

Thermomechanical evaluation of expanded ethylene-propylene diene monomers rubber mixed with recycled polyvinyl chloride foams

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ABSTRACT

The hazardous practice of landfilling at the end of life (EoL) of large amounts of rigid crosslinked interpenetrated network polyvinyl chloride (PVC) foams urgently requires development of effective recycling methods. In this work, PVC foam waste obtained from an industrial process was mixed with ethylene-propylene diene monomers (EPDM) rubber. The waste PVC foam naturally contained water (up to 35%) which was exploited for the expansion of EPDM rubber. For this, various amounts of recycled polyvinyl chloride (PVC) (20%, 30%, 40% by weight) were melt compounded with ethylene-propylene diene monomer rubber (EPDM) at 60 °C for 15 min. The obtained rubber mixtures were vulcanized at 160 °C for 20 min under varied compression time to create square sheets of expanded EPDM/PVC blends. The insertion of the humid PVC foam promoted the foaming of EPDM rubber up to a total porosity of 40% meanwhile geometrical density was decreased by 33% by the expansion of the rubber. Tensile tests revealed that, despite the presence of rigid PVC foams, the expanded EPDM/PVC blends manifested appreciable elongation at break values (up to 700%). Both compression set and shore A values of rubber increased with the addition of the rigid PVC foam. Moreover, all expanded blends showed low thermal conductivity values with a minimum of 0.07 W/m K.

1. Introduction

Due to their low density and limited thermal conductivity values, expanded polymers have gained significant interest in the last years for their use as insulating materials (Wang et al., 2006). Usually, rubber foams are expanded using a blowing agent releasing a gaseous phase. Based on the foaming parameters, the morphology of the pores can be different, and the resulting foams can be used for various applications. These include thermal gaskets, insulation panels, and impact sound damping products (Vahidifar et al., 2016; Wimolmala et al., 2009; Yamsaengsung and Sombatsompop, 2008). The thermal decomposition of a chemical blowing agent results in the generation of gases within the elastomeric matrix hence promoting the foaming process (Vahidifar et al., 2016; Reglero Ruiz et al., 2015). Several matrices are used to create rubber foams including, natural rubber (Bashir et al., 2012) or synthetic rubbers, such as styrene-butadiene rubber (Choi et al., 2004) acrylonitrile butadiene rubber, and ethylene-propylene diene monomer (EPDM) (Yamsaengsung and Sombatsompop, 2008; Zonta et al., 2020).

EPDM is a synthetic rubber made by the polymerization of ethylene and propylene with a non-conjugated diene monomer. Vulcanization is normally carried out at elevated temperatures for the formation of sulfur bridges (Sohn et al., 2003). EPDM rubber compounds usually find their application at industrial scale and are produced by mixing EPDM rubber with vulcanizing agents along with antioxidants, activators, fillers, and accelerants (Komalan et al., 2007). Such compounds possess good mechanical properties, appreciable resistance to UV radiation, weathering, ozone exposure, and aging. Owing to their peculiar characteristics, rubbers are used to produce various technical products such as water proofing membranes, electrical insulation for cables, belts, O-rings and gaskets, etc. (De and White, 2001).

Various EPDM based blends have been reported in the past aiming to induce the beneficial properties of EPDM in polymer matrices. Styrene butadiene rubber suffers from environmental factors like moisture, ozone, light and heat. Such issues are solved by blending it with EPDM (Vishvanathperumal and Gopalakannan, 2018). Eventually, the addition of EPDM in natural rubber (NR) has provided blends with improved chemical and ozone resistance with reduced compression set (Motaung et al., 2011; El-Sabbagh, 2003). Also, solvent resistance

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and adhesion properties of acrylonitrile–butadiene rubber (NBR) was improved by blending it with EPDM (Samaržija-Jovanović et al., 2011). The impact resistance of PVC at room temperature and low temperature was enhanced by using EPDM (Lee and Chen, 1987). Similarly, the impact strength of PVC was improved by a graft copolymer of EPDM with styrene-co-acronitrile (Sudhakar and Singh, 1992). Interestingly, EPDM composites with PVC has never been analyzed for its insulating properties. In fact, in the past, PVC has been used to improve the insulating properties of various materials like mortars and concrete (Senhadji et al., 2019; Latroch et al., 2018; Michel Murillo et al., 2019). Considering the fact that EPDM rubber finds its use in outdoor applications e.g., roofing panels, it would be worthwhile to investigate the synergetic effects of PVC in EPDM rubber.

