



Extracting oil from used auto tires at low temperature after chemical treatment



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ARTICLE INFO

Article history:

Received 13 October 2016

Revised 9 December 2016

Accepted 4 January 2017

Available online 19 January 2017

Keywords:

Tyres

Solvent extraction

Sulphur content

Oil yield

ABSTRACT

This study presents an alternative tactic to pyrolysis of auto tires avoiding the use of high temperature and increasing the yield of oil produced. It depends on a simple chemical treatment of auto tires with sodium carbonate at low temperature (50 °C) followed by solvent extraction. This treatment produced two folds of the yield that can be obtained using normal solvent extraction. The experimental results suggests that sodium carbonate is responsible for breakage of C–S bond in the main structure of auto tires making solvent extraction easier. Additionally, the sulphur content of the extracted oil using the sodium carbonate treatment is reduced significantly (by about 28%) making the product more favorable energy/fuel source. This technique allows about 30 wt.% of oil to be extracted from the used auto tires at 50 °C under atmospheric pressure resulted from the breakage of the sulphur cross-linking by the sodium carbonate.

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1. Introduction

According to the data reported by the International Rubber Study Group (IRSG), the total world production of tires in 2014 was estimated at around 15.85 million tons (JATMA, 2016). More than 100 raw materials are used in the production of automobile tires, including raw rubber (50.8%), reinforcing agent (24.9 wt.%), compounding ingredients (5.9 wt.%), bead wire (4.6 wt.%), and tire cord (13.9 wt.%) (JATMA, 2016). The rubber consists of two types (natural rubber 28.6 wt.% and synthetic rubber 22.2 wt.%) while the tire cord consists of two types (textile cord 3 wt.% and steel cord 10.7 wt.%).

Rubber tire production is a complicated process comprising several steps including milling, extruding, calendaring, cementing, marking, lubricating and finishing processes. Various ingredients are used such as synthetic and natural rubbers, sulphur, carbon black, silica, oil, zinc oxide, and other chemicals (Sienkiewicz et al., 2012).

Sulphur is incorporated into tires during the so-called vulcanization process to cross-link the initial polymers (i.e., rubbers) and make them more durable. The sulphur cross-linked material

has a limited solubility in organic solvents due to the strong chemical bond of C–S. The sulphur content in tires is typically about 1 wt.% (Williams, 2013). Carbon black is another important component that is added in a very pure powder form in order to act as a reinforcement material to increase the rubber strength, decrease its erosion by friction with road surface and decrease its photo tendering caused by exposure to sunlight. Synthetic rubber has some advantages over natural rubber, because it is purer and because it is very cost effective. About 55 wt.% of a tire's rubber content is in the sidewall and tread. Reinforcing chemical agents represent a high percentage of the chemical fillers used; the most common are carbon black, silica and resin. Silica is useful in reducing rolling resistance. This, of course, decreases the friction between tires and road which increases the tires life and save fuel consumption. In addition to silica, other chemicals are added such as bis (triethoxysilylpropyl) tetrasulfide. Manufacturing companies use anti-degradants (antioxidants, paraffin and waxes) as well as adhesion promoters (cobalt salts, brass on wire and resin on fabrics) in tires. Oils, softeners, cotton, aramid fibre, steel fabric, rayon, polyester and fiberglass are also common additives.

Several research works have been conducted on the scrap tires recycling and disposal. These include landfill, the reuse as construction material, the use as fuel and thermal decomposition to gas, oil fraction and char. Solvent and supercritical extractions

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are also investigated as a promising process to extract valuable compounds from disposal scrap tires (Sharma et al., 1998; Kyari et al., 2005; Murillo et al., 2006; Fernández et al., 2012; Williams, 2013; Al-Rahbi and Williams, 2016).

Different solvents have been used to extract oil from used automobile tires such as toluene, water and *n*-heptane under supercritical extraction techniques (Funazukuri et al., 1985, 1987; McMillen et al., 1989; Teng et al., 1995; Orr et al., 1996a, 1996b; Tang and Curtis, 1996; Mastral et al., 1997; Park and Gloyna, 1997; Cunliffe and Williams, 1998; Miguel et al., 1998). According to Funazukuri et al. (1985, 1987), 57 wt.% of tires can be converted into oil by using toluene supercritical extraction at a temperature of 380 °C and a pressure of 5.2 MPa. On the other hand, water supercritical extraction converted 68 wt.% of tires into oil according to Park and Gloyna (1997). Kershaw (1998) found that the oil yield obtained using ethanol supercritical extraction at low temperature is much lower than that of toluene, *n*-butanol and acetone. In this work, hexane, acetone, acetone/hexane mixture, DCM, methanol, methanol/DCM mixture were used as solvents to extract oil from tires using soxhlet extractor after specific chemical treatment for tires at low temperature (below 90 °C) and atmospheric pressure.

