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ORIGINAL PAPER

Tire-derived reclaimed rubber as a secondary raw material for rubber foams: in the framework of circular economy strategy

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Abstract

Improper disposal and accumulation of waste tire rubbers have posed a serious threat to the development of a circular economy, a sustainable environment, and human health. In light of the drawback of the current waste management of waste tires, the recycling and transformation of reclaimed rubber (RR) into valuable end products has received significant attention from industries and the academic field. Herein, we propose a facile method to reuse RR in developing closed-cell elastomeric foams based on ethylene propylene diene rubber (EPDM). Rheometry results revealed that the introduction of RR up to 20 phr, increased the cure rate from 11.7 to 13.48%/min, reduced curing time from 12.21 to 9.3 min and also increased ultimate torque from 6.51 to 7.24 N.m. Morphological studies indicated that the RR increased the cell density from 12 to 78 cell/mm^3 and reduced the number average cell size from 940 to 110 µm. The mechanical results indicated that the introduction of RR could be a feasible alternative for the fabrication of high-performance EPDM foams with improved hardness and resilience. By increasing RR content of EPDM/RR foams, the relative density and cell density of EPDM/RR foams increased, while cell size decreased. The introduction of 10 phr of RR, increased the hardness and resilience of the EPDM foam by 37 shore A and 68%, respectively. The research verified that the attempt to use RR to produce a good foam structure was found to be successful. The results open a way for EPDM/RR foam composites to be applied for sealing and gasket industries as an eco-friendly replacement for virgin products.

Highlights

- Use of reclaimed rubber from waste tires as secondary raw material for EPDM rubber foams
- Tire-derived reclaimed rubber/EPDM closed-cell foams support the circular economy of waste tires
- Tire-derived reclaimed rubber/EPDM closed-cell foams exhibit superior mechanical properties at the low cost

Keywords Reclaim rubber · Foam · EPDM · Foaming agent

Introduction

The circular economy (CE) model emphasizes closing use loops of the material by reusing, repairing, restoring, and recycling resources to retain material value [1]. In theory, CE builds upon and goes beyond waste management initiatives to an integrated systems perspective addressing both production and consumption practices maximizing the functional utility of materials [2]. Although the tire industry makes a significant contribution to the world rubber industry, the tremendous discharge of waste tires due to the expansion of the automobile industry is an issue faced by all countries which has raised significant environmental and economic challenges globally [3]. However, one effective and environmentally friendly strategy to deal with the problem is to reclaim and reuse these solid waste rubbers. Reclaiming the cross-linked rubber converts to rubber compounds that can be reprocessed; re-cross-linked and reshaped [4]. Studies have recommended the use of reclaimed rubber (RR) for reinforcing and improving the properties of rubber composites [5]. This emerges as a breakthrough in improving the

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efficient use of RR as an additive for virgin rubber, which in turn would preserve natural resources and the environment [6, 7].

One of the effective options to use RR as reinforcement is to introduce it into rubber foams to compensate for rubber foams' inherent low modulus and strength that cannot be improved just by using chemical modifications [8, 9]. Analysis of the GHG savings and cost-effectiveness of the asphalt pavement climate mitigation strategies revealed that the incorporation of RR is the most environmentally (− 1.6% GHG) and economically (5.9% cost saved) viable technology [10]. The implementation of RR in rubber foams would also provide a large greenhouse gases (GHG) emissions and costs saving potential related to the manufacturing and application of virgin elastomers [11, 12]. to be the most. Rubber-based foams have exhibited huge potential for practical applications due to their phenomenal complementary properties including low weight, low cost of raw material and energy consumption, exceptional strength/weight ratio, and thermal and acoustic barrier properties [13–15]. Rubber foams are expanded elastomeric materials generally fabricated using two types of foaming agents including physical ones and chemical ones, and each group has its pros and cons [16]. Morphologically, foams can be classified into two categories based on their pore structure; the open-cell structured foams and the closed-cell foams. The structure of cells in closed-cell foams provides higher dimensional stability and strength compared to open-cell structured foams while having a lower moisture absorption coefficient [14, 17–19].

Ethylene propylene diene monomer (EPDM) is one of the most widely used elastomers in the fabrication of rubber-based foams presenting appropriate mechanical properties, and high resistance to ozone, weathering, UV, and aging. On the other side, they are also characterized by a weak resistance to polar oils and fluids. In the framework of the rubber industry, a wide variety of successful applications have been proposed for EPDM foams such as conveyor belts, electrical insulation of cables, window seals, waterproofing membranes, and gaskets [20].

From the circular economy perspective, the incorporation of RR in the rubber foam industry is a re-utilization alternative that contributed to tire waste reduction. Recently, there is an ever-growing interest in the efforts to develop green rubber foam from RR [21, 22]. Mahmood et al. studied the effect of epoxidized natural rubber/RR blend ratio on the cell structure and physical performance of fabricated rubber foam [23] as an efficient energy absorbent. The incorporation of RR into the polyurethane foam and natural rubber/styrene-butadiene rubber blend foam was evaluated following the aim of decreasing ecological footprint [24, 25]. However, to the best of the authors' knowledge, no research has developed RR-reinforced EPDM foams to date. As a result, the aim of this work is to investigate the feasibility of reinforced EPDM foams produced from RR and their ability to provide high-performance end products. In this survey effect of RR content has been studied on the curing, morphological, thermal, and mechanical properties of EPDM/RR closed-cell foams. Furthermore, to fulfill the industrial requirement on foam processing procedures, the effect of various factors controlling the relative foam density of the products was investigated.

