ORIGINAL ARTICLE

Chemical recycling of waste tire in high-temperature organic fluid

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Abstract

The inadequate treatment of waste tire is currently a major environmental and economical issue. In this study, the effective solubilization of waste tire into diethyl ether was attained by the thermal treatment in organic solvent at 200–300 °C. Not only aromatic, but also aliphatic alcohols were effective on the solubilization. Among aliphatic alcohols, 1-heptanol was the most effective solvent on the solubilization and reached near 70% in the thermal treatment at 200 °C under inert atmosphere. Model compound study containing sulfur–sulfur bond suggests the disulfide–alcohol exchange reaction leading devulcanization of the waste tire.

Keywords Waste tire · Chemical recycling · Decomposition · Solvolysis

Introduction

The amount of waste tire increase in 2018 was 1 million tons in weight in Japan [1] and increases globally [2]. Recycling of the waste tire [3] is worldwide of growing importance due to increasing raw material costs, diminishing resources, and growing awareness of environmental issues and sustainability [4].

The main recycling methods of waste tires were classified into burning [5] for energy recovery [4], pyrolytic conversions to gas [6], oil and chemicals [7–15], and material recycling by transforming used rubber [6] into products including tires [16, 17]. Although pyrolysis is regarded as one of the promising routes [16], co-pyrolysis of waste tire with biomass is also considered as influential method [18] to attain effective conversion of tires [19–25].

As an environmentally friendly recycling process for waste tire treatment, mechanochemical procedures are also gaining attention [26, 27] and reclamation of crushed tire scrap by mechanochemical procedure was reported [28].

However, they could not avoid reverse reaction because of the reactions without solvent [29].

We have been working on the chemical recycling of polymeric wastes containing tire in high-temperature fluids [30–34]. In the thermal treatment of them, we have confirmed that waste plastics including thermosetting resin such as phenol resin decomposed into their monomeric compounds [35–37] and solvent plays important roles chemically and physically [38, 39]. Liquid phase treatment of polymeric materials under mild conditions is green methods for effective and realistic transformation of wastes into valuable products [40].

In this study, to obtain fundamental information on the thermal/physical reaction of waste tires orienting life-time recycling, two kinds of tires were treated in high-temperature fluid using a 10 ml tubing bomb reactor.

Experimental

Materials

Two waste tire samples were used in this study. Waste tire A was prepared by cutting the tire into the small cubic particle as shown in Fig. 1a. Outer and inner parts were characterized separately after smashing to powders. Their elemental analysis values were different between inner part (C, 83.0%; H, 8.1%; N, 1.0%; S, 2.0%) and outer part (C, 58.6%; H,

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Fig. 1 Two waste tire samples used in this study: **a** waste tire A; **b** waste tire B; **c** 10 ml tubing bomb reactor

(a) waste tire A

□ outer part of waste tire A
○ inner part of waste tire A

(b) waste tire B

(c) 10 ml tubing bomb reactor

7.4%; N, 0.3%; S, 1.4%). Tire contains inorganic fillers such as carbon black and silica (SiO_2). Actually, the presence of silica was confirmed by X-ray fluorescence (XRF) analysis of the tire. Elemental analysis values indicated that outer part contains more silica than inner part.

Independently another waste tire as shown in Fig. 1b was cut as flakes and mixed fully, and used as waste tire B (C, 85.8%; H, 7.7%; N, 0.4%; S, 1.2%).

Solvents were used as received without further purification. They were Reagent Grade.

Characterization

Thermal characterization of tire samples before and after the thermal treatment was clarified by using Seiko SSC 5200 apparatus (TG/DTA). About 3–7 mg of the sample was measured at a heating rate of 10 °C/min under nitrogen atmosphere from RT to 600 °C or 1000 °C depending on the sample.

The organic products after the reaction were identified qualitatively by gas chromatography–mass spectrometry (JEOL, JMS—T100GC) and were analyzed quantitatively by gas chromatography with a flame ionization detector (FID) (GC-14A, Shimadzu, Column SE-30, OV-1).

