

Effective recycling of end-of-life polyvinyl chloride foams in ethylene–propylene diene monomers rubber

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Abstract

Rigid cross-linked interpenetrated network polyvinyl chloride (PVC) foams are widely used as core materials in the composite industry. Nevertheless, no recycling methods exist today for these materials that are currently landfilled at the end of life (EoL). In this work, various amounts of EoL PVC cross-linked foams in the form of chips were mixed with ethylene–propylene diene monomer (EPDM) rubber and carbon black (CB) at 60°C for 15 min. The obtained rubber mixtures were vulcanized at 160°C for 20 min to create compacted square sheets of EPDM-CB-PVC composites using compression molding technique. Tensile tests revealed a progressive increase in modulus of EPDM-CB with increasing PVC content while a subsequent decrease of the elongation-at-break values of the composites was noticed. Scanning electron microscopy revealed a homogeneous dispersion of PVC particles with the EPDM-CB matrix. Improved interfacial adhesion of filler in the matrix was also observed at higher PVC content as well. Hardness tests revealed an increased Shore A values by 25% with added PVC waste in EPDM-CB. Concurrently, a steady increase in compression set was observed in the rubber composites with the added PVC filler. Additionally, due to added filler, thermal conductivity was decreased by 35% compared to pure EPDM-CB.

KEYWORDS

composites, ethylene–propylene diene monomers, polyvinyl chloride, recycling, rubber, thermal conductivity

1 | INTRODUCTION

With the progressive rise of global warming and environmental pollution, considerable research interest has been shown in recent years in the economic and strategic recycling of waste materials. Among many challenges in this field, the separation and utilization of polyvinyl chloride (PVC) from postconsumer waste like insulated wires

and cables, window profiles, and foams still present a daunting challenge for the world's community. With regard to its volume, it is considered to be the second most produced thermoplastic in the world. Out of total PVC manufactured in the world, approximately 8% of it is used in the production of the wires and cables.^[1] However, after their use, PVC becomes a waste only since the metal inside the wires is easily recycled for further use.

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Hence, the former is either incinerated or landfilled which presents an enormous hazard due to the chlorine content present in the polymer. For example, the widely used burning process produces deadly dioxins that could cause cancer as well as harm to the nervous system.^[2,3] In particular, rigid cross-linked interpenetrated network PVC foams are widely used as light core for composite sandwiches used in several applications from wind blades to marine structures. When the end of life of these artifacts is reached, the PVC foams are currently disposed in landfills with the related impacts in terms of wasted resources and environmental burden.

On this basis, the physical, chemical, or mechanical properties of recycled PVC can be beneficial when added to other virgin materials in the form of filler. Previous works have successfully confirmed the use of waste PVC in different products like roofing material, floors coverings, textiles, and other lost cost materials.^[4,5] In particular, the use of waste PVC in pavements has shown an improvement in the performance properties of the pavements.^[6,7] Hence, the use of recycled PVC in polymeric blends could represent an interesting option for the valorization of PVC wastes. Given that the worldwide plastic production in 2015 reached about 380 million metric tons,^[8] the use of PVC waste in virgin polymer matrices could result in great consumption of recycled PVC. Hence, it could be useful to produce novel materials with peculiar properties with respect to the pristine constituents, not only to substantially decrease the cost of the resulting compounds but also reduce the amount of raw material use.

Ethylene-propylene diene monomer (EPDM) is a synthetic rubber made of ethylene and propylene polymerized with a nonconjugated diene monomer. It is normally vulcanized at elevated temperatures to allow the formation of sulfur bridges.^[9] EPDM rubber compounds are normally used at an industrial scale and are generally obtained by mixing EPDM rubber with vulcanizing agents along with antioxidants, activators, fillers, and accelerants.^[10] Such compounds are generally recognized by their good mechanical properties, appreciable resistance to ultraviolet, weathering, ozone, and aging. Due to these properties, EPDM has attracted much attention in outdoor applications such as automotive sealing systems, building profiles, electrical power cables, proofing membranes, electrical insulation of cables, roofing sheets, belts, O-rings, and gaskets.^[11,12]