Experimental

Materials

EPDM K270 with a density of 0.86 g/cm^3, containing 57% ethylene and 38.5% propylene and Mooney viscosity of 71 (ML (1 + 4) at 100 °C) was purchased from Korean Kumho Polychem company. Reclaimed rubber (RR), UCD 103, with a density of 1.14 g/cm^3 and Mooney viscosity of 60 (ML (1 + 4) at 100 °C) was supplied from Union Commercial Development Co. Ltd., Thailand. The chemical blowing agent of Azodicarbonamide (ADC) was provided by Otsuka Chemical Company, Japan. The curing package including mercaptobenzothiazole disulfide (MBTS) and N-cyclohexyl-2-benzothiazole sulfonamide (CBS) as accelerators, sulfur as a curing agent, and zinc oxide and stearic acid as activators were obtained from Bayer Company, Germany. Carbon black N-330 grade and paraffin oil were of commercial grades.

Compounding of EPDM/RR with different amounts of RR

Prior to compounding, the RR was kept in a vacuum oven at 100 °C for 2 h in order to remove any trace of moisture. The mastication of EPDM was performed at ambient temperature via a two-roll mill (Polymix 200L, Schwabenthan, Germany) and then the different content of RR (0–20 phr) was added into the mixer and compounding continued for 5 min. Afterward, all the premixed ingredients were added to the compound and mixed for another 5 min. The speed of the two-roll mill in all the above steps was kept at 30 rpm. Table 1 shows the formulations and sample code notations employed for coding EPDM/RR compounds.

Foam fabrication

Prior to molding, all the prepared rubber compounds were stored at ambient temperature overnight to release the residual stress of rubber molecules built up in the polymer macromolecular during the mixing process. The cylindrical mold with dimensions of 3 cm in diameter and 1.3 cm in thickness was heated to the curing temperature, 150 °C, and stored at the same temperature for 30 min. Then, 5 g of compounds were placed in the mold inside a hot press. Compressing was continued at curing temperature

Table 1 Formulation of the prepared EPDM/RR compounds

Sample Code Component	R0	R5	R10	R15	R20
EPDM	100	100	100	100	100
Sulphur	2	2	2	2	2
Zinc oxide	5	5	5	5	5
Stearic acid	2.5	2.5	2.5	2.5	2.5
MBTS	1.2	1.2	1.2	1.2	1.2
CBS	0.5	0.5	0.5	0.5	0.5
ADC	6	6	6	6	6
Carbon black	10	10	10	10	10
Oil	5	5	5	5	5
Reclaimed rubber (RR)	0	5	10	15	20

* All the contents are given in per hundred rubber (phr)

under the pressure of 50 bars for 30 min to prepare the EPDM/RR foams specimens for further characterization.

Characterization

The cure characteristics of foams were studied through a rotational rheometer (Zwick 4308, Germany) at 150 °C (according to ASTM D2084 standard). The cellular structure images of foam cells were investigated by an optical microscope (SZX2-ILLD, Tokyo, Japan) in order to analyze the cell parameters like average cell size and cell size distribution using Image J software. The compression tests were performed via Hiwa-200, Iran in accordance with ASTM D412. The Shore A hardness of the samples was measured by a Zwick-3100 hardness tester, Germany, as per ASTM D2240. The rebound resilience data were determined using a Frank GMBH elasticity tester, Germany, according to ASTM D1054.

Result & Discussion

Curing behavior

Evaluation of the curing process of the rubber compounds is a critical requirement for optimizing foam expansion, and cell morphology ensuring favorable physical and mechanical properties for the foams. Figure 1 shows the effect of various content of RR on the cure behavior of EPDM foams obtained from the oscillating disc rheometer measurements at 150 °C. The curing reaction parameters necessary to evaluate the curing process were also calculated. Equation 1 gives delta torque (ΔM), the difference between the ultimate and initial torque which is related to the crosslink density of the rubber.

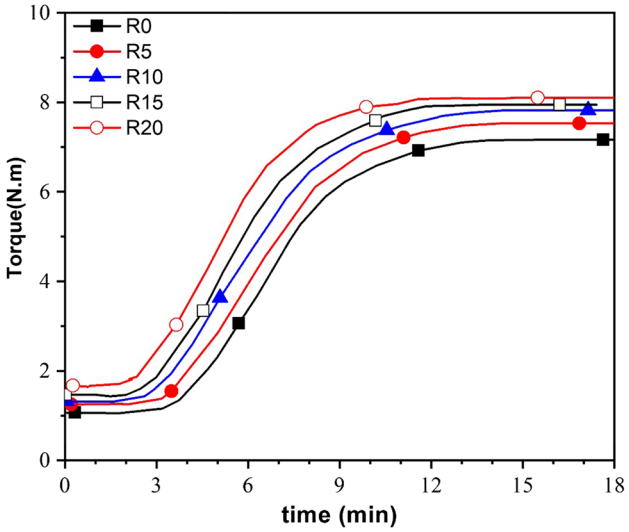

Fig. 1 Effect of RR content on foaming behavior of EPDM/RR foams at 150 °C

$$\Delta M = M_U - M_I \tag{1}$$

where M_I and M_U are respectively the initial and ultimate torques which indicate the stiffness (or shear modulus) of the unvulcanized rubber and fully-vulcanized rubber. M_{90}, the torque when the cure achieves 90% of its maximum is calculated using Eq. 2 [26]:

$$M_{90} = M_I + 0.9\Delta M \tag{2}$$

The cure rate index (CRI) or the slope of the curing is calculated as below (Eq. 3) [27]:

$$CRI\left(\%min^{-1}\right) = \frac{100}{t_{90} - t_s} \tag{3}$$

Table 2 Rheometric characteristics of EPDM/RR foams at 150 °C

Sample code	M_I (N.m)	M_U (N.m)	M90 (N.m)	ΔM (N.m)	t_s (min)	t_{opt} (min)	CRI (%/min)
R0	0.97	6.51	5.96	5.54	3.66	12.21	11.7
R5	1.06	6.68	6.12	5.62	3.05	11.32	12.09
R10	1.14	6.87	6.3	5.73	2.81	10.92	12.33
R15	1.23	7.04	6.46	5.81	2.17	10.05	12.69
R20	1.29	7.24	6.65	5.95	1.88	9.3	13.48