Reaction of waste tires A and B

Thermal treatment of waste tires in high temperature in organic solvent was carried out using a 10 ml tubing bomb reactor as shown in Fig. 1c in which an infrared image furnace was used as the heater. In the typical reaction, 0.1 g of powdered tire sample and 1 ml of solvent were introduced in the 10 ml reactor. The thermal treatment was carried out at 150–430 °C for assigning time. After the thermal treatment, obtained contents in the autoclave were mixed with dissolving solvent such as diethyl ether and filtered by using glass filter. The obtained solid residue was dried and the dried residue was weighed to calculate solubilization as follows:

Solubilization (wt %) = 100 (0.1 − residue (g))/0.1.

Results and discussion

Characterization of waste tire samples

Waste tire A was separated into two parts, outer part and inner part. They show different thermal properties as shown in weight loss curves in Fig. 2. In the case of the outer part, sharp Differential Thermo-Gravimetry (DTG) peak was observed at 445 °C. The peak corresponds mainly to the weight loss of Styrene–Butadiene Rubber (SBR) [41, 42]. Shoulder peak was also observed at 380 °C which corresponds to the weight loss of Natural Rubber (NR) and/or Isoprene Rubber (IR). Even after the heating to 1000 °C, 30% was remained as the residue. It is carbonization products and carbon black. In the thermal characterization of the inner part of the waste tire A, one clear DTG peak at 415 °C was observed. The peak corresponds to the weight loss of Butadiene Rubber (BR) and Butyl Rubber (IIR), and the peak temperature in inner part was lower than that of the peak in the outer part. The difference of the TG peaks between inner and outer parts of the tire results in the difference of the rubber components between inner and outer parts.

Thermal characteristics of the waste tire B show two DTG peaks at 446 °C and 373 °C, as shown in Fig. 3. The thermal characteristic of the waste tire B was as expected, because the waste tire B (flakes) contains all part of the tire.

Reaction of outer part of waste tire A

Outer part of the waste tire A was treated in various solvent, because outer part is more stable thermally than inner part. In the thermal treatment in water at 430 °C for 2 h, solubility
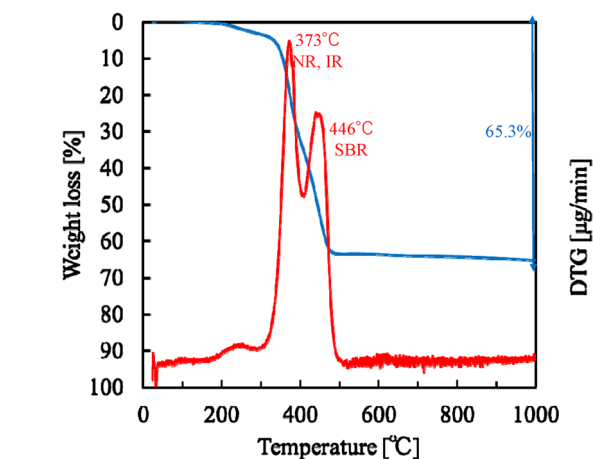

Fig. 3 TG/DTG curves of waste tire B

reached 56.8%. Water is superior solvent on the decomposition of polymeric materials at over 400 °C, because the high-temperature water promotes the cleavage of carbon–carbon bond via oxidation [32]. The production of monomeric compounds such as benzene and toluene were confirmed in the GC profile of the soluble matter in diethyl ether, as shown in Fig. 4a. Methanol was the better solvent than water and solubility increased to 68.7% at 430 °C for 2 h. At over 240 °C, sub- or supercritical methanol is expected, and high physical and chemical participation of methanol on the solubilization was considered. The same decomposition products with those in water were confirmed.

