

Physical properties and microstructure of bitumen with high content of ground tire rubber

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Abstract: In this work, ground tire rubber (GTR) was thermo-mechanically devulcanized in the planetary extruder and subsequently was used for preparation of bitumen modified with ground tire rubber (20–40 wt%). The physical and processing properties of prepared materials were characterized based on penetration, softening point and melt flow rate measurements. Fourier transform infrared spectroscopy, scanning electron microscopy, thermogravimetric analysis and volatile organic compounds' emission level were used for assessment of interfacial interaction between GTR and bitumen.

Keywords: waste tires, recycling, bitumen, devulcanization, planetary extruder.

Właściwości fizyczne oraz mikrostruktura asfaltu z dużą zawartością rozdrobnionych opon samochodowych

Streszczenie: W prezentowanej pracy rozdrobnione opony samochodowe poddano dewulkanizacji termo-mechanicznej w wylączarce planetarnej, a następnie użyto do wytworzenia asfaltu modyfikowanego gumą (20–40% mas.). Właściwości fizyczne i przetwórcze otrzymanych materiałów scharakteryzowano na podstawie pomiarów penetracji, temperatury mięknięcia oraz masowego wskaźnika szybkości płynięcia. Do oceny oddziaływań pomiędzy gumą a asfaltem zastosowano spektroskopię w podczerwieni z transformacją Fouriera, skaningową mikroskopię elektronową, analizę termogravimetryczną oraz pomiary zawartości lotnych związków organicznych.

Słowa kluczowe: zużyte opony, recykling, asfalt, dewulkanizacja, wylączarka planetarna.

According to European Tyre and Rubber Manufacturers' Association (ETRMA), in 2021 in European Union 3 123 353 tons of waste tires were generated, while 97% of waste were treated by material recycling and energy recovery [1]. For comparison, in Poland 252 600 tons of waste tires were generated and treated by: recycling – 45%, energy recovery – 30% and unknown/stock utilization – 25%. The level of unknown/stock utilization of

waste tires in Poland is the highest in Europe. This situation is due to the limited number of Polish companies active in the field of waste tires management and recycling.

Recently, Przydatek *et al.* [2] studied correlation between the generation, collection and recovery of waste tires considering an indicator of their mass accumulation per area in Poland. The results showed uneven accumulation of waste tires in Poland and significant differences between waste tire accumulation indicator at the national level – 2.12 Mg/km² and for one of the smallest voivodeships – 48.06 Mg/km² were observed. This confirms that waste tires accumulation indicator per country area might be a useful tool for proper selection of the locations for companies dedicated to collecting and recycling of waste tires.

Currently, the basic method of waste tires recycling is their mechanical disintegration by shredding, grinding and pulverization (~87.5% of all forms of recycling according to ETRMA) [3]. The obtained products can be categorized into three streams of materials: steel, textile cord, and ground tire rubber (GTR), which should be considered as a valuable source of secondary raw materials [4–6].

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Many attempts to apply GTR as low cost and environmentally friendly modifier of polymer composites [7, 8], concrete [9, 10] and bitumens [11, 12] have been made. However, a higher content of GTR in the composites filled with GTR usually results in the deterioration of their processing and mechanical properties. This is due to weak compatibility and limited interfacial adhesion between cross-linked GTR and fresh matrix, which can be improved by suitable treatment of GTR before use [13–15].

Reactive extrusion is the most promising method for devulcanization and/or modification of GTR [16–18], which allows making steps forward to further developing rubber recycling and upcycling technologies.

In this field of research, application of devulcanized GTR as modifier of bitumens, especially bitumen highly

modified with GTR (content of modifier more than 20 wt%), is gaining more attention [19, 20]. Recent works indicate that such systems can be characterized by self-healing behavior [21] and can be used in additive manufacturing technologies [22, 23], therefore their further development is fully justified.

In this work, ground tire rubber was processed by a planetary extruder and then devulcanized GTR (dGTR) was used to prepare ground tire rubber modified bitumen. Physical properties and microstructure as function of dGTR content were determined using penetration, softening temperature, penetration index, melt flow rate, Fourier transform infrared spectroscopy, scanning electron microscopy and volatile organic compounds emission measurements.