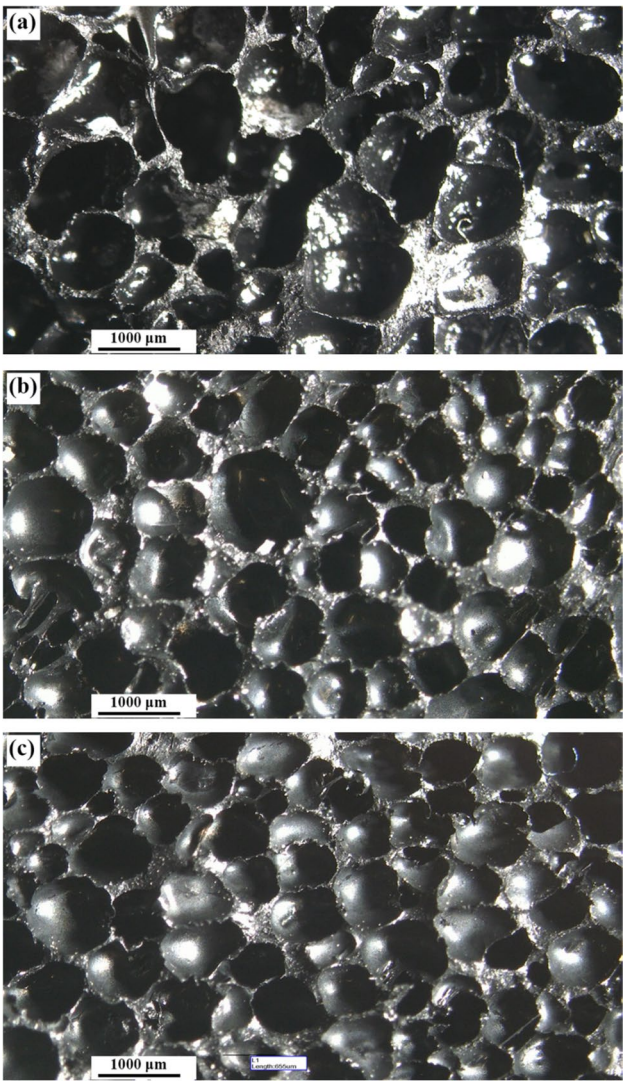

Fig. 2 Effect of RR content on morphology of EPDM/RR foams: **a** 0 phr, **b** 5 phr, **c** 10 phr, **d** 15 phr, **e** 20 phr

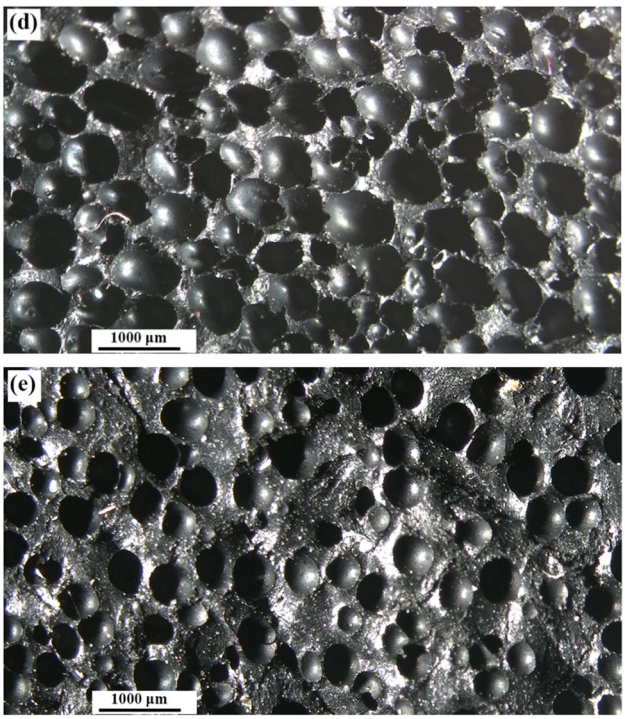

Fig. 2 (continued)

where t_s or t_5 (scorch time) and t_{opt} or t_{90} (optimum cure time) are the time to reach the 5% of maximum torque in the rheometry curing curve and the time to reach 90% of curing, respectively. The effect of RR content on curing characteristics, expressed in terms of M_I, M_U, ΔM, M_{90}, t_s, t_{opt}, CRI are presented in Table 2.

It can be observed in Fig. 1 that the torque increased linearly with the increase of RR content. The quantitative data given in Table 2 confirmed improved torque values including M_I, M_U and ΔM with increasing RR content. An increment in torque can be indicative of strong RR-EPDM rubber interactions and hence a stronger foam. Also the hidden CB content of RR can impart in improving the torque of the foam. An increase in initial torque with increasing content of RR is an indication of enhanced viscosity. As a result, increasing the percentage of RR from 0 phr in sample R.0 to 20 phr in sample R.20 increased M_I form 0.97 to 1.29 N.m. RR acted as the reinforcing agent by creating physical interactions with EPDM chains which leads to reduced and limited chain mobility and eventually raises M_I. Furthermore, both M_U and ΔM increased from 6.51 to 7.24 N.m and from 5.54 to 5.95 N.m, respectively by the establishment of crosslinks and improved interactions between the rubber chains. Results of rheometry in Table 2 revealed that increasing RR content from 0 to 20 phr decreased the scorch time (t_s) which is the time desired for the premature vulcanization of the foam to happen from 3.66 to 1.88 min. In a similar trend, the optimum cure time (t_{opt}) which measures the time taken to end vulcanization reduced from 12.21 to 9.3 min by RR addition. As seen in the table, the curing of the foams showed that RR-reinforced EPDM foams had faster CRI than the unreinforced ones. The incorporation of 20 phr of RR increased CRI from 11.7 to 13.48%/min. Active crosslinking sites and untreated curatives available in RR can accelerate the rate of crosslink formation and thus increase the curing rate of the foams [28–30]. In addition, the incorporation of fillers provides better heat distribution in the matrix which decreases scorch time values [31]. Besides, RR usually comprises some content of unreacted curatives or cross-linked precursors, which may lead to an increment in CRI and thereby decreasing t_{opt} and t_s [32].