Tetrahydronaphthalene (tetralin) is well-known hydrogen-donor solvent. In the treatment of waste tire A in tetralin at 430 °C for 2 h, solubility reached to 77.5%. Production of naphthalene was confirmed by GC. It suggests the hydrogen donation of tetralin to radicals. In the
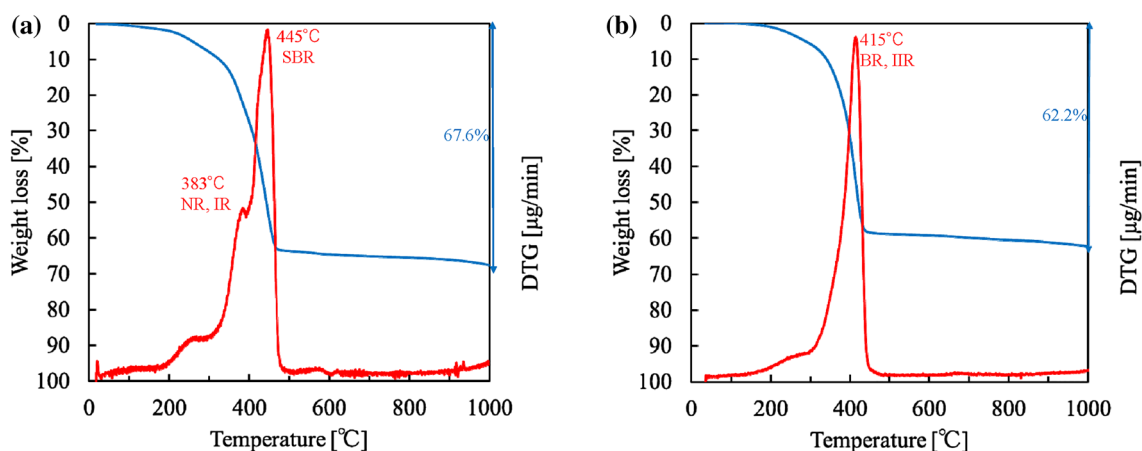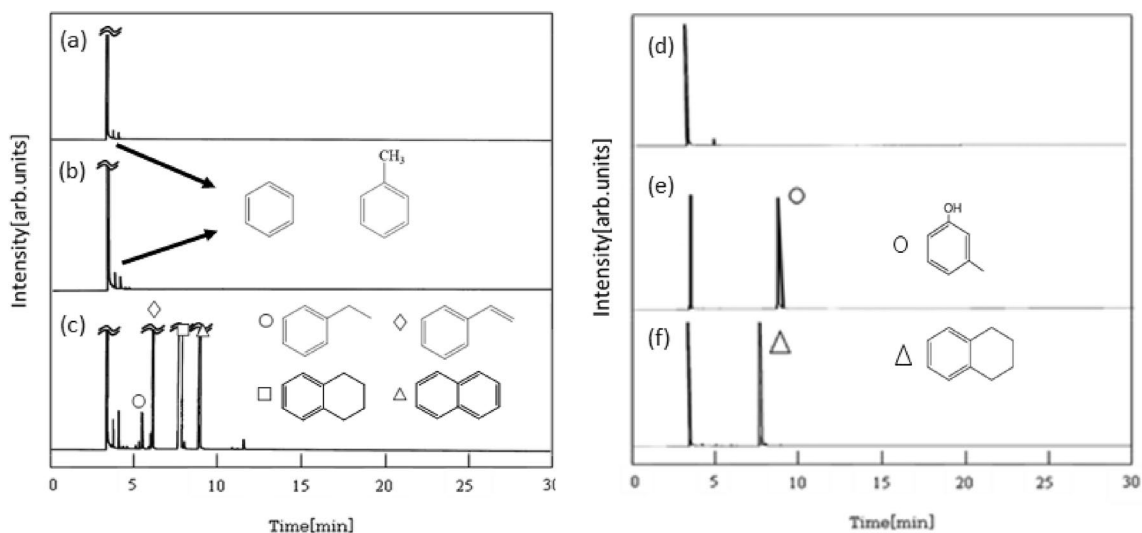

Fig. 2 TG/DTG curves of **a** outer part and **b** inner part of waste tire A. *SBR* styrene–butadiene rubber, *NR* natural rubber, *IR* isoprene rubber, *BR* butadiene rubber, *IIR* butyl rubber

Fig. 4 GC profiles of the products obtained in the thermal treatment of waste tire A at 430 °C in **a** water, **b** methanol, **c** tetralin, and waste tire B at 300 °C in **d** methanol, **e** *m*-cresol, and **f** tetralin

reaction in tetralin, conversion into monomeric compounds was also confirmed by GC, as shown in Fig. 4c.