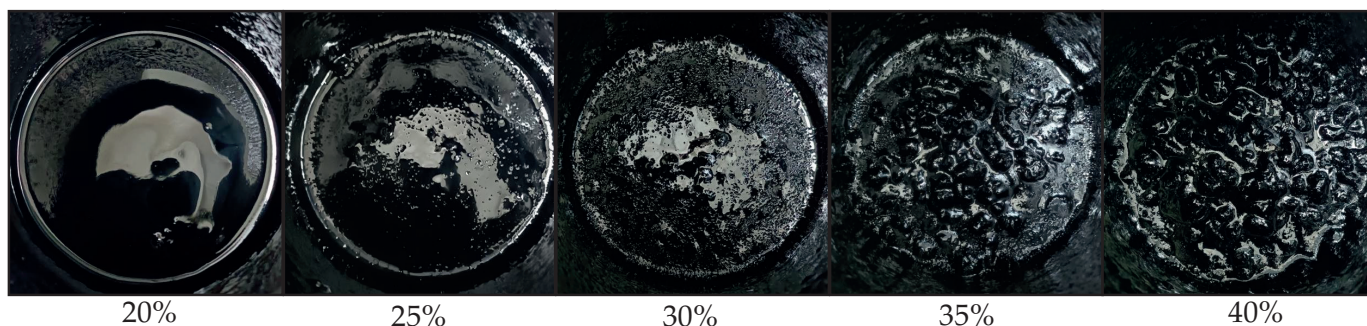
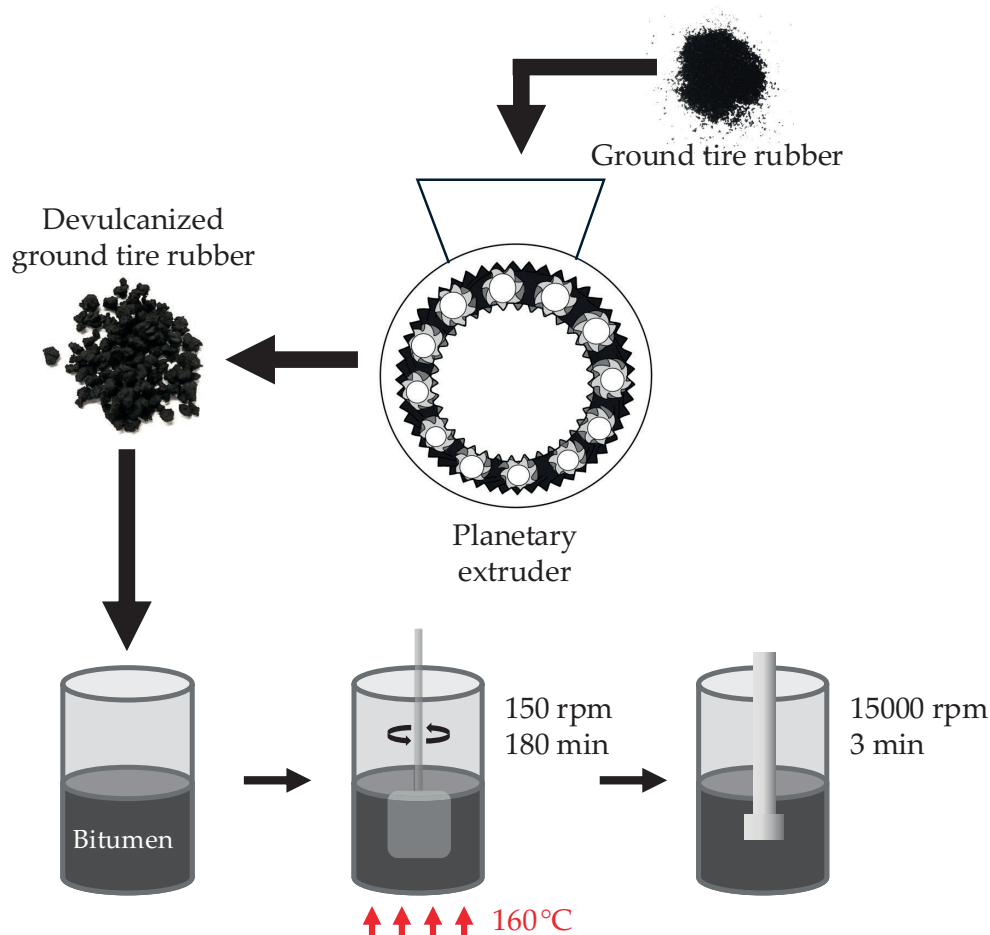


Fig. 1. Sample preparation scheme and appearance of bitumen modified with dGTR

EXPERIMENTAL PART

Materials

GTR with particle size up to 0.6 mm, obtained from car and truck tires mix was supplied by Grupa Recykl S.A. (Śrem, Poland). According to the technical data sheet, GTR particle size distribution determined by percentage of rubber powder remaining on the sieve is: <0.6 mm – 0.4%; 0.6 mm – 2.5%; 0.5 mm – 33.0%; 0.4 mm – 28.4%; 0.3 mm – 19.0%; 0.2mm – 15.5% and 0.1 mm – 1.2%.