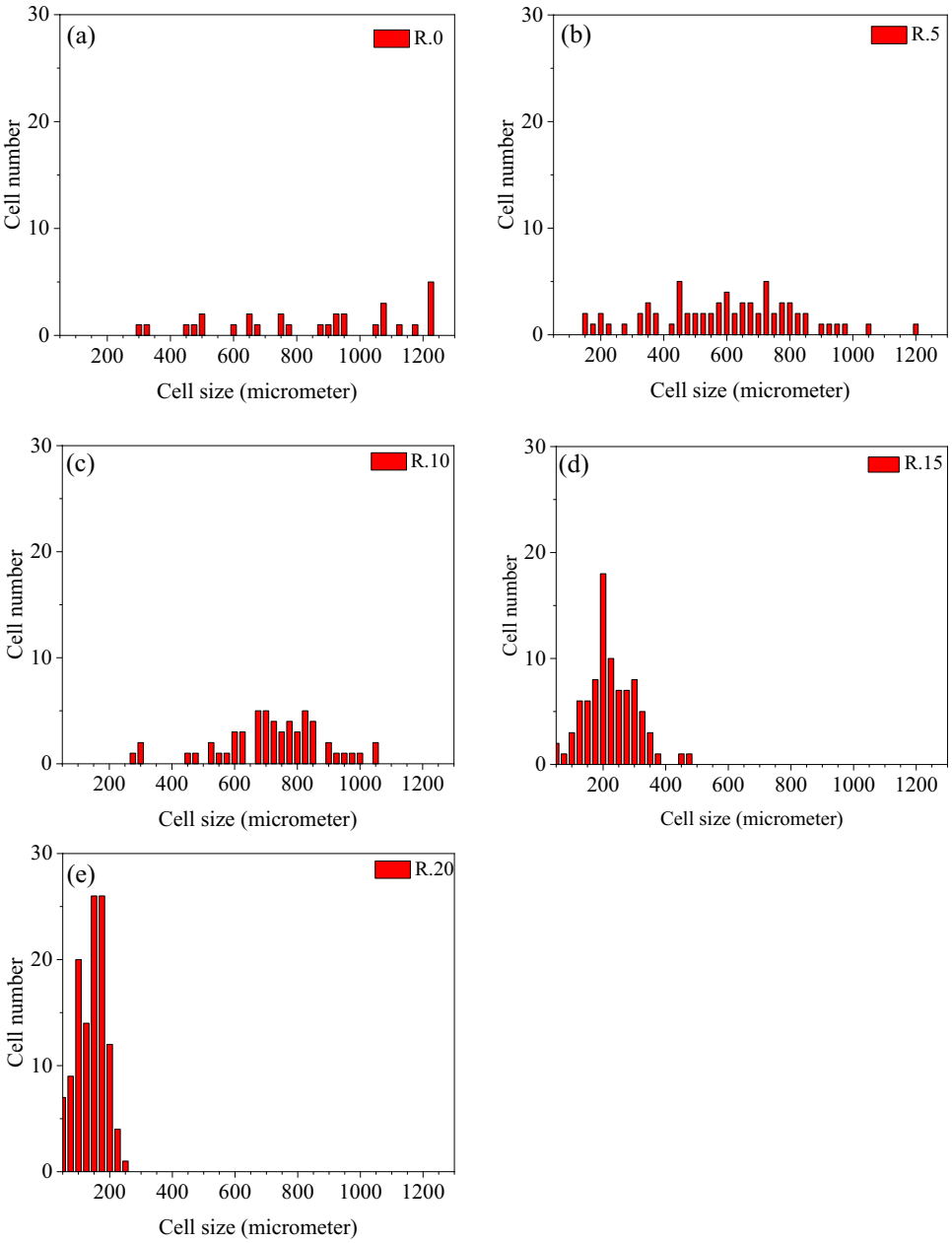

Fig. 3 Effect of RR content on cell size distribution of EPDM/RR foams: **a** 0 phr, **b** 5 phr, **c** 10 phr, **d** 15 phr, **e** 20 phr

Foam morphology

Morphological investigations of material allow the study of the dispersion and compatibility of fillers in composites. The results and information from the optical microscope test shown in Fig. 2 revealed that increasing the amount of RR increases cell number, and cell density and decreases cell size. Change in cell structure, as seen in Fig. 2, is related to the confrontation between the blowing agent press (which is the driving force for bubble formation) and the modulus and/or viscosity of the rubber (which is resistant to bubble growth). Investigation of the cell size, cell density, and cell size distribution of the rubber foams is an important topic because the structure has a direct effect on the final properties of the foams. For more detailed investigation and quantitative studies, image J software was used to analyze the results of optical microscopy (Fig. 3a-e). The cell size ranged from 50 to 1225 µm, depending on the RR content to give greater perspicuity. In addition, a quantitative study of the foam morphology was accomplished based on the cell density, the number average cell size (D_n), the weight average cell size (D_w) and the polydispersity index (PDI) using Eqs. 3 to 5, respectively, as follows:

