubber waste

Pyrolysis-GC/MS

TG/MS

FBC

Gasification

ABSTRACT

This paper presents important changes in the basic fuel properties of non-tyre rubber wastes during heating from ambient to the temperature of the thermal conversion reactor. Experiments were carried out on both macroscopic and chemical processes occurring throughout the sample path. Special emphasis was put on the possible utilisation of non-tyre rubber wastes in Fluidised Bed Conversion units. The results show that some potential fuels studied may build sticky surfaces in the fuel feeding paths while all the samples undergo extensive fragmentation. Thermogravimetry/mass spectrometry experiments demonstrate that the volatile production occurs in two distinct temperature ranges characterised by the limits of about 150–350 °C and 350–550 °C. The gas chromatographic analysis shows that most of the chlorine- and nitrogen-containing compounds are formed in the first temperature range of pyrolysis and these products are released from the additives of the rubber samples. The duration of devolatilisation is 1.5–2.5 min under the normal fluidised bed combustion of 50 mm rubber particles. Considering the results achieved, the possible ways of practical applications is also formulated in this paper.

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1. Introduction

Several sorts of waste rubber appear to be an attractive source for energy production because of their high calorific values, low or even negative prices, as well as legislative, financial, and environmental problems at conventional disposal [1–3]. The three main groups of available technologies are pyrolysis, gasification, and combustion.

Combustion seems to be the most widespread technology, which is characterised by the longest history, the high number of running commercial plants (see e.g. [4–6]) and its intensive research background [2,7–10]. Fluidised Bed Combustion (FBC) appears to be a very successful solution among the combustion

methods for its well-known flexibility against fuel quality, being a beneficial property when using waste rubbers of rather variable compositions. It is interesting to mention a worldwide boom of building FBC plants firing waste rubber. One of the leading countries in FBC technology is Japan, where between 2005 and 2010 nine FBC plants were put into operation firing waste rubber [11].

Pyrolysis is another rapidly developing technique for the thermal utilisation of waste rubber, although the pyrolysis research started later than the studies on rubber combustion [12–15]. An up-to-date summary about pyrolysis [3] discusses its technical basics, industrial units, main development directions as well as its legislative and policy background.

Even though gasification is well-known and widely applied for various solid fuels for several decades, its application for rubber waste seems to be a quite new idea. Nevertheless, recent theoretical and experimental results on different kinds of waste rubber show that gasification is a promising method as well [15,16].

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In all the above mentioned thermochemical conversion methods, a common and first event is the warming up of the fuel as it is evident that it has to be transported from the storage area into the reaction chamber. It is important to study the processes occurring in the fuel during transportation from the storage area to the feeding point since the temperature difference between these two endpoints is several hundreds of degrees Celsius and the fuel goes through the pipeline within a few tens of minutes.

The goal of the experimental investigation summarised in this paper was to gather information on the above topics applying the most relevant types of non-tyre waste rubber as a solid fuel. It was also considered that the physical and chemical processes occur dominantly in the feeding route at ambient pressure and gas composition while the last processes take place inside the reactor characterised by significantly elevated temperature, different atmosphere, and other conditions.

The phrase *rubber* covers an extremely wide range of materials described by various compositions, physical, and chemical properties [17,18]. Industrial rubber products are usually composed of 10 or more ingredients [17] and the main component is called *elastomer*. The repeating elements of these polymers are called *monomers*, the selection of which basically determines the properties of rubber. Therefore, the rubbers are grouped by the elastomers (i.e., monomers). A list containing 27 common elastomers, together with their ASTM (American Society for Testing and Materials) designations can be found in the work cited above [17]. The most important and most frequently applied additive is carbon black and a large variety of further additives (e.g., plasticisers, antioxidants, fire retardants) is used as well [17].

An evident way of categorising waste rubber fuels could be based on the types of elastomers and other chemical components. In practical applications, this approach cannot be used because of the unknown chemical compositions of the individual waste particles. Therefore, another approach is used in the waste industry, which is based on the origin of the rubber wastes. Some relations between these two categorising approaches exist, however, a strict correspondence between them cannot be formulated. In the present paper the later categorising aspect is followed as this approach allows the possible application of the results to be presented here.