Polymeric blends involving EPDM have been substantially reported in the past mostly aiming to utilize the beneficial properties of EPDM in polymer matrices. For example, various studies have been published reporting the effect of EPDM on polypropylene's rheological, morphological, and mechanical properties.^[13-17] Styrene-butadiene rubber, although possessing good mechanical properties

and abrasion resistance, suffers from environmental factors like moisture, ozone, light, and heat. Such issues are rectified by blending it with EPDM.^[18] Eventually, the addition of EPDM in natural rubber has given rise to compounds having great ozone and chemical resistance with reduced compression set.^[19] Hence, such systems have found tire applications like sidewall.^[20] Also, EPDM was blended with acrylonitrile-butadiene rubber to improve solvent resistance and adhesion properties of the latter.^[21] In one study, the impact resistance of PVC at room temperature and low temperature was improved by using EPDM.^[22] A similar work was reported by Sudhakar et al. in which the impact strength of PVC was improved by a graft copolymer of EPDM with styrene-co-acrylonitrile.^[23]

The use of EPDM as an ablative, flame-retardant, or insulator has been researched in the past. This is because elastomers have high-char retention characteristics for which they find their use in the solid rocket motor.^[12,24] In this regard, various blends of EPDM have been researched to improve its thermal properties. Jiang et al. blended EPDM with microspheres of polyphenylsilsesquioxane which served as an effective ablative additive and flame-retardant for EPDM composites.^[25] In another work, micro/nanoparticles of boron nitride-blended EPDM composite showed improved thermal conductivity and stability, thus impeding the thermal depolymerization of EPDM.^[26] Recently, it was demonstrated by George et al. that the addition of Kevlar fibers (KF) in EPDM enhanced its thermal stability.^[27] The same group also reported the use of nanosilica in EPDM/KF composites for improving the thermal stability and mechanical properties of the composites produced.^[28] In one work, carbon nanotubes were also used to reduce the thermal conductivity and ablation rates of the char layer skeleton of EPDM composites at high temperature.^[29,30]

Interestingly, EPDM rubber composites with PVC as a filler have 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.^[31-33] Considering the fact the EPDM finds its use in outdoor applications, for example, roofing panels, it would be worthwhile to investigate the synergetic effects of PVC in EPDM rubber. In this work, it was hence aimed to investigate the beneficial effects derivable from the addition of recycled PVC material in the elastomeric rubber which would provide cost-beneficial effects by lowering the final rubber content in the product which would provide cost-benefit results by lowering the final rubber element required in the product. Based on these considerations, this work investigates the effect of PVC on the physical, thermal, and mechanical properties of compacted EPDM rubber. The properties of the resulting compounds were compared with the corresponding unmodified EPDM rubber.

2 | EXPERIMENTAL

2.1 | Materials

The EPDM rubber used in this work was Vistalon[®] 2504 purchased from Exxon Mobil (Irving, TX, USA). It is an amorphous terpolymer containing 58 wt% of ethylene and 4.7 wt% of ethylidene norbornene with 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 disulfide, and zinc dibutyl dithiocarbamate were obtained from Vibiplast srl (Castano Primo [MI], Italy). As reinforcing filler, carbon black (CB) N550 from Omsk Carbon Group (Omsk, Russia) was used. 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 cross-linked PVC foams were provided by Diab SpA (Longarone, BL, Italy). In particular, Divinycell was received in the form of chips (Figure 1) which were a waste of the cutting process involved in the production of PVC-foamed panels. According to the technical data sheet of the company, the composition of the foam includes PVC (30%–75%), aromatic polyurea (15%–50%), and polyamide (3%–10%). The glass transition temperature (T_g), bulk density, and water content of the PVC chips are 78.7°C, $0.065 \pm 0.001 \text{ g/cm}^3$, and 2%, respectively (measured after 7 days of drying at 50°C under vacuum).

2.2 | Sample preparation

EPDM-CB-PVC compounds with different PVC content (from 0 to 30 wt%) were mixed by using an internal mixer equipped with counter-rotating rotors (Thermo Haake

Rheomix[®] 600). The compounding was performed at 60°C for 15 min while mixing with a rotor speed of 50 rpm. Initially, EPDM was fed in the mixer with the CB and compounded for 1 min. Then, the vulcanizing agent and the additives were added and mixed for another 4 min to achieve homogeneity of the compound. Next, PVC was added in determined quantities slowly to avoid aggregation in the EPDM-CB matrix. Finally, the vulcanization of rubber composites was performed at 160°C under a pressure of 6.9 MPa for 20 min using Carver laboratory press (Model 2699). In this way, compacted square sheets of $100 \text{ cm} \times 100 \text{ mm}^2$ were obtained. Table 2 reports the nomenclature and the composition of the samples produced.