$$D_n = \sum n_i.D_i / \sum n_i \quad (4)$$

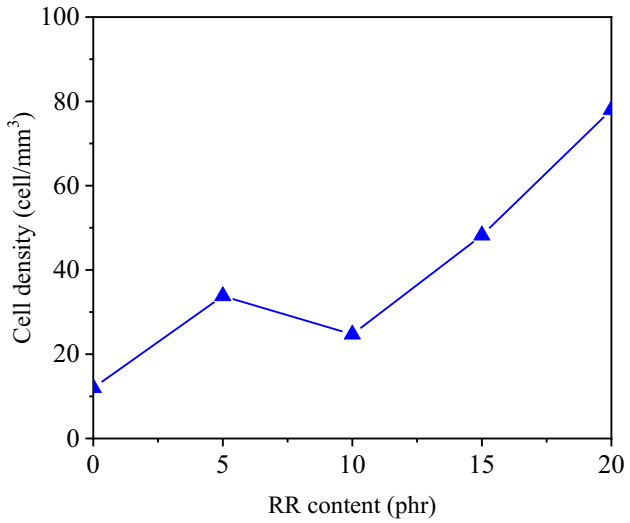

Fig. 4 Effect of RR content on cell density of EPDM/RR foams

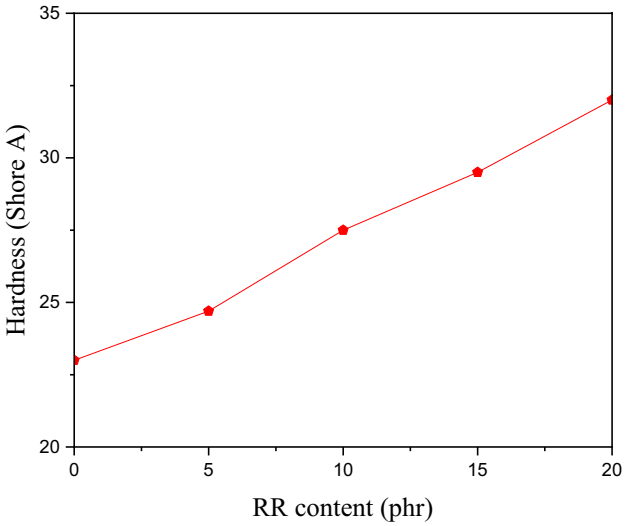

Fig. 6 Effect of RR content on the hardness of EPDM/RR foams

$$D_w = \sum n_i.D_i^2 / \sum n_i.D_i \tag{5}$$

$$PDI = \frac{D_w}{D_n} \tag{6}$$

wherein n_i is the number of cells with a diameter of D_i. The foams with smaller sizes and more uniform cell sizes generally offer improved properties, especially mechanical performance [33]. When a foam exhibits uniform cell size and structure all over the sample, the PDI approaches unity. These tendencies are in consent settlement with the literature [34, 35]. The cell size distribution curve's peak in sample R0 was located at 1225 µm, but it shifted into lower amounts with increasing RR content, where it is 175 µm for sample R20. It can also be seen in Fig. 3 that the cell size distribution of the R20 sample showed a narrower peak in comparison with the neat foam sample, R0. As a result, the PDI value in R20 sample is closer to unity than that of sample R0. The variation of average cell size vs. RR content is given in Fig. 4. According to the figure, with increasing the amount of RR, the average cell size decreases. In other words, the inhibiting force which resists the foam cell's expansion, was increased by increasing the RR loading, although the total amount of expansion is the same. Therefore, the average cell size was decreased and the cell size distribution became narrower, similar to what was observed in previous studies [36]. As can be seen, the number average