Bitumen 50/70 (B50/70) from Rafineria Gdańska Sp. z o.o. (Gdańsk, Poland), which according to producer is characterized by penetration at 25°C in the range of 50–70 (0.1 mm) and softening point temperature in the range of 46–54°C.

Sample preparation

Rubber devulcanization

Lab-size planetary extruder model PLATEX 80 produced by Takimsan Disli Kesici Ltd. Sti. (Istanbul, Türkiye) was used for thermo-mechanical devulcanization of GTR. Reactive extrusion of GTR was performed at output 24 kg/h. Obtained devulcanized GTR (dGTR) was characterized by Mooney viscosity ML(1+4)100°C and is equal to 82, which fits well to values described in the literature for commercially available reclaimed rubbers [24].

Preparation of bitumen modified with dGTR

In this study, 50/70 bitumen was stirred with devulcanized rubber with a Ministar 80 control overhead stirrer produced by IKA® (Staufen im Breisgau, Germany) with a speed of 150 rpm for 180 min at 160°C (temperature of bitumen, the hotplate was set to 300°C). The samples were then sheared with an IKA® T 25 digital Ultra-Turrax® disperser with a speed of 15000 rpm for 3 min. Amounts of modifier in obtained samples were 20 wt%, 25 wt%, 30 wt%, 35 wt% and 40 wt%. Fig. 1 shows a diagram of the modification process and the appearance of the obtained products depending on the content of devulcanized GTR.

Methods

Penetration measurement for the tested samples was performed in accordance with PN-EN 1426 at a temperature of 25°C. The softening point was determined in accordance with PN-EN 1427 using the ring and ball method. The penetration index (PI) was calculated in accordance with equations 1 and 2:

$$PI = \frac{20(1 - 25A)}{1 + 50A} \quad (1)$$

$$A = \frac{\log 800 - \log(\text{pen}T_{25^\circ\text{C}})}{T_{\text{softening}} - T_{25^\circ\text{C}}} \quad (2)$$

The melt flow rate (MFR) was determined based on ISO 1133 at a temperature of 50°C and a load of 15 kg. A non-standardized capillary with the following dimensions was used: diameter – 4.195 mm and height – 8 mm. Fourier transform infrared spectroscopy (FT-IR) analysis was performed using IRTracer-100 from Shimadzu (Kyoto, Japan) equipped with a single reflection ATR accessory and a germanium crystal prism. Measurements were performed in attenuated total reflection mode at a resolution of 4 cm⁻¹ and 32 scans in the wavenumber range of 4000–500 cm⁻¹. Morphology was assessed using a FlexSEM 1000 II scanning electron microscope from Hitachi (Tokyo, Japan) using an accelerating voltage of 20 kV. Before measurement, the samples were coated with a thin layer of gold to increase their conductivity in a vacuum chamber using a 108 Auto Sputter Coater from Cressington Scientific Instruments (Watford, UK). Thermogravimetric analysis (TGA) was performed using a Netzsch TG 209 apparatus from Netzsch Group (Selb, Germany). The samples of approximately 10 mg were investigated in the temperature range of 25–650°C and under a nitrogen atmosphere at a heating rate of 10°C/min. Total volatile organic compounds' (TVOCs) emissions from studied materials were determined using microscale stationary emission chamber - Markes' Micro-Chamber/Thermal Extractor™ – μ-CTE™ 250 (Markes International Ltd., Llantrisant, UK) in which samples of approx. 1 g were conditioned for 15 min at 30°C. Volatile organic compounds emitted from the modified GTR were collected using stainless steel tubes filled with Tanax TA as a sorption bed (Markes International Ltd., Llantrisant, UK) under the influence of a carrier gas (nitrogen) with a rate of 12 mL/min. Then, the adsorbed analytes were released through a two-stage process of thermal desorption (Markes Series 2 Thermal Desorption Systems; Unity/TD100, Llantrisant, UK). Quantitative analysis of TVOCs released from modified GTR was performed on a GC-FID system (Agilent 7820A GC, Agilent Technologies, Inc., Santa Clara, California, USA), while qualitative analysis was performed on a gas chromatograph (Agilent Technologies 6890) combined with a mass spectrometer (5873 Network Mass Selective Detector, Agilent Technologies). More detailed information about the equipment used and methodology is presented in the literature [25, 26].