PVC is one of the most widely consumed plastics worldwide (Ye et al., 2017) due to its durability, good fire resistance and low cost. PVC is widely used in building, construction, transportation, packaging, electrical and healthcare applications etc. However, PVC is non-degradable. Proper waste management processes are required to govern the end-of-life of plastics. One of the possible processes is burning the waste (incineration) which obviously brings along with it the major problem of releasing harmful gases in the air. Another option is to landfill the waste which is simply burying the material in the earth. In fact, this process is also detrimental to the environment since it not only can produce toxic gases but also could result in affecting the growth of agricultural products. There is also the risk of polluting surface or ground water (Aji et al., 2020). A more environmentally friendly option is to reuse or recycle the materials by mechanical or chemical processes (Soto et al., 2018) or to use them for energy recovery (Miandad et al., 2017). A cost-effective but technically challenging recycling option is to reuse the waste material in the form of a filler embedded in a matrix. This could either bring down the overall cost of the final material or in addition, impart certain properties advantageous for specific applications, such as strengthening the matrix material, improving its thermal stability or modify its thermal or electrical properties etc.

Often the waste materials are compacted and stored in depository before heading for waste management like landfilling. If not done properly, such processes would result in the contamination of the waste material due to environmental effects like exposure to rain and humidity hence drawing more problems in the waste management process. In this work, the presence of a certain moisture content in the PVC foams was exploited by combining it with EPDM rubber. It has been demonstrated in the past that the expansion of polymer foam is performed using various foaming agents which include the use of water as well. Hence, it was expected that such PVC waste with moisture would not only be added in the EPDM rubber as a filler but also would be useful to expand the rubber during its vulcanization process. Based on these considerations, this work investigates the physical, thermal, mechanical properties of the expanded EPDM rubber. The properties of the resulting compounds were compared with the corresponding unmodified expanded EPDM rubber.

2. Experimental part

2.1. Materials

The EPDM rubber used in this work was Vistalon® 2504 by Exxon Mobil (Irving, TX, USA). It is an amorphous terpolymer containing 58 wt% of ethylene and 4.7 wt% of ethylidene norbornene having a low Mooney viscosity ($ML_1 + 4$, 125 °C) of 25 MU. Zinc oxide (curing activator), stearic acid (curing activator and lubricating agent), and sulfur (vulcanizing agent) were supplied by Rhein Chemie (Cologne, Germany). The accelerants, tetramethylthiuram disulphide and zinc dibutyl dithiocarbamate were obtained from Vibiplast srl (Castano Primo, Milan, Italy). As a reinforcing filler, carbon black (CB) N550

from Omsk Carbon group (Omsk, Russia) was used to improve the properties of the EPDM rubber. Table 1 reports the composition of the elastomeric compound used for the preparation of the samples in this work (quantities expressed in phr). All the materials were used as received.

The crosslinked PVC foams were provided by Diab SpA (Longarone, BL, Italy). In particular, Divinycell was received in the form of circular disks which were in fact compacted scraps of foams PVC (Fig. 1). According to the technical data sheet of the company, the composition of the original foam includes PVC (30–75%), aromatic polyurea (15–50%) and polyamide (3–10%). The PVC disk manifested a glass transition temperature (T_g) measured by differential scanning calorimeter (DSC) of 85.8 °C, the density measured by helium pycnometry was 1.441 ± 0.023 g/cm³ and the water content of were, and 35% respectively. The latter was measured by drying the PVC for 8 days at 50 °C under vacuum and calculating its weight lost.

2.2. Sample preparation

First EPDM/PVC compounds with various PVC content (20%, 30% and 40% by wt) were prepared by melt compounding using an internal mixer equipped with counter-rotating rotors (Thermo Haake Rheomix® 600). The mixing was performed at 60 °C for 15 min with a rotors speed of 50 rpm. EPDM was compounded with carbon black (CB) for 1 min in the mixer followed by the addition of the vulcanizing agent and the additives. These were mixed for other 4 min to achieve homogeneity in the compound. Subsequently, PVC waste was added in determined quantities slowly to avoid agglomeration in the EPDM matrix. Finally, expansion and vulcanization process of the resulting compounds were carried out simultaneously under a hydraulic laboratory press (Carver model 2699) at a pressure of 3 MPa for 2, 3 or 4 min (T_1). Afterwards, only the pressure was released in order to allow the expansion of the blend for 18, 17 or 16 min (T_2). This way, the vulcanization process always was performed for a total of 20 min to create expanded square sheets of 100×100 mm². Table 2 summarizes the list of blends created using PVC waste along with the expansion conditions employed. For comparison purposes, neat EPDM-CB matrix was also expanded by using water in an amount equal to that present in the blends with PVC. Since the PVC foams originally contained 35% of moisture, a corresponding calculated water amount was added in the CB particles. For example, according to Table 2, exp-EPDM-CB-20H₂O contained water amount almost equal to that of the blend exp-EPDM-CB-20PVC.