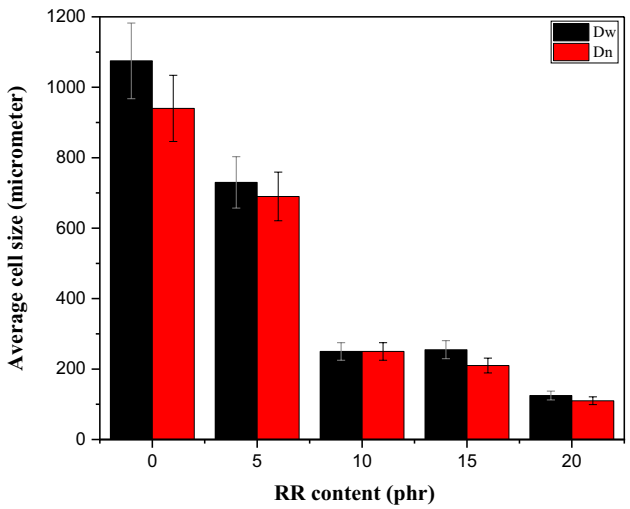

Fig. 5 Effect of RR content on average cell size of EPDM/RR foams

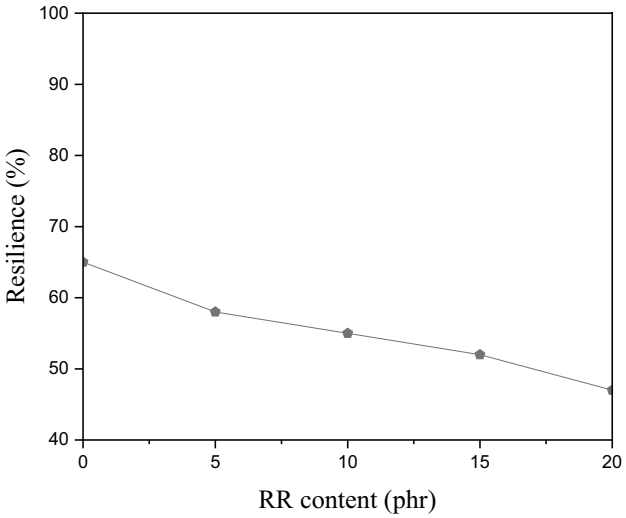

Fig. 7 Effect of RR content on resilience of EPDM/RR foams

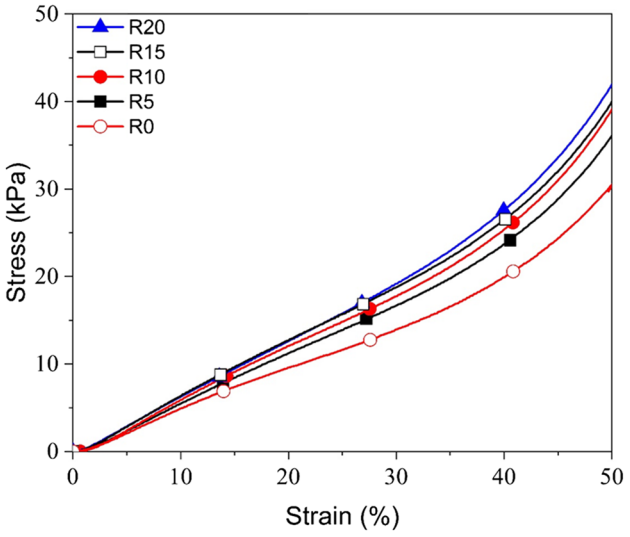

Fig. 8 Effect of RR content on stress–strain behavior of EPDM/RR foams

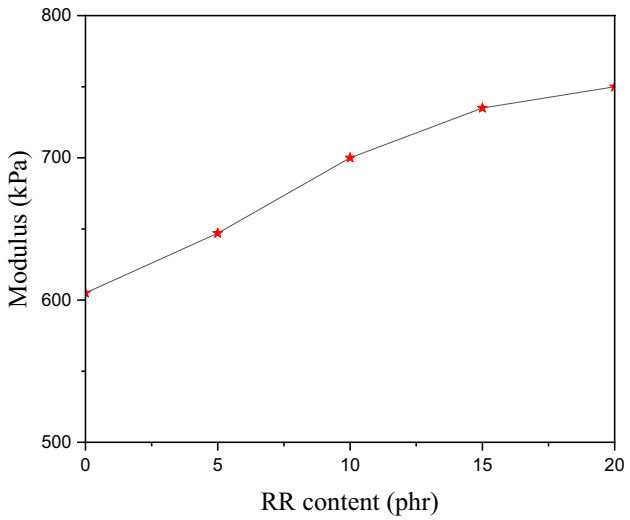

Fig. 9 Effect of RR content on the modulus of EPDM/RR foams

cell size is 940 and 110 µm in R0 and R20 samples, respectively, and the weight average cell size is 1075 and 120 µm in R0 and R20 contents, respectively. In addition, thicker cell wall was witnessed with increasing RR content which can be the direct result of the high cure rate, high viscosity and short scorch time. All of the mentioned factors reduced the expansion ratio leading to smaller cells with thicker wall. Cell density is defined by two terms using Eq. 7; the first term indicates surface cell density, the number of cells per unit area, obtained from the optical microscope image analysis, while the second one shows the ratio between the matrix density and the foam density [37]:

$$Cell\ density = \left(\frac{Cell\ number}{Area}\right)^{3/2} * \frac{Bulk\ density}{Foam\ density} \quad (7)$$

As shown in Fig. 5, the addition of 20 phr RR increased the cell density to 78 $cell/mm^3$, which is 6.5 times more than pure EPDM foam. Any factor that can reduce the mobility of the chain and increase the viscosity or modulus will be able to increase the resistance of the polymer chain to movement and deformation. Therefore, the resistance of the chain to gas pressure increases and this factor causes the cell size decrement and thereby increasing the cell density.

Mechanical properties

Various rubber foams have different mechanical properties to fulfill the requirements of any specific application. The incorporation of filler has been widely known as a promising approach to increase the hardness of the rubber compound [8, 38] depending on the type of polymer and the filler that should portray at least a good compatibility [39, 40]. Figure 6 shows the effect of RR content as a reinforcing filler on the hardness of EPDM/RR foams. As shown in the figure, with increasing the content of RR, the hardness of elastic foams also increases, e.g. by the addition of 20 phr of RR into EPDM foam, the hardness enhanced from 23 to 32 shore A which can be related to the reinforcing effect of RR on the EPDM foam. The higher RR content increased the EPDM foam's hardness by the presence of the higher amount of carbon black embedded in RR, increasing the crosslink density due to more active crosslink sites in the RR, as well as the RR-rubber interactions, where both physically hinder the rubber chain mobility [41]. Such observation is in

Table 3 Variables determined by the relative density of the foam

Sample	Compound		Mold		Blowing agent		Rubber		Other components	
	Weight (g)	Volume (cm^3)	Volume (cm^3)	Free volume (cm^3)	Weight component (phr)	Weight (g)	Weight component (phr)	Weight (g)	Weight component (phr)	Weight (g)
D 0.3	3	3.01	9.189	6.089	6	0.132	100	2.27	26.2	0.594
D 0.4	3.8	4.02	9.189	5.169	6	0.176	100	3.26	26.2	0.793
D 0.5	4.5	4.52	9.189	4.669	6	0.198	100	3.44	26.2	0.892
D 0.6	5.1	5.03	9.189	3.159	6	0.220	100	3.782	26.2	0.991