To avoid the carbonization reactions occurred at high temperature, outer part of the waste tire A was treated at 300 °C for 2 h. Solubilizations in water and methanol were 7.2 and 10.8%, respectively. It suggests that they were not effective solvents in the treatment at 300 °C, because the cleavage of carbon–carbon bond was scarcely occurred.

Aromatic solvents were effective and solubilization reached to 99.8% in the treatment in tetralin. In this treatment, no production of naphthalene was confirmed indicating the occurrence of no hydrogen-donor reaction. Thermal characteristics of the filtrate at 600 °C indicated the presence of the residue, suggesting that the filtrate solution contains dispersed and suspended tire components such as silica and carbon black. Importance of physical interactions among the composition units in the tire matrix is well known. Such non-covalent interactions among the composition units [43, 44] might be broken by tetralin.

m-Cresol and phenol were also effective solvents on the solubilities and the values reached 75.2 and 73.8%, respectively. In these solvents, the small cubic particles were broken, and the residue was obtained as coarse particles. Sulfur-containing compounds were found in the filtrate. It suggests the occurrence of chemical reaction of sulfur-containing compounds at 300 °C, because bond dissociation energy of sulfur–sulfur bond is smaller than those of carbon–carbon bonds.

Table 1 Thermal treatment of outer part of waste tire A and waste tire B under inert atmosphere

Waste tire	Solvent	Temperature (°C)	Time (h)	Solubility (wt%)
A	Water	430	2	56.8
A	Water	300	2	7.2
A	Methanol	430	2	68.7
A	Methanol	300	2	10.8
A	Tetralin	430	2	77.5
A	Tetralin	300	2	99.8
A	*m*-Cresol	300	2	75.2
A	Phenol	300	2	73.8
B	None	200	6	0.1
B	Methanol	300	2	37.3
B	Methanol	200	6	42.7
B	*m*-Cresol	300	2	75.0
B	*m*-Cresol	200	6	56.4
B	Tetralin	300	2	89.1
B	Tetralin	200	6	41.4
B	1-Methylnaphthalene	300	2	90.8
B	1-Methylnaphthalene	200	6	86.8
B	1-Heptanol	300	2	69.9
B	1-Heptanol	200	6	66.8
B	1-Heptanol	200	24	65.5

Reaction of waste tire B

In the thermal treatment of waste tire A, participation of the solvent with the tire proceeds from the surface of the cubic particles. To avoid heterogeneous effects of solvents, tire flakes as waste tire B were used to evaluate the solvent ability.

To make clear the effective solvent on the solubilization, waste tire B was treated in various solvent at 300 °C avoiding carbonization reaction.

Flakes were more reactive than cubic particles and solubility reached 37.3% in the reaction in methanol. *m*-Cresol was more effective solvent than methanol, and the solubility reached 75.0%, as shown in Table 1. However, in the reactions in methanol and *m*-cresol, monomeric compounds were not observed in GC patterns of the products, as shown in Fig. 4d, e. Furthermore, solubility around 90% was obtained in the treatments in tetralin and 1-methylnaphthalene. Production of naphthalene was not confirmed in the reaction in tetralin, suggesting that the hydrogen donor was not important also in the thermal treatment of flake samples at 300 °C. Tetralin and 1-methylnaphthalene were superior solvent physically to disperse and suspend tire components such as silica and carbon black.