2. Experimental

2.1. Samples

Samples of tire shreds with a size of 1.5 mm were obtained from a local tire recycling factory. Samples were washed with water to remove any dust or dirt. Then the washed samples were dried in a vacuum oven at 80 °C for 2 h.

2.2. Solvents

Different solvents were employed in this study comprising polar and non-polar solvents. They include acetone, hexane, dichloromethane (DCM), and chloroform. HPLC grade chemicals were used in this study. A mixture of acetone and hexane were also employed as a combined solvent.

2.3. Treatment of tires

Different chemical treatments were suggested in this study that were believed to enhance solvent extraction at low temperatures. Sodium carbonate (Na₂CO₃), sodium bicarbonate (NaHCO₃), and hydrogen peroxide (H₂O₂) were used as chemicals for tires pre-treatment.

2.4. Experimental procedure

Washed samples of used tires of 100% passing 1.5 mm were treated with different chemicals at different concentrations and different temperatures in a controlled temperature vessel while mixing. The treatment took place for 2 h. After the treatment is completed, the samples were washed with an excess amount of water and dried in vacuum oven at 80 °C for 2 h. The following chemicals and conditions were used for treatment:

- 2 M Na₂CO₃ solution at 50 °C
- 1 M Na₂CO₃ solution at 50 °C
- 1 M NaHCO₃ solution at 50 °C
- 1 M NaHCO₃, and 1 M Na₂CO₃ at 50 °C
- 10 vol.% H₂O₂ solution at 30 °C
- Water at 105 °C followed by 10 vol.% H₂O₂ solution at 30 °C.

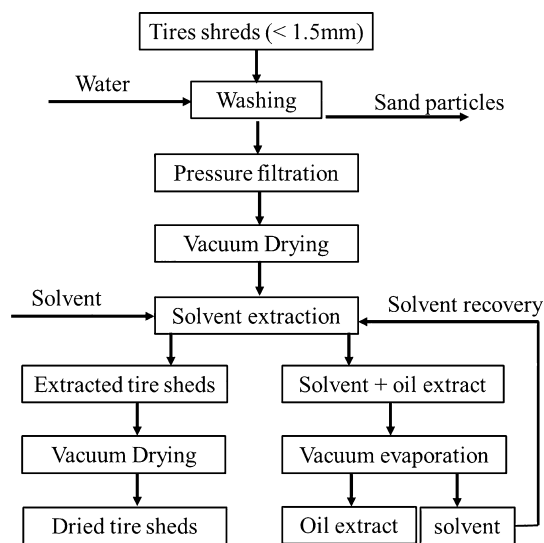


Fig. 1. Schematic diagram showing the extraction procedure of oil from tire shreds.

The samples were dried after the chemical treatment; they were then extracted using different solvents using a soxhlet extractor. The solvents used were acetone, hexane, dichloromethane, 50 vol.% acetone + 50% vol.% hexane (50A/50H) and 30 vol.% acetone + 70% vol.% hexane (70H/30A). The extracted oil was separated from the solvent using a rotavap, while the extracted solid tires were dried in a vacuum oven. The weight of the oil extracted as well as the weight of the extracted solid tires were recorded. A schematic diagram of the procedure employed is shown in Fig. 1. The extraction method used allows the recovery of solvent by vacuum evaporation to be reused for solvent extraction again.

2.5. Characterization

After completely removing the whole solvent from the extracted oil by drying, the resulting oil was separated into different fractions based on the polarity of the extracted oil using Biotage® Isolera flash chromatography system. The system utilised gradient elution using two solvents; *n*-hexane, and ethyl acetate starting with 100% hexane and ending with 100% ethyl acetate. The absorbance was measured at two wavelengths; 254 and 365 nm.

NMR spectra were recorded on a Bruker-Avance 400 MHz spectrometer. The residual solvent protons (¹H) were used as internal standards. Visualization of TLC was accomplished with UV light (254 nm).

¹H NMR analysis for fractions obtained from the Isolera autoflash chromatography system was analysed using ¹H NMR. More specifically, the analysis was performed for samples obtained from untreated samples at 90 °C and samples treated with 2 M Na₂CO₃. This was done for the purpose of comparison on type of molecules before and after treatment.

FT-IR spectroscopy, Bruker, TENSOR II with an ATR attachment was used in this study to identify the functional groups present in both oil and tires.

The sulphur and carbon contents were analysed using LECO carbon sulphur analyser (model CS 244).

3. Results and discussion

3.1. Extraction yield

The effect of using different chemicals for treatment for tire shreds followed by solvent extraction using different solvents is presented in Figs. 2 and 3. The treatment using 2 M Na₂CO₃ has

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