RESULTS AND DISCUSSION

The penetration and softening point of bitumen modified with dGTR as function of modifier content are presented in Fig. 2a and 2b. As could be expected, the higher content of dGTR resulted in decreased penetration from range of 50–70 characteristic for pure bitumen 50/70 to 24.4 for bitumen with 20 wt% dGTR and 5.5 for bitumen modified with 40 wt% dGTR. Moreover, it was observed that for content of dGTR higher than 35 wt% the effect on penetration of bitumen highly modified with GTR was negligible. The softening tem-

Table 1. Comparison of processing and mechanical properties of GTR modified asphalts described in the literature

GTR content, wt%	Bitumen modification conditions	Physical properties		Ref.
		Penetration, 0.1 mm	Softening point, °C	
20–40	Temperature: 160°C Time of mixing: 180 min	5.5–24.3 50–70	65.7–92.7 46–54	This study
15–35	Temperature: 170–230°C Time of mixing: 180–225 min	~35–55* 67.0	~62–78* 48.9	[27]
22.5	Temperature: 177±10°C Time of mixing: 30–90 min	60.3–63.9 59.3	29.7–34.3 48.0	[28]
20–50	Temperature: 180–190°C Time of mixing: ~75 min	44–90* 65.4	~67–73* 49.9	[29]
35	Temperature: 190°C Time of mixing: ~300 min	60.5 63.9	71.2 49.9	[30]
15–40	Temperature: 180°C Time of mixing: 60 min	70.8–81.6 67.2	56.1–93.8 47.0	[31]

* Values estimated from graphs.

temperatures of all evaluated samples was higher than the 46–54°C range typical for B50/70, increasing almost linearly with higher content of modifier and for bitumen modified with 40 wt% dGTR reached 92.7°C. For better understanding, the physical properties determined for the studied materials were compared with results obtained by other research groups and summarized in Table 1. As can be noticed, bitumens modified with GTR are usually prepared in temperatures in the range of

170–230°C and mixed in the time range of 60–300 min. The content of GTR in systems described in the literature varies between 15–50 wt%. Literature data indicates that the penetration of bitumen modified with GTR is in the range of 35–90, while softening point is in the range of 29.7–93.8°C. Compared to the literature data, bitumens modified with ground tire rubber prepared in this study were characterized by lower penetration and similar range of softening point.