2.3 | Experimental techniques

Differential scanning calorimetry (DSC) measurements were carried out by using a Mettler DSC30 calorimeter (Mettler-Toledo GmbH, Columbus, Ohio, USA). Three thermal ramps at $10^\circ\text{C min}^{-1}$ were performed, that is, a first heating stage from -100°C to 150°C , a cooling stage from 150°C to



FIGURE 1 As received PVC chips used. PVC, Polyvinyl chloride

TABLE 1 Composition of the elastomeric compound used for the preparation of the composites

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

Abbreviations: TMTD, tetramethylthiuram disulfide; ZDBC, zinc dibutyl dithiocarbamate.

TABLE 2 List of prepared samples

Sr.	Sample	EPDM (wt%)	PVC (wt%)
1	EPDM-CB	100	0
2	EPDM-CB-10PVC	90	10
3	EPDM-CB-20PVC	80	20
4	EPDM-CB-30PVC	70	30

Abbreviations: CB, Carbon black; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride.

–100°C, and the second heating stage from –100°C to 150°C. The tests were carried out under a nitrogen flow of 100 ml min⁻¹. Glass transition temperature (T_g) was calculated as the inflection point of the resultant thermograms.

Tensile properties under quasi-static conditions were measured using an Instron 5969 tensile testing machine equipped with a load cell of 1 kN. Tests were performed on International Organization for Standardization (ISO) 527 type 1BA specimens at a crosshead speed of 100 mm min⁻¹ until break. The elastic modulus was measured 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 composite. Prior to testing, the 1BA specimens were weighed by precision balance (Mettler Toledo ME104), and their geometrical densities were calculated by considering the surface area of 1 BA specimens as 2500 mm².

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

The fracture surfaces of samples tested through quasi-static tensile tests were observed through a Zeiss Supra 40 field-emission scanning electron microscopy (FESEM), operating at an acceleration voltage of 3.5 kV. Prior to the analysis, the samples were coated with a platinum-palladium alloy (80:20) coating having a thickness of about 5 nm.

The thermal conductivity was measured in nitrogen atmosphere using a heat-flow meter (HFM; NETZSCH 446 Lambda Small, Selb, Germany) in accordance with the ISO 8301 standard. The area of the HFM was 50 mm × 50 mm. The instrument was calibrated by

measuring a NETZSCH reference sample of expanded polystyrene. Typical accuracy of the HFM is ±1%. The measurements were performed at mean temperatures of 0°C, 5°C, and 10°C while a 20°C temperature difference between hot and cold plates was maintained.

3 | RESULTS AND DISCUSSION

Representative DSC thermograms collected during the first and second heating stages of the EPDM-CB, PVC, and EPDM-CB-PVC composites are reported in Figure 2, while the glass transition temperatures (T_g) of the EPDM-CB and PVC phases collected during the first and the second heating stage are summarized in Table 3. In the rubber samples, EPDM showed a T_g around –52°C while in the composite specimens, the T_g of PVC varied considerably during the first scan. This could be justified by the weak signal associated with the T_g of PVC phase in the composite specimens. However, in the second scan, the T_g of PVC was rather similar, that is, around 82°C. Overall, the addition of PVC filler in the EPDM-CB rubber matrix does not significantly affect the T_g of the rubber. Note that in the first heating, the T_g of PVC is not mentioned in Table 3 since the representative thermogram of PVC did not allow its determination due to an overlapping endothermal signal due to the presence of adsorbed water in the sample (Figure 2A).

Table 4 compares the geometrical density of the compacted EPDM-CB-PVC composites. It can be noted that with the increase of PVC content, the density tends to increase, which suggests the compaction of the PVC phase in the EPDM-CB matrix during either internal mixing or compression molding or both. However, the