2.3. Experimental techniques

Differential scanning calorimetry (DSC) measurements were performed by employing a Mettler DSC30 calorimeter (Mettler-Toledo GmbH, Columbus, Ohio, USA). Three thermal ramps at 10 °C·min⁻¹

Table 1

Composition of the elastomeric compound used for the preparation of the blends.

Material	Quantity (phr)
Vistalon® 2504	100
Sulfur	3
Zinc oxide	3
Stearic acid	1
Carbon black (CB)	20
TMTD	0.87
ZDBC	2.5

Acronyms: TMTD, tetramethylthiuram disulphide; ZDBC, zinc dibutyl dithiocarbamate.



Fig. 1. As received compacted PVC disk used.

were performed, i.e. a heating stage from -100 to 150 °C, a cooling stage from 150 to -100 °C and the second heating stage from -100 to 150 °C. The tests were carried out under a nitrogen flow of $100 \text{ mL} \cdot \text{min}^{-1}$. A glass transition temperature (T_g) was calculated as the inflection point of the resultant thermograms.

Pycnometric density (ρ_{picn}) measurements were carried out at room temperature by using a helium pycnometer AccuPycII 1330 Pycnometer (Micrometrics Instrument Corporation, USA) using a crucible of 3.5 cm^3 . For each sample, 30 measurements were performed. Geometrical density (ρ_{geom}) i.e., mass over the total volume inclusive of solid, closed, and open porosity was carried out. In this regards, 1BA specimens were cut from the samples using a die cutter. Their masses were weighed with a precision balance (Mettler Toledo ME104) while the volume was measured by taking surface area equal to 2500 mm^2 . By using ASTM D6226 standard, total porosity (P_{tot}) and the fraction of open porosity (OP) were calculated according to Equations (1–3):

$$P_{\text{tot}} = 1 - \frac{\rho_{\text{geom}}}{\rho_{\text{bulk}}} \quad (1)$$

$$OP = 1 - \frac{\rho_{\text{geom}}}{\rho_{\text{picn}}} \quad (2)$$

$$CP = P_{\text{tot}} - OP \quad (3)$$

where ρ_{bulk} is the density of the blend without porosity (i.e., 0.99 g/cm^3 for the EPDM-CB sample).

Tensile properties under quasi-static conditions were tested using an Instron 5969 tensile testing machine equipped with a load cell of 1 kN . Tests were carried out on ISO 527 type 1BA specimens at a cross-head speed of 100 mm/min until break. The elastic modulus was mea-

sured as a tangent modulus whereas the maximum tensile strength and the strain at break were also determined. At least five specimens were tested for each blend.

The fracture surfaces of samples tested through quasi-static tensile tests were observed through a Zeiss Supra 40 field emission scanning electron microscope (FESEM), operating at an acceleration voltage of 4 kV . Prior to the analysis, the samples were coated with a platinum-palladium alloy (80:20) coating having a thickness of about 5 nm .

Compression set measurements were carried out according to ASTM D395 standard for 22 h at a temperature of 23 °C and 70 °C. Recovery measurements were made 30 min after unloading. The test samples had a diameter of 12 mm while the ratio between the thickness of the spacing bars and the thickness of the samples was equal to 0.75 . The compression set (C_b) has been evaluated according to Equation (4):

$$C_b = \frac{t_0 - t_i}{t_0 - t_u} \quad (3)$$

where t_0 is the initial thickness of the specimen, t_i the final thickness, and t_u the thickness of the spacing bars.

Shore-A hardness measurements were performed with a Durometer Prüfstander model OS-2 (Hildebrand, Germany) following the ASTM D2240 standard. At least five tests were carried out for each composition.

The thermal conductivity was measured using a heat-flow meter (NETZSCH 446 Lambda Small, Selb, Germany) in accordance with the ISO 8301 standard. The area of the specimen was $50 \text{ mm} \times 50 \text{ mm}$. The instrument was calibrated by measuring a NETZSCH reference sample of expanded polystyrene. Typical accuracy of the heat-flow meter (HFM) is $\pm 1\%$. The measurements were performed at mean sample temperatures of 0 °C, 5 °C and 10 °C under nitrogen flux (10 mL/min) while a 20 °C temperature difference between hot and cold plates was maintained.

3. Results and discussion

DSC results of expanded EPDM-CB-PVC blends along with EPDM-CB and PVC are summarized in Table 3. Apparently, the T_g values for EPDM-CB and PVC did not significantly change neither with the PVC content in EPDM-CB matrix nor with the change of the expansion parameters. Rather the values showed irregular trend especially for the glass transition temperature associated to PVC due to weak signals found in the thermograms. However, in general EPDM-CB in the blend panels showed a glass transition temperature around -52 °C either in first or second heating scan demonstrating that the presence of PVC phase in the blends does not affect the T_g of the rubber. Note that the presence of water in the PVC waste meant that the T_g of PVC in the first scan couldn't be analyzed.