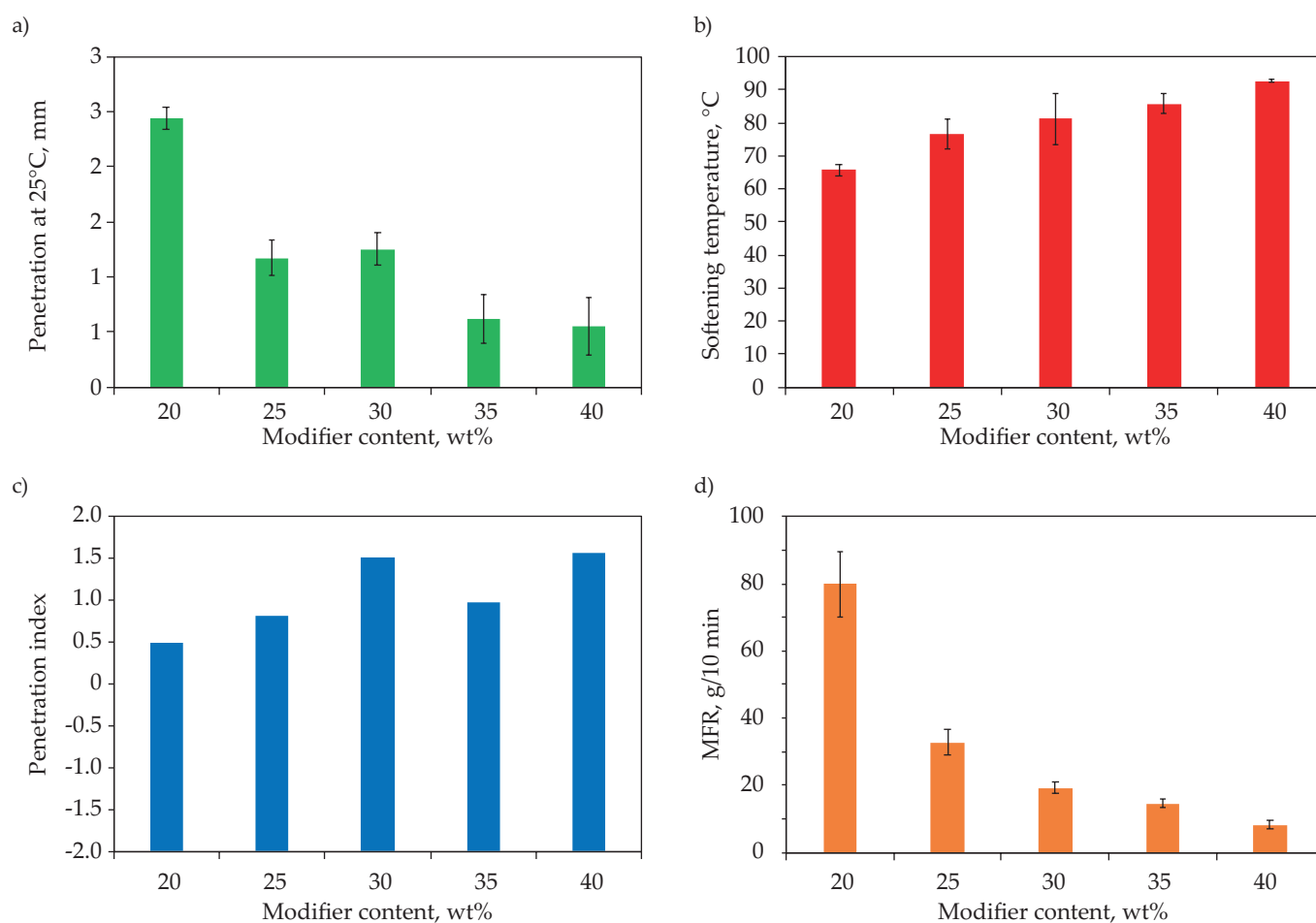


Fig. 2. Physical and rheological properties of bitumen modified with GTR: a) penetration, b) softening temperature, c) penetration index, d) MFR