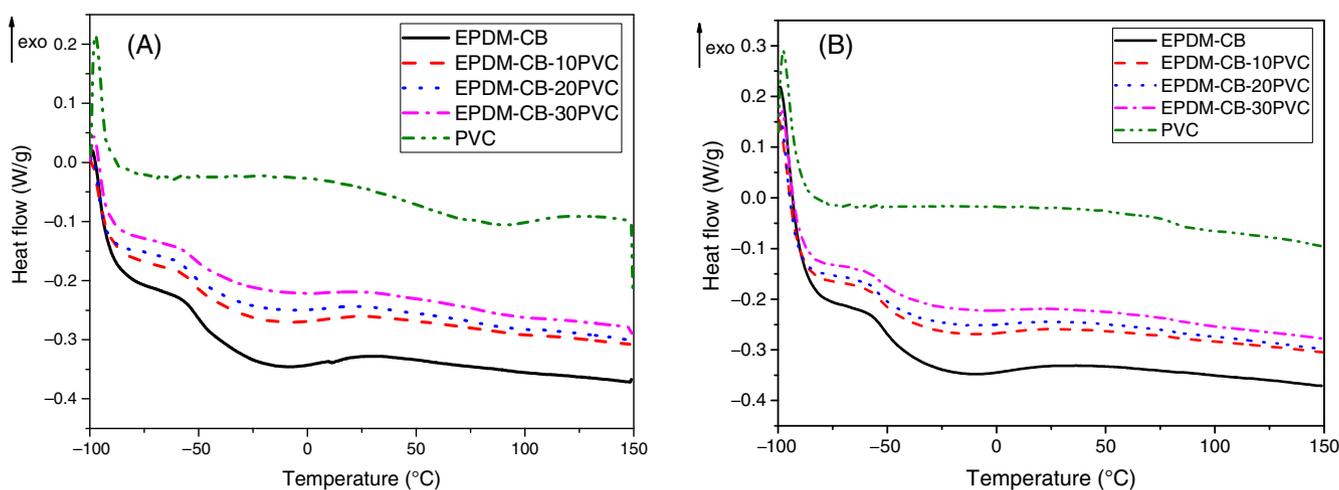


FIGURE 2 DSC thermograms of compacted EPDM-CB and relative EPDM-CB-PVC composites: (A) first heating scan and (B) second heating scan. CB, Carbon black; DSC, differential scanning calorimetry; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride

TABLE 3 Results of DSC tests on neat EPDM-CB and PVC and relative EPDM-CB-PVC composites

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	—
EPDM-CB-10PVC	-52.49	87.11	-53.65	80.84
EPDM-CB-20PVC	-53.41	78.48	-54.90	81.75
EPDM-CB-30PVC	-52.15	77.23	-53.32	83.40
PVC	—	—	—	85.10

Abbreviations: CB, Carbon black; DSC, differential scanning calorimetry; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride.

TABLE 4 Density of compacted EPDM-CB-PVC composites

Sample	Experimental density (g/cm ³)	Theoretical density (g/cm ³)
EPDM-CB	0.9506 ± 0.0025	—
EPDM-CB-10PVC	0.9624 ± 0.0027	0.86204
EPDM-CB-20PVC	0.9800 ± 0.0009	0.77348
EPDM-CB-30PVC	0.9564 ± 0.0047	0.68492
PVC ^a	0.0650 ± 0.0010	—

Abbreviations: CB, Carbon black; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride.

^aBulk density.

composite containing 30 wt% of PVC showed lower density compared to that containing 20% owed to lower quantity of rubber matrix. Comparatively, the theoretical density calculated for the produced composites is very low since the bulk density of PVC was about 15 times low compared to the EPDM-CB matrix. This confirms the compaction of PVC particles during the mixing and/or compression molding process of the rubber composites.

The mechanical properties of the compacted EPDM-CB-PVC composites are compared in the plot shown in Figure 3. Pure EPDM-CB matrix is normally characterized by a high elongation at break (ca. 800%; Figure 3A). With the progressive introduction of PVC phase in the matrix, however, the mechanical properties tend to be affected significantly. The addition of PVC in rubber matrix significantly reduced the strain at break of the composites. Looking at Figure 3B, the introduction of just 10% of PVC in the rubber phase, the modulus nearly doubled compared to the neat EPDM-CB sample, whereas the elongation at break nearly halved. The same can be verified by the declining strength value (also half with respect to pure EPDM-CB). Afterward, the addition of PVC does not significantly affect the mechanical properties with the exception of modulus values which rose to about 10 MPa in the case of EPDM-CB-30PVC. On the contrary, the strength values nearly stabilized but still

declined slightly along with the elongation at break values. The variations in modulus and elongation-at-break values are consistent with the fact that the addition of the rigid PVC filler in the soft rubber phase increases the stiffness of the blends. This consequently increases the brittleness of the compacted composites while reducing the elasticity of the material.