In general, the morphology of the expanded materials is strictly connected to their density and porosity. Hence, density of the pro-

Table 2

List of prepared samples with respective composition and expansion parameters.

Sr.	Sample	EPDM-CB (wt. %)	PVC (wt. %)	Vulcanization time under pressure (T_1) (min)	Vulcanization and expansion time (T_2) (min)
1	exp-EPDM-CB-20H ₂ O(3–17)	100	0	3	17
2	exp-EPDM-CB-30H ₂ O(3–17)	100	0	3	17
3	exp-EPDM-CB-20PVC(2–18)	80	20	2	18
4	exp-EPDM-CB-20PVC(3–17)	80	20	3	17
5	exp-EPDM-CB-20PVC(4–16)	80	20	4	16
6	exp-EPDM-CB-30PVC(2–18)	70	30	2	18
7	exp-EPDM-CB-30PVC(3–17)	70	30	3	17
8	exp-EPDM-CB-30PVC(4–16)	70	30	4	16
9	exp-EPDM-CB-40PVC(2–18)	60	40	2	18
10	exp-EPDM-CB-40PVC(3–17)	60	40	3	17
11	exp-EPDM-CB-40PVC(4–16)	60	40	4	16

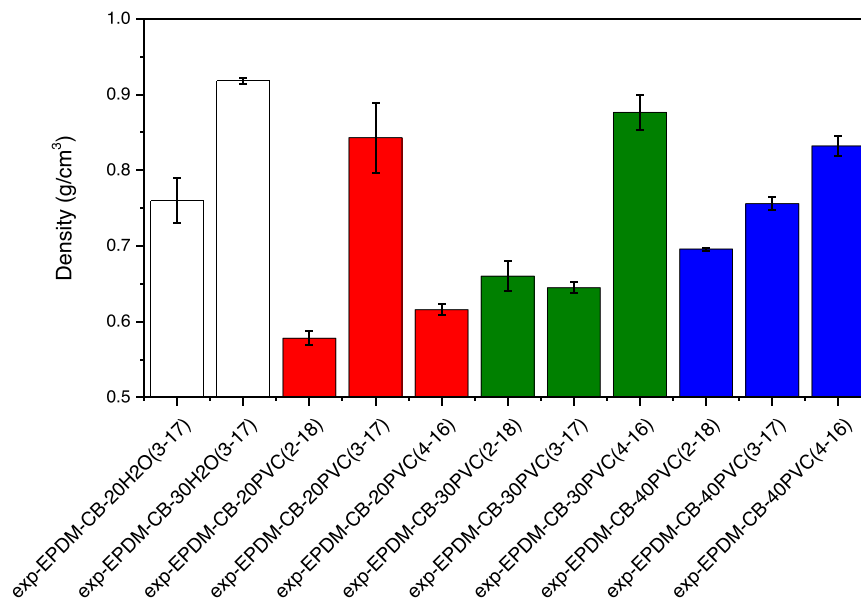
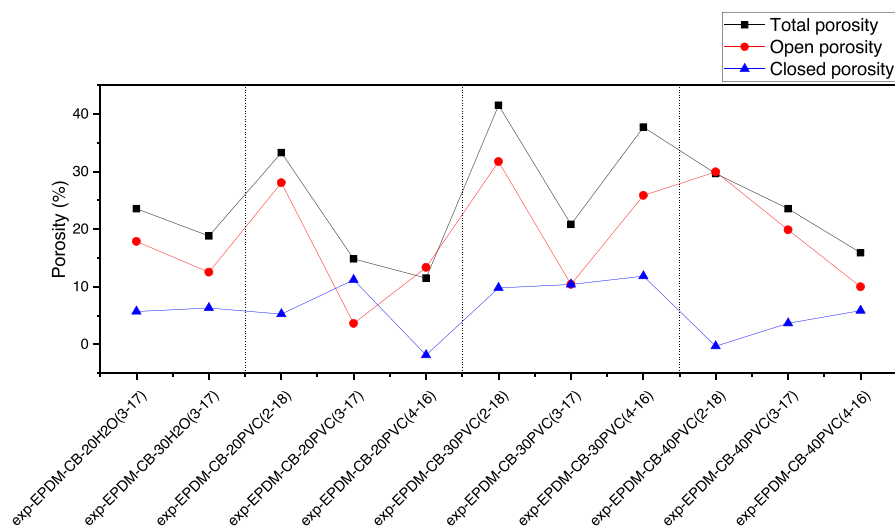
Table 3

Results of DSC tests on expanded EPDM-CB and PVC and relative EPDM-CB-PVC blends.