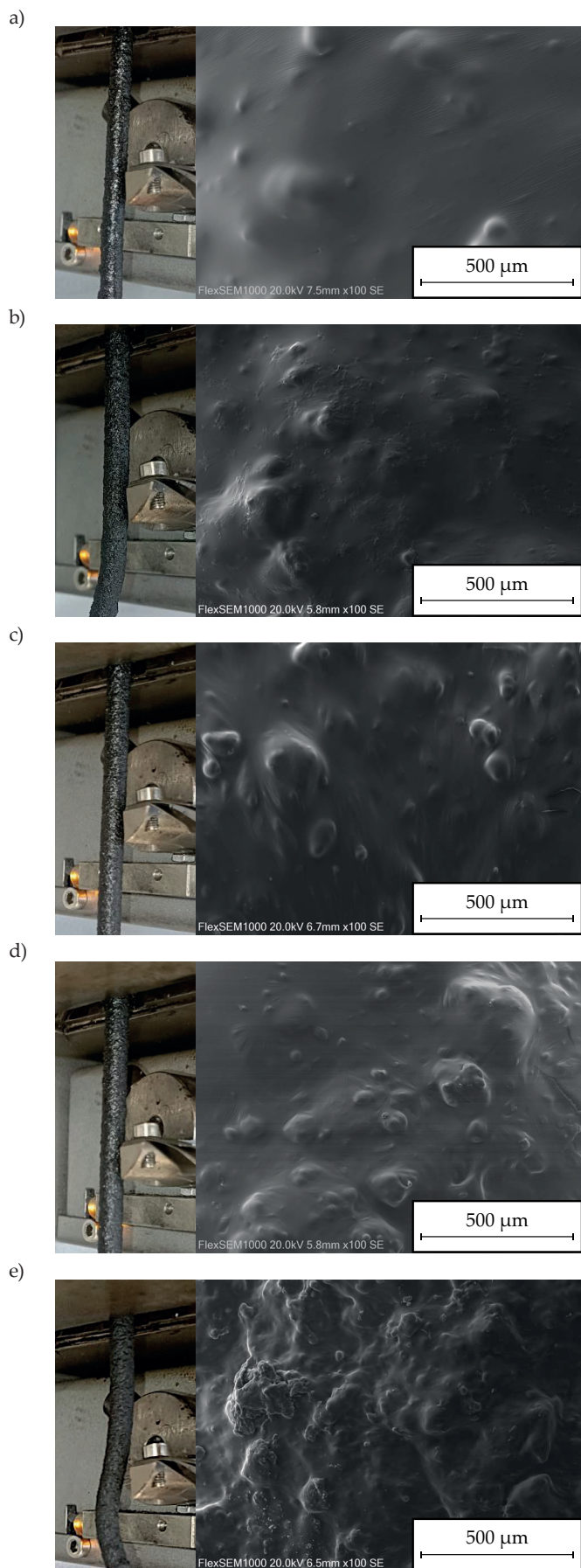


Fig. 3. SEM images of bitumen modified with dGTR: a) 20 wt% dGTR, b) 25 wt% dGTR, c) 30 wt% dGTR, d) 35 wt% dGTR, e) 40 wt% dGTR; magnification 100×

As can be observed in Fig. 2c, penetration index for samples of bitumen modified with dGTR was higher than for pure B50/70. Liu *et al.* [32] showed that application of 20 wt% of GTR to bitumen increased penetration index from -1.2 for pure bitumen (penetration: 64.7; softening point: 49.7°C) to 0.3274–0.6876 depending on waste tire source and particle size. For comparison in this study, for bitumen modified with 20 wt% of dGTR penetration index was 0.4794, which fits well the values described in the literature. Increase of penetration index indicates a shift from sol to gel structure in bitumen matrix [33]. This is due to the swelling and dissolution of dGTR into bitumen matrix, resulting in the higher viscosity of studied systems.

From Fig. 2d, the MFR for bitumen with 20 wt% of dGTR was 79.9 g/10 min, while that for the 40 wt% modified bitumen was 8.1 g/10 min. As can be seen, the MFR decreased significantly for bitumens with modifier content higher than 20 wt%, which is caused by the decrease in the swelling and dissolution efficiency of dGTR in the bitumen matrix.

Figure 3 shows the microstructure of bitumen as a function of dGTR content assessed by SEM. During the studies, the morphology of the surface formed after MFR measurements was examined. It was observed that a higher dGTR content in the bitumen/dGTR systems tested resulted in a higher content of undissolved dGTR particles, which led to lower fluidity of the prepared materials (Fig. 2d). The average size of dGTR particles dispersed in the bitumen matrix was in the range of 50–200 μm, while the diameter range of dGTR before use was 1–10 mm.

Fig. 4a shows FT-IR spectra of pure bitumen 50/70 (B50/70) and bitumen modified with dGTR. The bands in the range of 2960–2850 cm^{-1} are attributed to the symmetric and asymmetric stretching vibrations of C–H bonds in CH_2 groups and $-\text{CH}_3$ end groups present in bitumen. Signal around 1458 cm^{-1} is related to scissor vibrations of C–H bonds in CH_2 groups, while 1375 cm^{-1} is characteristic to the vibrations of C–H bonds in $-\text{CH}_3$ end groups [34]. Similarity of FT-IR spectra for pure bitumen and modified bitumen indicating good encapsulation rubber particles by bitumen matrix and confirms the physical interactions.

Furthermore, it was observed that the thermal stability defined as 2% weight loss ($T_{2\%}$) determined for neat B50/70 bitumen was 305.6°C, while for 20 and 40 wt% dGTR modified bitumen it was 306.3°C and 287.6°C, respectively (Fig. 4b). This indicates that the thermal stability of dGTR modified bitumen decreased with increasing rubber swelling efficiency and degradation into bitumen matrix. Ren *et al.* [35] reported similar finding. DTG curves showed that degradation occurred in one step, indicating good compatibility between dGTR and bitumen. T_{max} from DTG curve for neat B50/70 was 448°C, while addition of 20 wt% and 40 wt% dGTR resulted in increase of T_{max} to 454.6°C and 450.5°C, respectively. This is due to the presence of carbon black in the ground tire rubber.