The investigation of the morphology of the broken specimens after the tensile tests was performed using FESEM and the resultant images are compared in Figure 4. The pictures of pure EPDM-CB (Figure 4A) showed a flat fracture surface; however, with the addition of PVC chips, a rough fracture surface can be observed. In all three images of EPDM-CB-PVC composite fracture surfaces (Figure 4B,C,D), an appreciable adhesion between the matrix and filler can be observed with no evident debonding of the phases. At PVC concentration of 10 wt% in the rubber matrix, the fracture surface appears to be a mixture of EPDM-CB matrix and PVC filler. However, at 20 and 30 wt% of PVC, the fracture surface is prevalently dominated by PVC. It has been reported in past^[23] that the PVC and EPDM do not show any compatibility; hence, the mixture is completely heterogenous without any physical/chemical interactions between the two phases. Hence, compatibilizers of different nature have been used to improve adhesive properties between rubber and PVC phases.^[23,34,35] For the current work, the compounding was performed without any compatibilizer to evaluate the effect of added PVC particles in EPDM.

To determine the effect of PVC loading on the strain recovery of EPDM-CB, the compression set test was carried out. The experimental results are compared for all EPDM-CB-PVC composites in Figure 5. It is clear that for the pure EPDM-CB, the compression set is low; however, as the PVC content increases, the compression set progressively increases at both testing temperatures. At 10, 20, and 30 wt% of PVC loading, the compression set values increased by 50%, 103%, and 160% at 23°C, whereas at 70°C, the increase was 16%, 31%, and 75%, respectively. As the PVC content increases, the availability of soft matrix phase decreases, hence reducing the capability of the blends to recover the strain applied for a prolonged time.

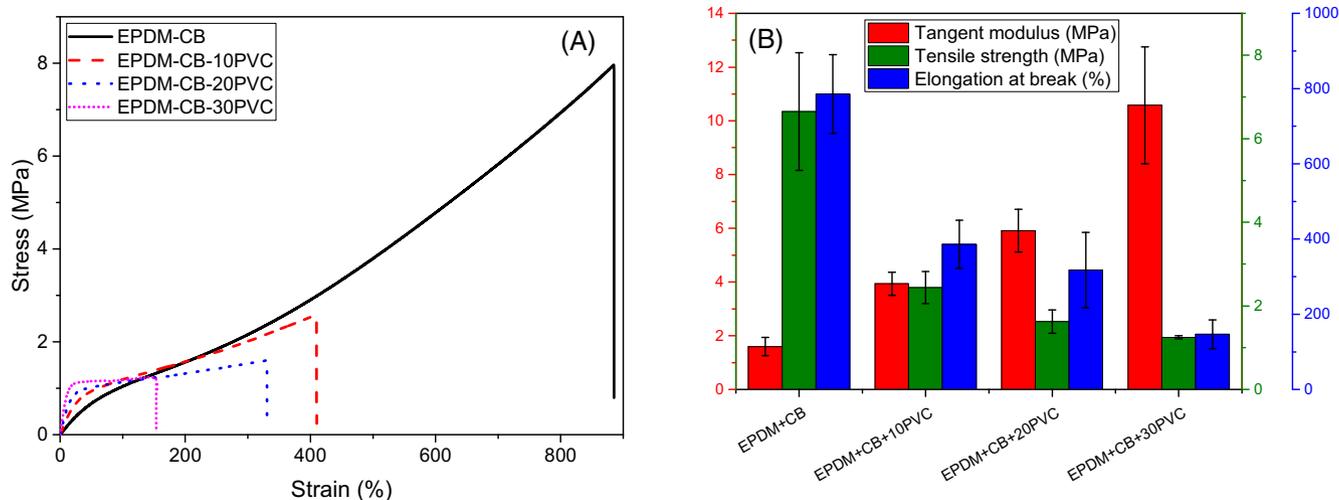
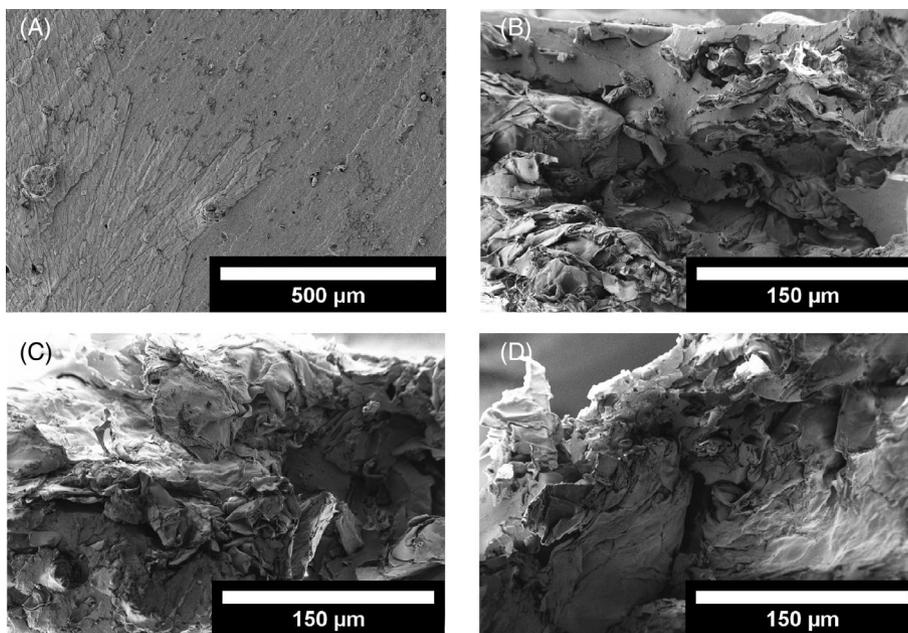