Rubber wastes are often categorised into two major groups of *tyres* and *non-tyre* products [19], both of which can be further subdivided. The current study focuses on two, industrially relevant subgroups of *non-tyre* products, namely hoses and manufacturing sprues. They were chosen as a result of previous feasibility studies with special emphasis on their qualities, amounts and predictable availabilities.

Although waste tyres were excluded from this study, the published literature and experience on the applications were carefully studied and considered. Tyres, especially those of private cars, motorbikes, and small lorries are mainly made of styrene-butadiene rubber (SBR) [19], however, the quality and amount of additives still allow a high variety within this widely investigated group.

It is interesting to note that the main problem of tyre utilisation for power generation seems to be the presence and removal of wires [4–6]. In addition to a few unusual solutions [20,21], most of the technical papers report about 94–95% success rate in removing wires from various locations of the fluidised bed boilers [6]. It is also important to mention that milling or cutting rubber waste, even after a proper wire removal, is difficult and energy intensive. This makes FBC even more advantageous because of its particle size flexibility.

The scientific papers on the thermochemical conversion of tyres report some interesting findings, which may be important for non-tyre fuels as well. The phenomenon of primary fragmentation was

described by a research group [9,22], which seems to be the characteristic of all sorts of rubber. This process has very high importance in the fluidised bed conversion technique. The same group also gives a theoretical description of tyre pyrolysis, which was divided into three phases [2,23]. It was verified by measurements of the weight losses while heating up the tyre particles.

Another research group measured the hydrodynamic characteristics of combusting tyre in an FBC and the mass loss of tyre was monitored during heating up [10]. It was observed that pyrolysis of the tyre occurs in two stages but the composition of the evolved products in the two stages were not detected. Dynamic thermogravimetric investigations were carried out on rather big samples (about 30 g) of SBR [14] and the yield of gas, tar, and char components were measured under various pyrolysis conditions. A strong influence of the heating rate and the oxygen concentration was shown in an earlier publication [24]. The differential thermogravimetric (DTG) curves showed a double-peak character, which was not further investigated at that time.

Non-tyre waste rubbers as a promising resource for thermal utilisation were investigated at a lower intensity than tyres. Important data and observations were reported on the combustion behaviour and the pollutant emission during firing nitrile-butadiene rubber (NBR) balls and woodblocks in an FBC reactor [16,25,26]. FBC technology was used [12] for a non-tyre chloroprene rubber (CR) which has a high annual production in Germany. Gas chromatographic (GC) analysis was carried out focusing on the chlorinated aromatic compounds in the volatile products. The final temperature of the reactor was 593 °C, and the important GC results obtained refer to this single temperature. The total amount of syngas was measured [15] during pyrolysis and gasification of butadiene rubber (BR) at higher temperatures (800 °C and 900 °C). An important observation was that the duration of rubber gasification was several minutes under these conditions.

It is a generally known macroscopic behaviour of rubber that the pieces fall apart into powder-like, very small particles during heating, which is called primary fragmentation. Another intermediate macroscopic change may occur, however, it is very rarely mentioned. Some rubber materials have the tendency for getting sticky, which is essentially important for the design of fuel feeding routes. The only source found where this behaviour is described calls this phenomenon as “sticky combustion surface” [27].

2. Materials and methods

2.1. Test materials

The non-tyre rubber samples were taken from waste management enterprises, which appear to be long-term suppliers of a commercial power plant in Hungary. The suppliers' rules and methods of waste rubber classification were adopted and followed.

Two subgroups of *non-tyre* rubber wastes were selected in a preliminary study because of their permanent availabilities and approximately equal qualities. Both of them are production scraps, which assures their relative cleanliness without much unpredictable external pollutions.

The term hose refers to the first type of non-tyre rubber wastes investigated and discussed in this paper. Hoses are produced from a wide range of elastomers but butadiene is one of the most important monomers constituting butadiene rubber and nitrile butadiene rubber [17,19]. The investigated samples originated most probably from car industry. However, their diameters and appearance varied considerably and their constructions also had various elements. Hoses contained (see Fig. 1a) several layers of the rubber body, a braided fibre reinforcement mesh, and an inner liner. Some

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