Thermal treatments were carried out at 200 °C to elucidate the effects of solvents on the solubilization of tire. At this temperature, no solubilization was observed in the reaction without solvent even by the treatment for 6 h. As shown in Figs. 5a and 3, thermal characteristics of the

Fig. 5 TG/DTG curves of **a** waste tire B and the residue after the thermal treatment of waste tire B at 200 °C for 6 h **b** without solvent and in **c** methanol, **d** *m*-cresol, **e** tetralin, and **f** 1-methylnaphthalene
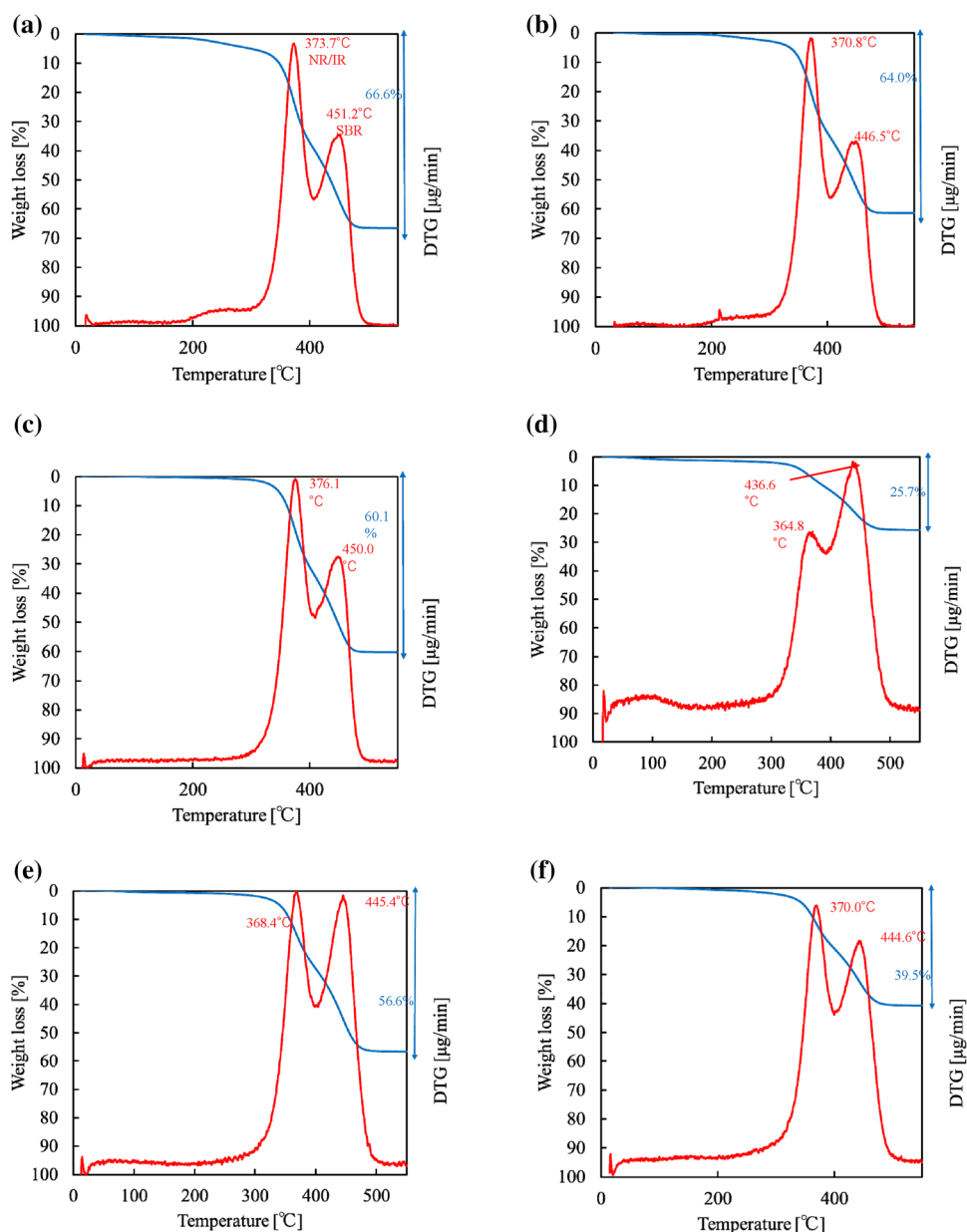
Fig. 6 TG/DTG curves of the residue after the thermal treatment of waste tire B at 200 °C in 1-heptanol for **a** 0.5 h, **b** 2 h, **c** 6 h, and **d** 24 h
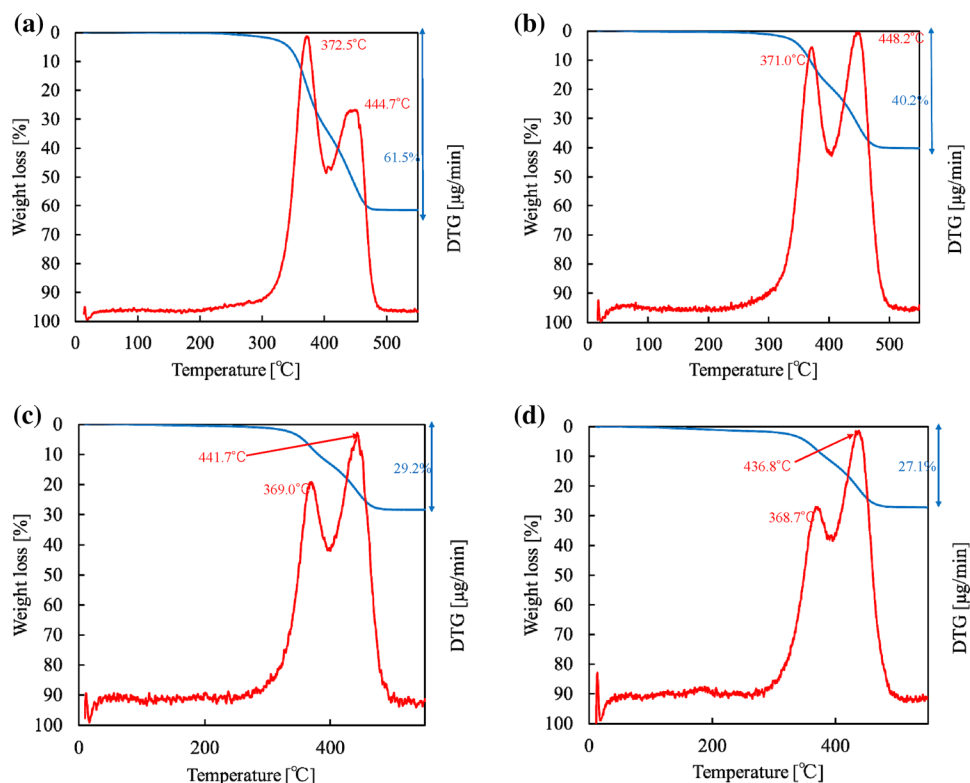
residue treated without a solvent were similar with that before the treatment.