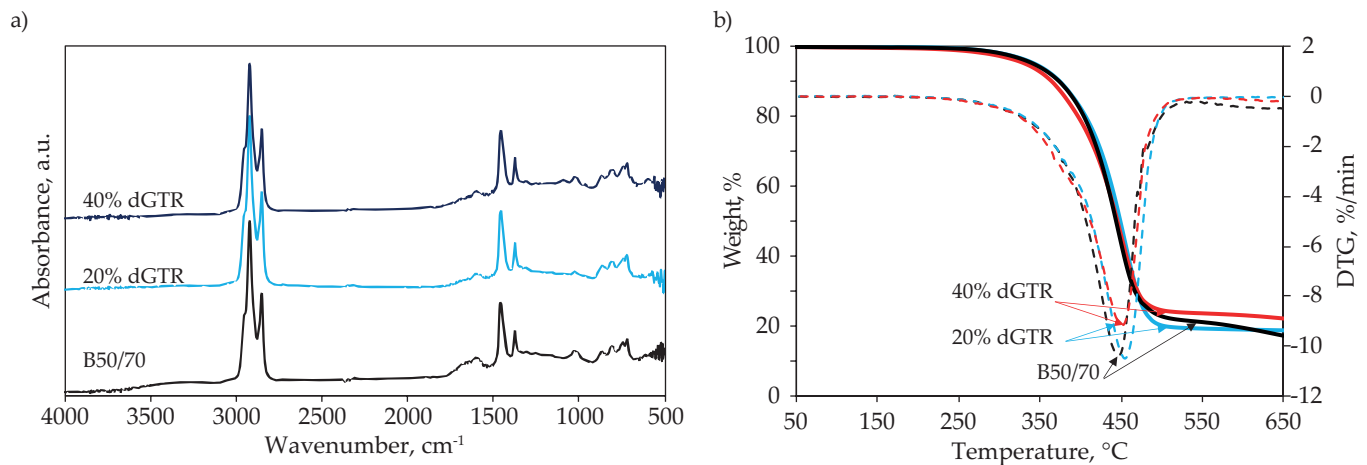


Fig. 4. FT-IR spectra (a) and TGA/DTG curves of bitumen/dGTR (b)

Characterization of volatile organic compounds emitted during preparation of ground tire rubber modified bitumens is a serious environmental problem and a new field of research [36–38]. Knowledge about emission profile from bitumen modified with ground tire rubber as function of composition or processing conditions allows suitable design of neutralization and deodorization protocols for such systems. Fig. 5 presents measurements of total volatile organic compounds (TVOCs) and sepa-

rate compounds (toluene, xylene, and ethylbenzene) for pure bitumen and bitumen modified with dGTR. It was found that TVOCs emission for bitumen with 20 wt% of dGTR increases by 10% compared to the pure bitumen. On the other hand, bitumen modified with 40wt% of dGTR emission level increased more than twice, which is related to degradation of dGTR. The results showed that bitumen with 20 wt% of dGTR emitted more than twice as much toluene, 17× more xylene and almost 10×