FIGURE 3 (A) Representative stress–strain curves and (B) comparison of the mechanical properties of the compacted EPDM-CB and relative EPDM-CB-PVC composites. CB, Carbon black; EPDM, ethylene–propylene diene monomer; PVC, polyvinyl chloride

FIGURE 4 SEM images of EPDM-CB and relative EPDM-CB-PVC composites after tensile test. (A) EPDM-CB, (B) EPDM-CB-10PVC, (C) EPDM-CB-20PVC, and (D) EPDM-CB-30PVC. CB, Carbon black; EPDM, ethylene–propylene diene monomer; PVC, polyvinyl chloride



Consequently, it results in a permanent deformation of the composite specimens during compression set tests.

Shore A hardness data are compared in Figure 6. The values substantially reflect the trend of the tangent modulus seen in the mechanical properties (Figure 3). In fact, as the concentration of PVC filler increased in the rubber matrix, Shore A values linearly increased up to EPDM-CB-20PVC. Such increase confirms the effect of rigid PVC particles added in the rubber matrix which contribute to the hardening of the composite panels.

Considering the improved mechanical properties by the addition of PVC particles, tear strength of the produced rubber composites was also investigated, of which the obtained results are summarized in Table 5. As

evidenced from the previous mechanical tests, the tear strength values also improved with the addition of PVC particles. This improvement could be credited to the increased stiffness of the EPDM-CB rubber by the added rigid particles which had increased the tensile modulus of the composite. The same was also responsible for the increased shore hardness values of the rubber panels.

The thermal conductivity values of produced rubber panels at various temperatures are compared in Figure 7. It is worthwhile to note that the thermal conductivity decreased almost linearly with the addition of PVC filler in the rubber matrix until 20% concentration; afterward, it remained practically constant for EPDM-CB-30PVC. One reason for such declining trend of the thermal

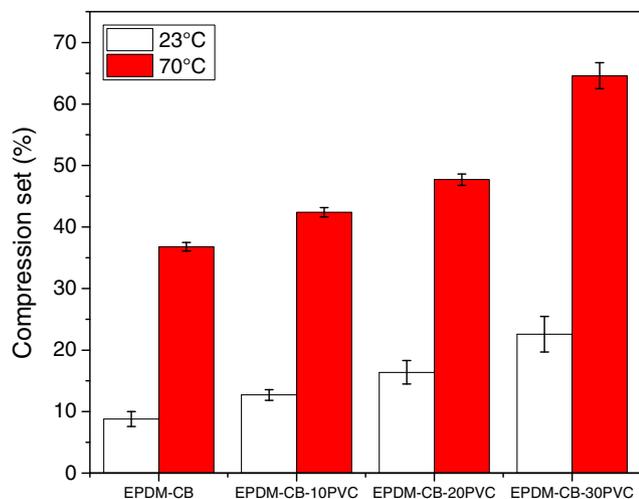


FIGURE 5 Compression set of neat EPDM-CB and relative EPDM-CB-PVC composites. CB, Carbon black; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride

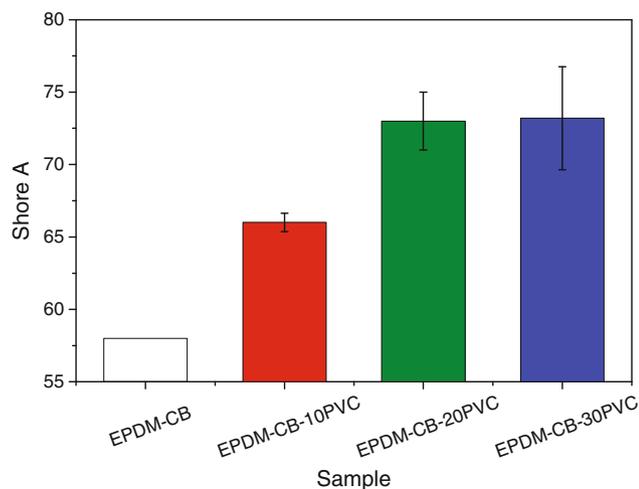


FIGURE 6 Shore A hardness measured of compacted EPDM-CB and relative EPDM-CB-PVC composites. CB, Carbon black; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride

TABLE 5 Tear strength values of compacted EPDM-CB-PVC composites

Sample	Tear strength (kN/m)		
	Peak	Valley	Mean
EPDM-CB	2.82	1.99	2.41
EPDM-CB-10PVC	3.69	2.76	3.23
EPDM-CB-20PVC	4.32	3.14	3.73
EPDM-CB-30PVC	3.91	2.92	3.42

Abbreviations: CB, Carbon black; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride.

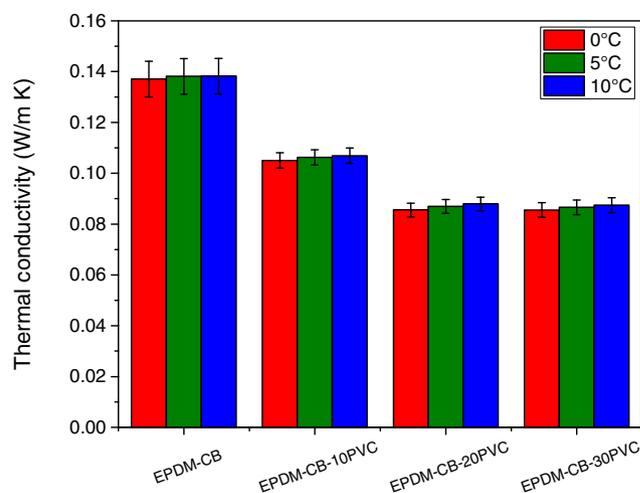


FIGURE 7 Thermal conductivity of compacted EPDM-CB and relative EPDM-CB-PVC composites measured by heat-flow meter HFM 446. CB, Carbon black; EPDM, ethylene-propylene diene monomer; PVC, polyvinyl chloride

conductivity values could be the disruption of thermal conductive chains of EPDM rubber due to the added PVC particles. In addition, the increasing PVC content in the continuous matrix of EPDM-CB results in the increase of EPDM-CB/PVC interfaces which contributed to the resistance to heat flow. Thermal conductivity values became stable at 20% PVC concentration; afterward, increasing the PVC content to 30% did not change the thermal conductivity values.

4 | CONCLUSIONS

In this work, recycled PVC chips were mixed with EPDM rubber at 60°C and the composites were vulcanized at 160°C for 20 min by a compression molding. Tensile tests revealed an increase of modulus but a decrease in ultimate mechanical properties of EPDM-CB-PVC composite with the progressive addition of PVC chips. Fractography of broken specimens showed a homogenous distribution of the PVC phase within the EPDM-CB matrix, where at higher concentration of PVC loading, the fracture plane dominated with the presence of PVC particles only. The increased stiffness of EPDM-CB-PVC composites resulted in increased Shore A hardness as well as compression set values. However, the added PVC filler in EPDM remarkably decreased the thermal conductivity of the composite, hence opening the possibility of industrial applications of EPDM-CB-PVC composites.

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AUTHOR CONTRIBUTIONS

Haroon Mahmood performed the experiments, analyzed the data and wrote the manuscript; **Fabiano Nart** provided materials for experiments and helped perform the analysis with constructive discussions; **Alessandro Pegoretti** obtained the funding, conceived the idea, verified the analyzed data, corrected the manuscript and monitored the project.

DATA AVAILABILITY STATEMENT

No original data is available to public.

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