It was confirmed that 42.7 and 56.4% of waste tire B was solubilized in the thermal treatment at 200 °C for 6 h in methanol and *m*-cresol, respectively. Thermal characteristics of the residue obtained in the thermal treatment in *m*-cresol are shown in Fig. 5d. The peaks at 365 °C and 437 °C correspond to the weight loss of Natural Rubber (NR) and/or Isoprene Rubber (IR) and Styrene–Butadiene Rubber (SBR), respectively. The ratio of NR/IR components to SBR was low compared to that before the treatment. It indicated that more NR and/or IR component was converted than SBR to filtrate.

Also 41.4% and 86.8% were solubilized in the thermal treatment in tetralin and 1-methylnaphthalene at 200 °C. Filtrates of them were black suspension of tire components. Certainly, in the thermal treatment in 1-methylnaphthalene, the ratio of NR and/or IR components to SBR as shown in Fig. 5f was larger than that obtained in *m*-cresol.

To make clear the role of *m*-cresol, 1-heptanol whose number of carbons is seven was used as the solvent. As shown in Table 1, high solubility as 66.8% was observed in the thermal treatment at 200 °C for 6 h. Longer treatment time, 24 h, was not effective to proceed further solubilization. However, thermal characteristics of the residue obtained in 1-heptanol indicated that NR and/or IR component reacted gradually with an increase in the reaction time as shown in Figs. 6a–d from 0.5 to 24 h.