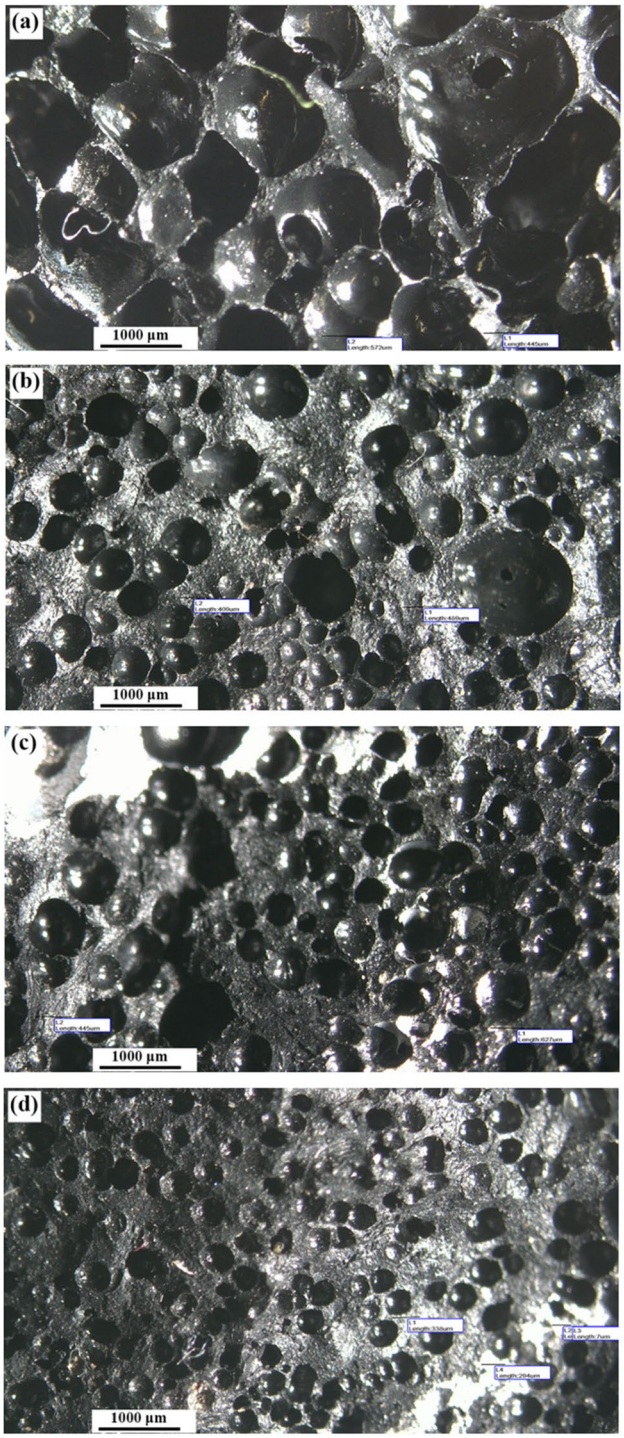

Fig. 10 Effect of relative density on the morphology of EPDM/R.10 foams: **a** D 0.3, **b** D 0.4, **c** D 0.5, **d** D 0.6

accordance with what has been reported in the literature [42]. Figure 7 shows the resilience of the EPDM/RR foams, where the neat EPDM foam had a resilience of 65% while the addition of 20phr RR decreased the EPDM foam resilience to around 47%. Resilience is linearly dependent on the friction between the rubber chains [43]. Consequently, with increasing the content of RR, the friction between the chains increases which imparts in reducing the resilience of EPDM/RR foams, which agrees with what was reported before for RR-reinforced acrylonitrile butadiene rubber [43]. Stress–strain curves of the EPDM/RR foam are shown in Fig. 8. The results revealed that increasing RR content improves the stress–strain behavior of the EPDM/RR foams. As seen in the figure, this increase is more noticeable at higher strains which can be due to a decrease in resilience and an increase in hysteresis loss in the presence of RR [44]. Following the compression behavior, Fig. 9 indicated that the modulus increases as the higher amounts of RR are introduced into the foam compound, i.e. with the addition 20 phr of RR, the modulus increased to around 750 kPa. The increasing trend in the strength of polymer chains is in agreement with decreased cell size and increased cure observed above due to the presence of RR.

Effect of relative density

In this section, the effect of relative density on the morphological and mechanical properties of the foams produced was studied. The same procedure of compounding was applied as mentioned in the previous section, but the amount of RR was kept constant (10 phr) and compounding of EPDM/R.10 was carried out at different relative density amounts including D0.3, D0.4, D0.5, and D0.6, as is showed in Table 3. To produce foams with mentioned relative density amount 3, 3.8, 4.5, and 5.1 g of EPDM/R.10 samples were placed in the mold, respectively. The effect of increasing the relative density on the morphology of rubber closed-cell foams is shown in Fig. 10. The results of the optical microscope in the figure revealed that increasing the foam density increases cell density and decreases average cell size. For more detailed investigation and quantitative studies, image J software was used to analyze the results of optical microscopy (Fig. 11a-d). For a further explanation, the results reported in Table 4 and the histograms of Fig. 11 show that by increasing the foam density, the average cell size decreased from 810 µm to 145 µm, the cell density increased from 18 $cell/mm^3$ to 92 $cell/mm^3$ and the cell size distribution became narrower: from (500–1250) microns for M3 to (75–480) microns in M6. It should be mentioned that the mold volume was fixed (9.189 cm^3) for all the specimens to follow the morphological changes with density (filling ratio). To increase the relative density of the foam, the compounds' volume placed in the mold from M3 to M6 increases resulting in a reduction in the free volume of the mold. For example, increasing the compound volume from 3.01 cm^3 to 5.03 cm^3 caused lower mold free-volume for foam expansion which decreased from 6.089 cm^3 to 3.159 cm^3. Hence, the cells of foam cannot grow freely [45], leading to higher foam density and a higher