Sample	T _g ¹ (°C) (EPDM)	T _g ¹ (°C) (PVC)	T _g ² (°C) (EPDM)	T _g ² (°C) (PVC)
EPDM-CB	-51.64	–	-51.82	–
exp-EPDM-CB-20PVC(2-18)	-53.09	83.85	-53.58	82.37
exp-EPDM-CB-20PVC(3-17)	-53.32	82.06	-52.64	89.60
exp-EPDM-CB-20PVC(4-16)	-51.18	–	-54.49	82.44
exp-EPDM-CB-30PVC(2-18)	-55.09	80.67	-54.25	82.70
exp-EPDM-CB-30PVC(3-17)	-51.56	85.87	-53.18	88.58
exp-EPDM-CB-30PVC(4-16)	-54.52	87.41	-54.52	98.43
exp-EPDM-CB-40PVC(2-18)	-54.96	67.23	-53.15	82.92
exp-EPDM-CB-40PVC(3-17)	-51.54	82.12	-53.02	82.50
exp-EPDM-CB-40PVC(4-16)	-53.83	–	-55.31	81.29
PVC	–	–	–	85.80

duced panels was calculated by both geometrical and pycnometer measurements. In this way, the relative amount of open and close pores, and total porosity was thus determined. Fig. 2 compares the

geometrical density of the expanded EPDM-CB-PVC blends and compared to those of neat EPDM-CB samples. It can be noted that with the increase of PVC content (from 20% to 40%), the density tends to

**Fig. 2.** Geometrical density of expanded EPDM-CB-PVC blends.**Fig. 3.** Porosity of expanded EPDM-CB-PVC blends.

increase, which suggests the filling of the PVC phase in the exp-EPDM-CB matrix. In other words, although the rubber expands, the added filler also increases the weight of the composite and hence the density increases. In addition, it is worthwhile to notice that with the increase in T_1 time during vulcanization i.e., the compression time, the density tends to increase as well. This suggests that the vulcanization process overcomes the expansion of the rubber and hence the composite's density increases. Except for exp-EPDM-CB-20PVC(3–17) sample which showed anomalous result, all the three composition groups showed the increasing trend of density with increasing compaction time.

Subsequently, the porosity levels of produced blend panels were evaluated and compared in Fig. 3. Apart from a couple of anomalous

data, an appreciable amount of porosity can be seen in the exp-EPDM-CB-PVC blends which is even higher than that of the neat EPDM-CB expanded panels. In particular, the highest total porosity was found in the case of exp-EPDM-CB-30PVC(2–18). Along with the exp-EPDM-CB-30PVC(4–16), these high values of porosity suggest that the maximum possible expansion of EPDM-CB rubber is reached by the addition of 30 wt% PVC waste. This corresponds to higher moisture content that would result in the expansion of rubber during the vulcanization process. The same could have happened with the addition of 40 wt% PVC waste but in this case the expansion process was limited by the relatively low amount of EPDM-CB matrix. Observing carefully, it can be noticed that in almost all the

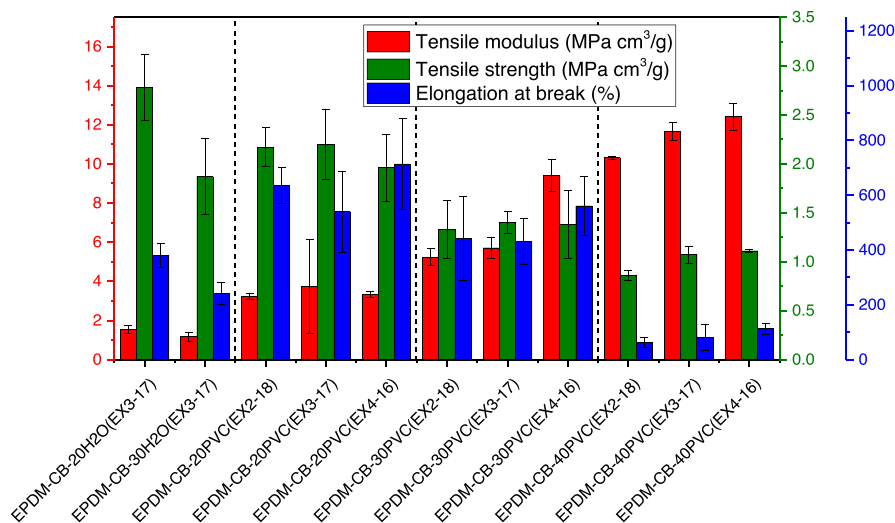


Fig. 4. Comparison of the mechanical properties of the expanded EPDM-CB and relative EPDM-CB-PVC blends.