Reaction of waste tire B in aliphatic alcohol

The number of carbons of 1-heptanol is the same with that of *m*-cresol; however, 1-heptanol is non-aromatic compound and acidity of 1-heptanol is fairly lower than that of *m*-cresol. These facts suggested that ionic reaction and π–π interactions were not important on the solubility.

Waste tire B was treated in 1-heptanol at 200 °C varying the treatment atmosphere and the post-treatment solvent as shown in Table 2. THF was the most effective dissolving solvent and solubility increased to 64.3% even by the treatment for 2 h. In these reaction conditions, solubilities between under argon and air were within experimental error and the effects of oxygen on the solubility were small.

Thermal treatment of waste tire B was carried out using various aliphatic alcohols, as shown in Table 3. Solubility increased with an increase in carbon number and maximum solubility; 64.3%, was observed in the treatment in 1-heptanol. Solubility at 250 °C was 25.0% in the treatment in 1-heptanol at 150 °C and increased to 76.8% in the treatment at 250 °C.

Reaction of sulfur-containing compound

In the thermal treatment of waste tire B at 200 °C, participation of sulfur on the solubilization reaction of tire was

Table 2 Thermal treatment of waste tire B in 1-heptanol at 200 °C

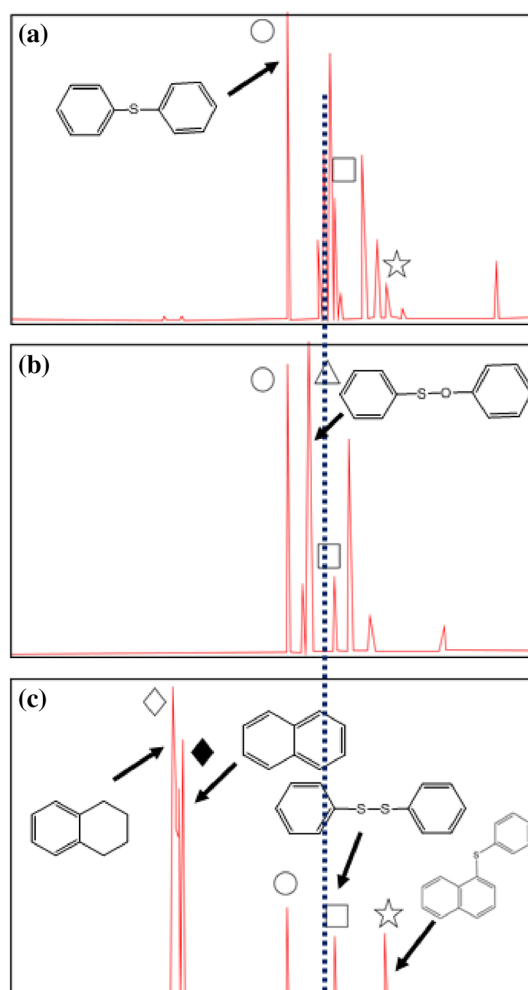| Time (h)	Post-treatment solvent	Atmosphere	Solubility (wt%)
0.5	Diethyl ether	Air	15.5
2	Diethyl ether	Air	32.1
2	Hexane	Air	46.4
2	THF	Air	64.3
6	Diethyl ether	Air	51.5
6	THF	Air	71.2
6	Diethyl ether	Ar	66.8
24	Diethyl ether	Air	82.8
24	Diethyl ether	Ar	65.5

Table 3 Solubilization of waste tire B in alcohol for 2 h

Solvent	Boiling point (°C)	Temperature (°C)	Atmosphere	Solubility (wt%)
Methanol	65	200	Air	13.4
1-Propanol	98	200	Air	26.3
1-Butanol	119	200	Air	51.7
1-Pentanol	138	200	Air	57.0
1-Hexanol	157	200	Air	60.6
1-Heptanol	176	150	Air	25.0
1-Heptanol	176	200	Air	64.3
1-Heptanol	176	250	Air	76.8
1-Octanol	196	200	Air	31.6
1-Decanol	231	200	Air	29.3

Fig. 7 Thermal treatment of diphenyl disulfide at 300 °C for 2 h in **a** *m*-cresol, **b** phenol, and **c** tetralin

suggested, because the bond dissociation energy of sulfur–sulfur bond is fairly smaller than that of carbon–carbon bond.