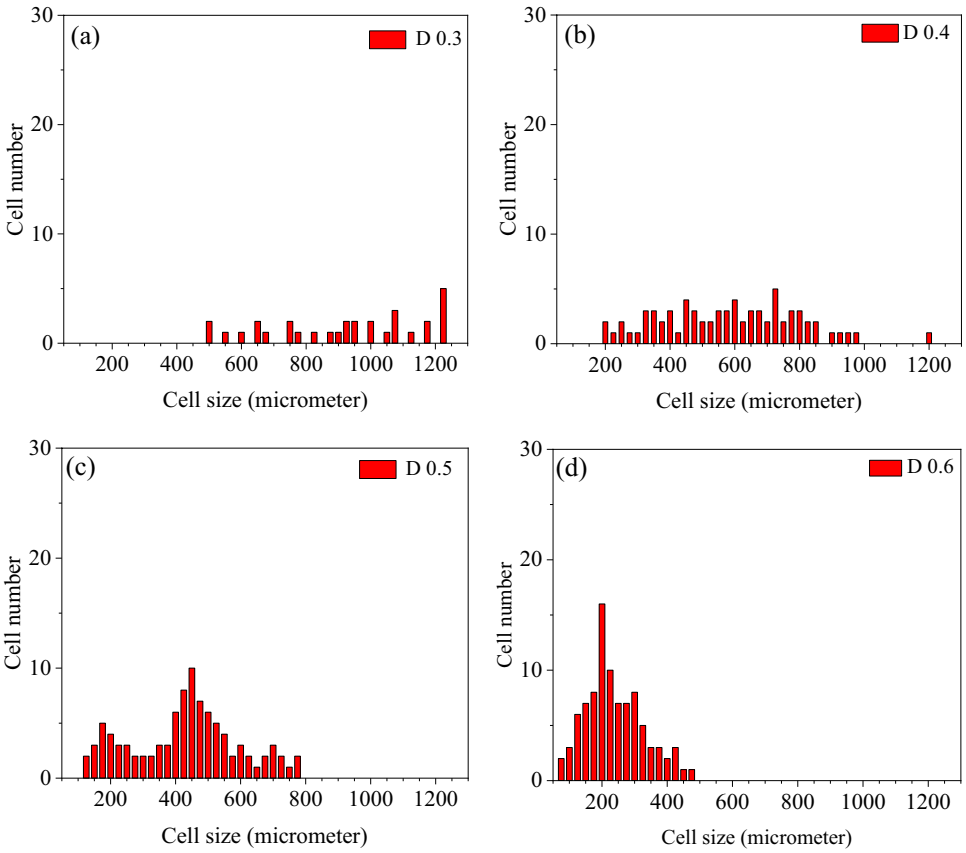

Fig. 11 Effect of relative density cell size distribution of EPDM/R.10 foams: **a** D0.3, **b** D0.4, **c** D0.5, **d** D0.6

filling ratio. In addition, since the formulation of the compound placed in the mold was fixed the weight of the blowing agent (ADC) increased in the mold. Therefore, in the foam with higher density, the amount of gas created is more and the free volume of the mold where the produced gas can occupy decreases. Therefore, before all the blowing agent in the compound is decomposed, the mold is filled and consequently, the cell size remains small. Studies show that [46] the foams with a higher density/filling ratio have a homogeneous structure and the foams with a lower density/filling ratio have a more heterogeneous structure. A homogenous structure composes of a higher number of smaller cells since the rate of cell nucleation is much greater than the growth of cells. While in a heterogeneous structure the cell growth is more predominant leading to larger cells and a higher probability of broken/connected cells (structural defects). This is schematically illustrated in Fig. 12. To investigate the effect of relative density on mechanical properties of the foams, hardness, compression modulus, and resilience were measured. It can be seen in Table 4, the hardness, compression modulus, and resilience increased from 16 Shore A, 80 kPa, and 38% in sample M3 to 37 Shore A, 785 kPa, and 68% in sample M6. However, it has been previously reported that the foams with higher density not only have higher rubber content (lower cell volume) but also have smaller cell sizes and a more uniform structure, which are known to produce better mechanical properties [37, 47].

Table 4 Physical and mechanical properties of EPDM/R.10 foams with various relative density

Sample	Cell density ($cell/mm^3$)	Average size (micron)	Modulus (kPa)	Hardness (Shore A)	Resilience (%)
M3	18	810	80	16	38
M4	26	320	220	23	57
M5	51	238	430	26	64
M6	92	145	785	37	68

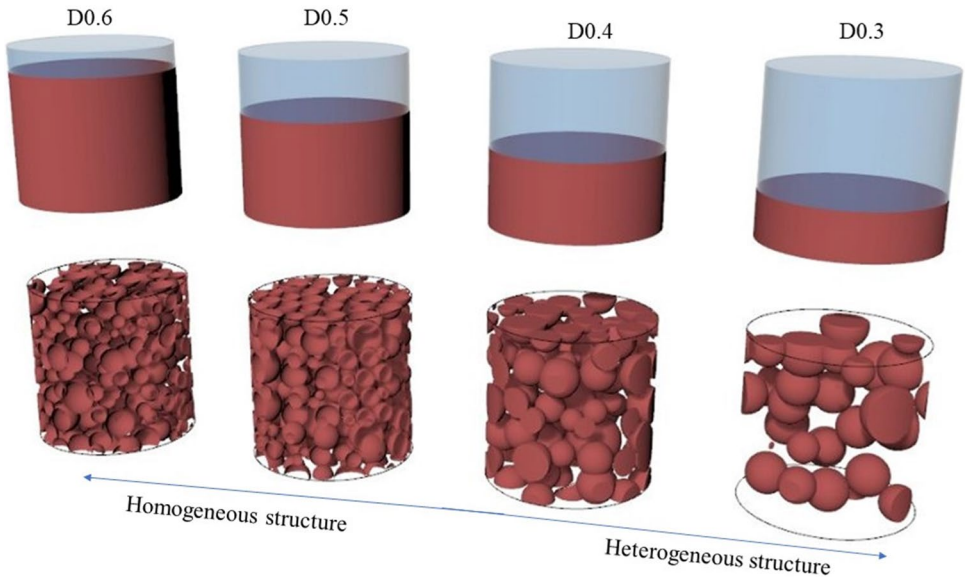

Fig. 12 Schematic display of the foaming process with variant relative density

Conclusion

As in today's world, the circular economy concept needs to be extended to all industries including elastomers. In this work, we have demonstrated the potential of RR as a promising reinforcing filler in the fabrication of EPDM foams. Cure characteristics of the EPDM/RR foams indicate that increasing the RR content from 0 to 20 phr increased the curing rate, initial torque, and delta torque while reducing the scorch time and the curing time. The introduction of the RR into the EPDM compound resulted in a foam of narrower cell size distribution and smaller average cell size. The cell size distribution curve's peak for R0 and R20 samples occurred at 1225 µm and 175 µm, respectively. According to the compression tests, all RR-reinforced EPDM foams demonstrated superior mechanical properties compared to the neat foam, mainly due to the high amount of carbon black in the RR, higher crosslink density of the foam due to more active crosslink sites in the RR, and improved RR-rubber interactions. The results showed that with the addition of RR content, the modulus and hardness of the foams were increased and the resilience of the foams compound decreased. The effect of increasing the relative density on the morphology of rubber closed-cell foams showed that with increasing the relative density of foams, cell density increased and cell size decreased. The mechanical properties such as hardness, compression modulus, and resilience were improved by increasing the relative foam density from 0.3 to 0.6.

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Declarations

Conflicts of interest The authors declare no conflict of interest.

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