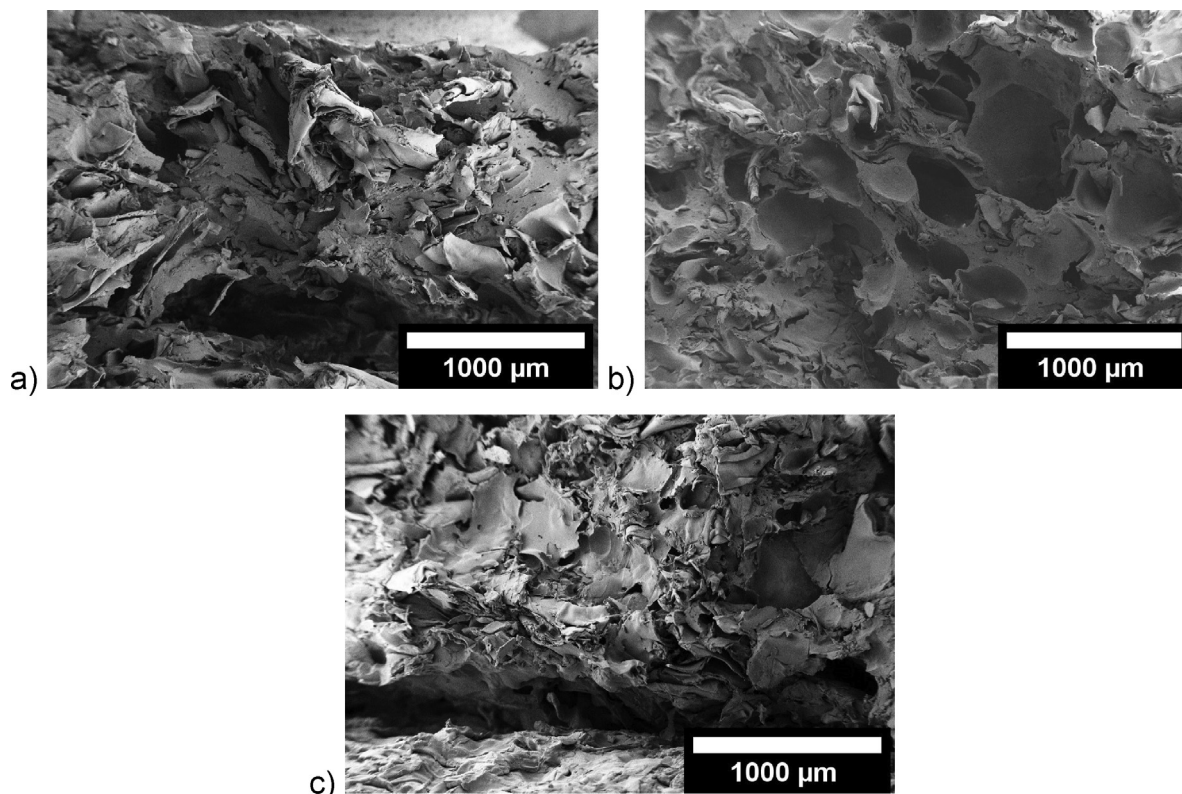


Fig. 5. FESEM images of a) exp-EPDM-CB-20H₂O(3–17) b) exp-EPDM-CB-20PVC(3–17), c) exp-EPDM-CB-30PVC(3–17) and d) exp-EPDM-CB-40PVC(3–17).

sample groups, increasing compression time decreases the open porosity while increases the close porosity. It can be hypothesized that the increased compression duration results in the promotion of vulcanization of rubber hence reduces the possibility for the moisture to escape. Correspondingly the porosity is reduced, and more close porosity is obtained in the samples.

The expanded EPDM-CB-PVC blends were evaluated for their mechanical properties by tensile testing. The obtained normalized values are compared in Fig. 4. It can be clearly seen that increasing the quantity of PVC foams in the blends caused the modulus values to rise almost linearly. Consequently, such increase resulted in the diminishing values of elongation at break of the blends which were still significantly higher in the presence of the rigid PVC particles. As reported in a previous study, the unexpanded EPDM-CB blends with added PVC foams manifested a lower elongation at break, for example of about 150% with a PVC content of 30% by weight (Mahmood and Pegoretti, 2021). However, the minimum for exp-EPDM-CB-30PVC obtained here was approximately 420% which is 2.8 times higher. Hence the expansion caused by the moisture present in the PVC waste provided significant positive advantages in terms of improved mechanical properties of EPDM-CB rubber blends.