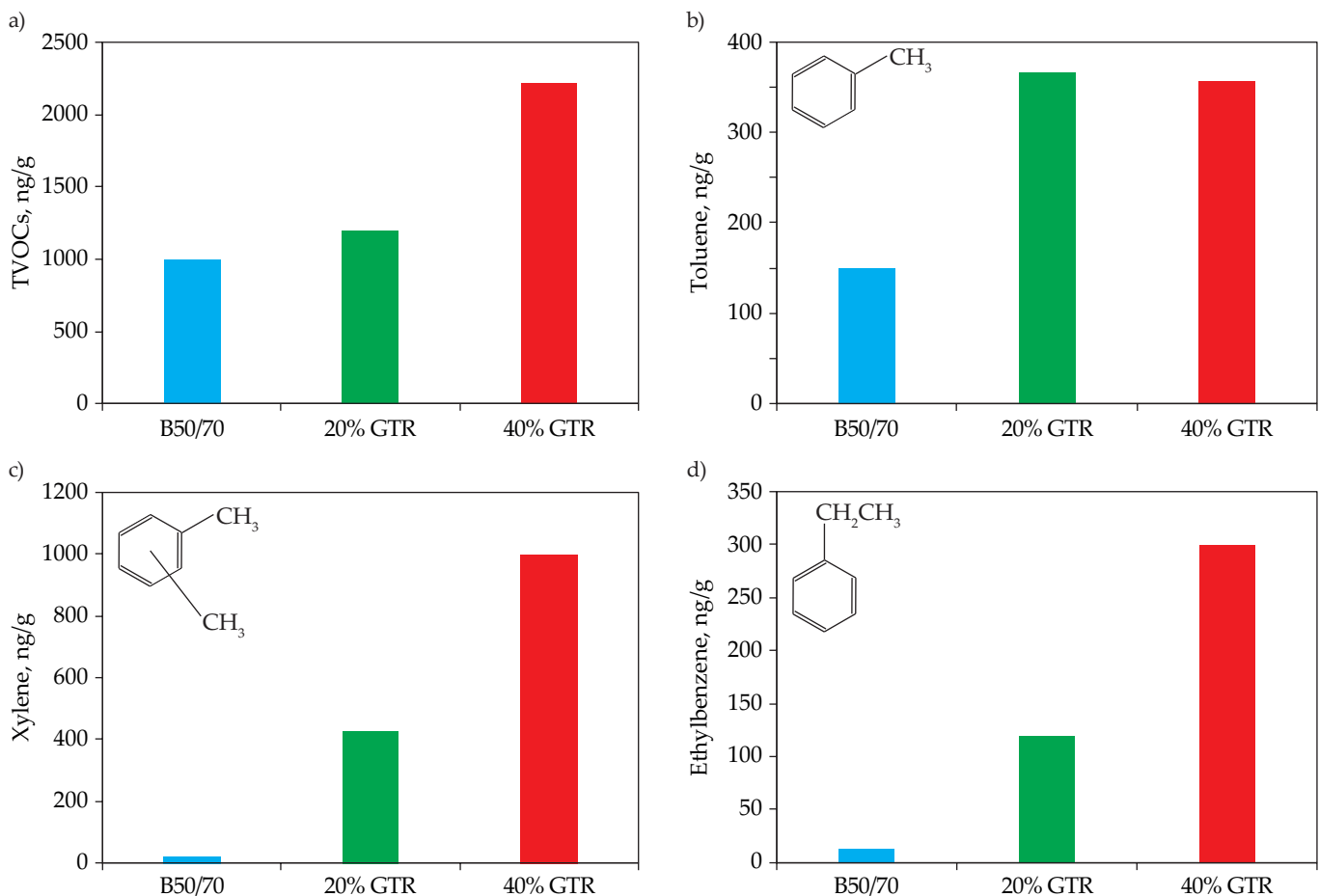


Fig. 5. Volatile organic compounds emitted from bitumen modified with dGTR: a) TVOC's, b) toluene, c) xylene, d) ethylbenzene

more ethylbenzene than the pure bitumen, while bitumen with 40 wt% of dGTR emission level was 2× toluene, 40× xylene and 24× ethylbenzene compared to unmodified bitumen.

CONCLUSIONS

In this work, ground tire rubber (GTR) was thermo-mechanically treated using a planetary extruder, which resulted also resulting in devulcanized GTR (dGTR) characterized by Mooney viscosity ML(1+4)100°C equal to 82. dGTR was successfully used to prepare highly modified bitumens with modifier content in the range of 20–40 wt%. It was found that increasing content of dGTR resulted in the increase of softening point and significant decrease of penetration. As could be expected, the flowability of bitumen modified with ground tire rubber defined by MFR decreased with increased content of dGTR. This is related to the increasing content of undissolved dGTR particles with a diameter in the range of 50–200 μm (SEM). FT-IR confirms good encapsulation of dGTR particles by bitumen matrix. Thermal stability decreased with higher content of dGTR and simultaneously the total volatile organic compounds emission level increased. Higher content of dGTR in bitumen/dGTR system resulted in higher emission of xylene and ethylbenzene, while the changes of toluene emission level were negligible.

The obtained results indicate that tested materials are more like bituminous masses than conventional road bitumens, and therefore can be used as sealants and adhesives. In addition, the MFR indicates that the tested materials can be a good basis for the development of additive manufacturing technologies in construction and building materials. Considering the current trends in the field of bitumen and road pavements modified with ground tire rubber, further research should focus on:

- application of ground tire rubber with different devulcanization degree and its combination with other additives (e.g., thermoplastics, oils, curing agents. etc.) to increase the compatibility and homogeneity of bitumen/GTR systems;
- reduction of time and energy applied during bitumen modification (e.g., new mixing devices or protocols, application of thermoplastic elastomers based on waste tire rubber, etc.);
- quantitative and qualitative analysis of volatile organic compounds emitted during preparation, storage and usage of ground tire rubber modified bitumens – investigation of their impact on the environment and human health (e.g., new methodology, standardized protocols, etc.);
- assessment of the storage stability and accelerated aging of bitumens modified with GTR, which is necessary for industrial scale application.

Authors contribution

J.Z. testing, data curation, investigation, visualization, writing-original draft; C.S. – testing, data curation, investigation,

visualization, writing-original draft; S.W. – data curation, formal analysis, writing-original draft; K.F. – research concept, methodology, testing, data curation, formal analysis, investigation, validation, visualization, writing-original draft.

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Conflict of interest

The authors declare no conflict of interest.

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