Diphenyl disulfide as the model compound of sulfur–sulfur bridge bond contained in the tire was treated in organic solvent at 300 °C for 2 h. In the treatment in *m*-cresol, the production of diphenyl sulfide was confirmed by GC–MS of the reaction products, as shown in Fig. 7. In the treatment in phenol, the production of diphenyl sulfide was also confirmed. By comparing the GC–MS profile with that obtained by the treatment in *m*-cresol, the production of the compound having S–O bond was supposed. The production of benzenethiol was confirmed by GC. In the treatment in tetralin, production of naphthalene was confirmed indicating the hydrogen-transfer reaction.

Disulfide (SS)–thiol (SH) exchange reaction is well known and the abundance of the S–S bond was determined using the exchange reaction [45]. The reaction products suggest the occurrence of the disulfide (SS)–alcohol (OH) exchange reaction, as shown in Fig. 8. The exchange reaction could cause S–S-bond scission resulting in the solubilization of the tire.

In the mechanochemical treatment of tire, reductive cleavage of S–S bond occurs because of the low-bond dissociation energy of sulfur–sulfur bond [46–48]. In the mechanochemical conditions, local high temperature is attained by the uneven temperature distribution. Such high temperature may cause bond scission reactions. On the contrary, liquid-phase treatment attains homogeneous cleavage of sulfur–sulfur bond.

Conclusions

Tire is heterogeneous composite materials and different thermal properties were observed between inner and outer parts. In the reaction of the outer part, high solubilization to monomeric compounds was attained in the hydrogen-donor solvent at 430 °C through stabilization after carbon–carbon bond cleavage reaction. Solubilization of waste tire flakes was attained by the thermal treatment at 300 °C in aromatic solvent such as tetralin and

Fig. 8 Possible reaction pathway of diphenyl disulfide in the thermal treatment at 300 °C in *m*-cresol or phenol
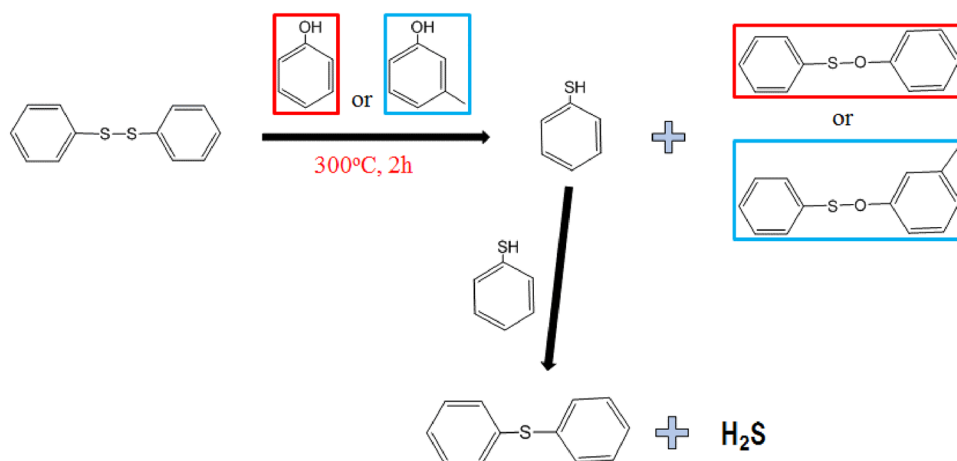
1-methylnaphthalene. In these solvents, solubilization occurred without devulcanization of the tire. Certainly, the solubilized products are inappropriate for raw materials of recycled tire because of a possibility of insufficient properties of the recycled tire by the heterogeneous mixing.

The effective solubilization was attained even at 200 °C in aliphatic alcohol. In the thermal treatment in alcohol, solubilization reached at near 70% by the devulcanization reaction. The products bring the prompt material for recycling tire by mixing with virgin raw materials.

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