The fracture surfaces of expanded EPDM-CB and EPDM-CB-PVC samples after tensile tests were analyzed by FESEM and the resultant images are compared in Fig. 5. Pure EPDM-CB sample expanded by water showed a smooth surface with big pores due to water escape during vulcanization process. Whereas, samples with PVC showed a rough surface with a varying degree of porosity on the fracture plane. In particular, exp-EPDM-CB-30PVC(3-17) showed big sized pores which indicates volumetric expansion of the rubber obtained from added PVC foamed particles. Such expansion was caused by the evaporation of entrapped moisture in the PVC foams (which contained about ~35% of entrapped water). Such expansion was limited in case of exp-EPDM-CB-PVC20(3-17) and almost zero in case of exp-EPDM-CB-PVC40(3-17). The former can be justified by the presence of a lower amount of PVC foamed particles hence less water content, while the latter could be attributed to a lower amount of rubber matrix available for expansion. Nonetheless, a fair level of adhesion between the PVC particles with the EPDM-CB matrix can be observed.

Strain recovery behavior of expanded EPDM-CB-PVC blends was evaluated by compression set tests at two different temperatures (23 °C, 70 °C) and compared with neat EPDM-CB expanded samples in Fig. 6. As expected, pure expanded EPDM-CB showed low compression set values in comparison to the panels reinforced with rigid PVC foams. Compression set values then gradually rose with the addition of PVC foams. In particular, the values were considerably higher for exp-EPDM-CB-40PVC samples (about 400%) due to the presence of rigid PVC particles in higher quantity. This is because as the PVC content rose, the amount of the elastomeric matrix phase decreased hence diminishing the capability of the blends to recover the strain applied for an extended time. Hence a permanent deformation of the blends during compression set tests was observed.

It was worthwhile to evaluate the hardness of expanded rubber panels reinforced by rigid PVC foams. Hence Shore A hardness test was performed, and the obtained values are compared in Fig. 7. The

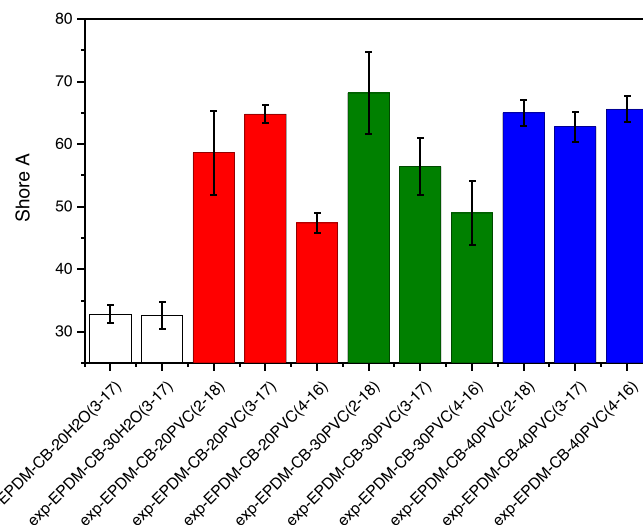


Fig. 7. Shore A hardness measured of expanded EPDM-CB and relative EPDM-CB-PVC blends.

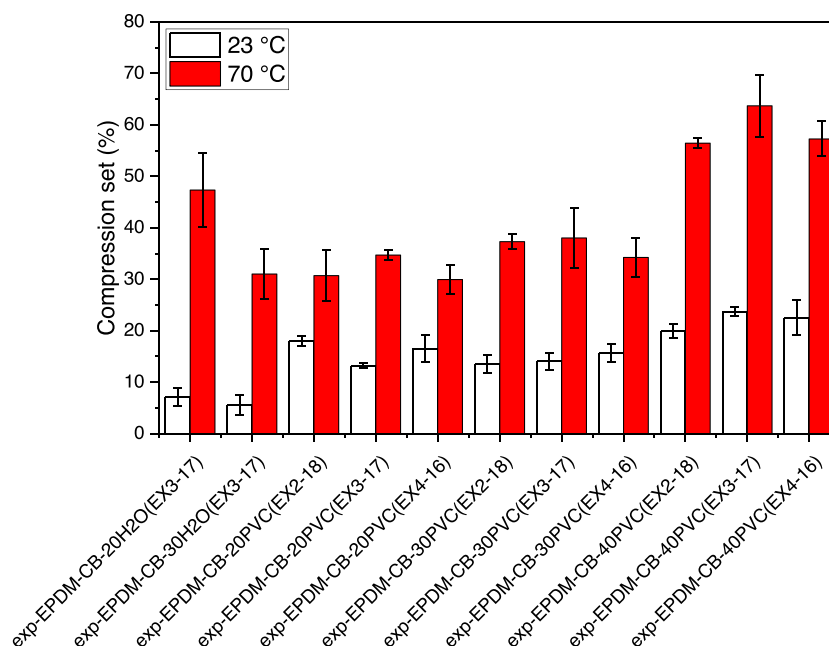


Fig. 6. Compression set of expanded EPDM-CB and relative EPDM-CB-PVC blends.

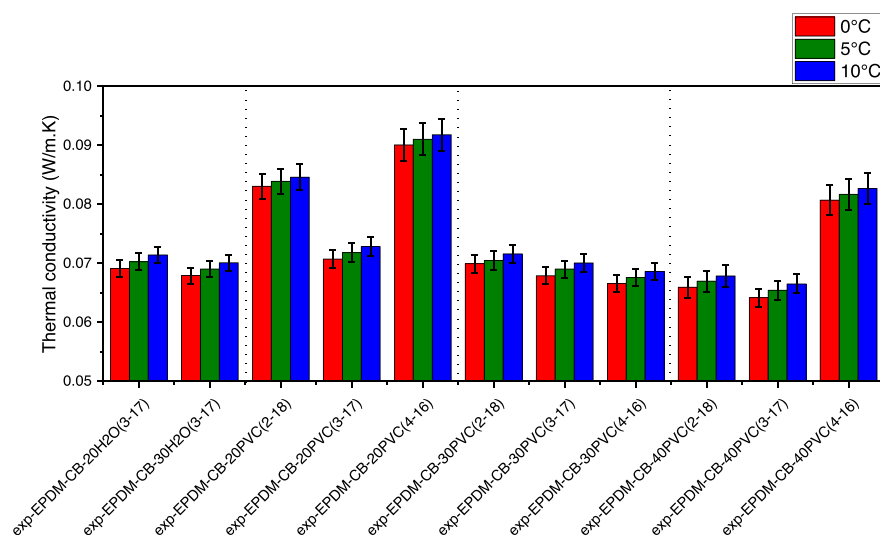


Fig. 8. Thermal conductivity of expanded EPDM-CB and relative EPDM-CB-PVC blends measured by heat flow meter HFM 446.

neat EPDM-CB expanded panels showed Shore A values of about 35 that increased with the addition of the rigid PVC foams but in an irregular manner. This asymmetry could be credited to the non-uniform volumetric expansion of the panels owed to the initial material used. In fact, the PVC waste was deliberately used un-sieved for the sake of simplicity of the use of the waste material. Hence the final volumetric expansion of the rubber panels was rather irregular. However, the increased compression time during vulcanization showed a trend of decreasing Shore A hardness. It can be hypothesized that the increased compression time increased the expansion of the rubber panels due to the presence of water vapor. Hence this caused the progressive softening of the rubber matrix for which the Shore A hardness decreased with the increased compression time. Overall, among the exp-EPDM-CB-PVC blend panels, the Shore A values achieved were in the range of 50 to 70 which, i.e., from 56% to 118% higher than the neat EPDM-CB expanded panels.

Due to the expansion of the EPDM-CB rubber, the low-density blend panels were then evaluated for their insulating behavior. The thermal conductivity values of all the samples were obtained at three different mean sample temperatures (i.e., 0 °C, 5 °C and 10 °C) and are compared in Fig. 8. As expectedly, all the samples including the pure expanded EPDM-CB panels showed roughly similar thermal conductivity behavior apart from some exceptions. Notably, exp-EPDM-CB-20PVC(2-18) showed relatively higher thermal conductivity values probably due to lack of optimum time required to evaporate the moisture in the blend panel. On the other hand, exp-EPDM-CB-20PVC(4-16) also showed higher thermal conductivity owed to the fact that too much time during compression resulted in the escape of water vapors from the panel before the expansion was allowed. The same could be presumed for the exp-EPDM-CB-40PVC(4-16) also showed peculiar result respect to other blend panels. Overall, the positive effect of PVC foam recycling in EPDM-CB can be seen in which the PVC foam not only reduces the thermal conductivity by 35% (Mahmood and Pegoretti, 2021) but also provides the possibility to expand the EPDM-CB rubber due to the presence of moisture in the waste PVC foam. Overall, such contribution led to achieve lowest thermal conductivity values for samples containing 30% PVC by weight.

4. Conclusions

Recycling of PVC waste material along with its water content was explored in this work for the creation of expanded EPDM-CB rubber blends. These expanded panels were created by first mixing EPDM-

CB rubber with the wet PVC foams coming from industrial scraps followed by a vulcanization process at 160 °C for 20 min with varying pressure conditions. This allowed the modification of porosity levels (up to 40%) in the rubber matrix hence providing expansion of the matrix of different level. Due to this, the elongation at break values were found to be remarkably high (up to 700%) even in the presence of rigid PVC foamed particles. Concurrently, compression and Shore A set values of the EPDM-CB matrix increased with the increasing PVC content (400% and 118% respectively). Additionally, the expansion in the rubber matrix positively induced thermal insulation behavior where the thermal conductivity values reached a minimum as low as 0.07 W/m.K.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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