Development of SWCNTs-reinforced EPDM/SBR matrices for shock absorbing applications

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Abstract

The present research investigates the effects of various concentrations of single-walled carbon nanotubes (SWCNTs) on the viscoelastic and thermomechanical characteristics of styrene-butadiene/ethylene propylene diene polymer (SBR/EPDM) blended polymeric matrices (60:40). Standard elastomeric methods are used to synthesise SWCNTs-reinforced EPDM/SBR nanocomposite blends. The results reveal that high SWCNTs concentrations offer remarkable thermal stability enhancement. SWCNTs reinforced with 0.6 mass% EPDM/SBR become 20% harder, with Tangent Delta values boosted by 70% with maximum cross-linking, elongation enhanced by 38% and tensile strength improved by 35%. With the 38% elongation enhancement, storage modulus is increased by 80%, whereas compressive strain is reduced by 20%. The planned hybrid nanocomposites exhibit viscoelastic characteristics against applied shock, in which the viscous component is associated with spongy structure, whereas the elastic component is associated with spring-like response.

1. Introduction

At present, much research is being undertaken developing polymeric materials by blending two or more elastomers. The most notable benefit of such approaches is that in certain instances the intermediate properties of the blend are superior to those held by either constituent elastomer. Elastomer blends are extremely useful in many industrial applications, as they provide superior solid-state properties, are easier to process and have a longer service life [1].

Researchers in both industry and academia have recently shown extreme interest in rubber-carbon nanotube (CNT) hybrid composites, because of their vastly superior mechanical and thermal stability, as well as their superior flammability and barrier properties. Much interest has been expressed regarding rubber hybrid composites with nanofiller reinforcement. Iqbal et al reported the influence of different quantities of carbon nanoparticles on the spectral, thermomechanical and rheological aspects of ethylene propylene diene terpolymer (EPDM) sponge nanocomposites [1]. Bashir et al studied the vulcanisation rheology outcomes of blending natural rubber (NR) with EPDM. They found that the curing features and viscoelastic nature of the designed composites were influenced by the blend ratio of the host elastomeric matrix [2]. Naskar et al described thermoplastic vulcanisates with superior characteristics for heat and oil ageing behaviour. Hydrogenated nitrile butadiene rubber/Nylon 12 (HNBR/PA12) and carboxylated HBNR (XHNBR)/PA12 with various blend ratios were fabricated with favourable cross-linker to check the dynamic vulcanisation properties [3]. Nouri et al presented the study of blends of nitrile butadiene rubber (NBR) and ethylene-vinyl acetate with various concentration of NBR and organoclay loadings prepared in a dispersion kneader. They investigated the spectral, mechanical and frequency-dependence characteristics in order to confirm the documented enhancements [4]. Tang et al conducted a degradation study for crumb rubber modifier with the production of terminal blend asphalt binder [5]. Kim et al documented enhancement in the thermomechanical properties of sponge EPDM rubber composites [6]. They studied the mechanical and microstructural properties of sponge composites other than EPDM and established a relationship between foam cell density and mechanical properties.

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The purpose of the present study was to develop high-strength, foamy-structure polymer composites with an optimal filler-to-matrix ratio for advanced engineering applications. We observed that progressive addition of carbon content in the rubber matrix efficiently modified the structural, vulcanisation, viscoelastic and mechanical properties of fabricated composites. EPDM is widely recognised as an engineering elastomer that is resistant to the effects of weather, chemical attack and oxidation, while also showing useful dielectric and mechanical properties of fabricated composites. EPDM is widely recognised as an engineering elastomer that is of particular interest, as they show superior electrical, thermal and mechanical properties. They also show potential as a reinforcement in blended nanocomposites, in which they greatly enhance mechanical, thermomechanical, thermal stability and barrier properties. Meanwhile, the development of sponge structures in elastomers can be achieved by introducing a gaseous phase that evolves from blowing agents used in vulcanisation. Specific commercial applications for sponge/cellular elastomeric materials require high-impact resistance and buoyancy, for example, shock absorbers, gaskets and body armour. Nanocarbon is incorporated into the host elastomeric matrix SBR to attain the levels required for various mechanical properties. In use, SBR performs at a superior level in terms of mechanical strength and thermal resistance when nanofiller is incorporated.

Nanoreinforcement using nanotubes has the potential to produce hybrid composite systems with superior properties. The novelty of the present research is to tune the thermomechanical, rheological and viscoelastic response characteristics of hybrid nanocomposite specimens of EPDM/SBR reinforced with SWCNTs for improved efficacy of shock absorption in advanced engineering applications.

2. Experimental

2.1. Materials

Ethylene propylene diene monomer (EPDM; KELTAN 4331 A) and SBR were supplied by Technical Rubber Products (China). Sulphur, CB, zinc oxide and stearic acid were provided by Merck (Darmstadt, Germany). Blowing agent azodicarbonamide (ACPW) was obtained from Qingdao Xiangsheng (Shandong, China). Mercaptobenzthiazolesulphide (MBTS) and diphenyl guanidine (DPG) were bought from Dalian Richon Chemical Co. Ltd (Dalian, China). Aromatic oil was purchased from International Petrochemicals (Pvt.) Ltd (Lahore, Pakistan). Single-walled CNTs (chirality, carbon > 90%, ≥77% [carbon as SWNT], 0.7–1.1 nm diameter, catalytic carbon vapour deposition method) were provided by Sigma Aldrich (St. Louis, MO, USA).

2.2. Fabrication

A dispersion kneader was employed to guarantee uniform dispersion of SWCNTs when added to rubber (EPDM/SBR). This kneader was operated at 110 °C for 30 min. Activators (zinc oxide) and lubricators (aromatic oil and wax) were then added to the kneader. Two roller mixing mills operating at 60 °C were run at 40 rpm for 20 min to mix the SWCNTs-EPDM/SBR rubber with accelerators (MBTS and DPG) and cross-linker (sulphur). The basic formula for the blend was a ratio of 60% EPDM and 40% SBR. This mixture underwent further modification when SWCNTs were incorporated. Table 1 shows the preparation of four pre-cured hybrid composite sheets with a variety of levels of SWCNTs: 0%, 0.2%, 0.4% and 0.6% of total mass.

| Materials | Pphr |
|-----------|------|
| Ethylene Propylene Diene Monomer (EPDM) | 100 |
| Single Walled Carbon Nanotubes (SWCNTs) | 0 |
| Styrene Butadiene Rubber (SBR) | 0 |
| Wax | 1 |
| Zinc Oxide (ZnO) | 5 |
| Stearic Acid | 1 |
| Diphenyl guanidine (DPG) | 1 |
| Sulphur | 1.5 |
| MBTS(Mercaptobenzthiazolesulphide) | 1 |
| Azodicarbonamide (ACPW) | 5 |
| Aromatic Oil | 10 |

Table 1. Base formulation for SWCNTs-EPDM/SBR blended nanocomposite.
A hot isotactic press was employed to cure the hybrid composites at 130 °C for 30 min at 15 MPa pressure. Once cured, the hybrid composites underwent conditioning (24 h maturation at 25 °C) before being tested.

2.3. Characterization

Scanning Electron Microscopy (JEOL JSM-6490A) was undertaken for the analysis of sponge structure in SWCNTs-EPDM/SBR hybrid composites. Cross-sections of the hybrid composites were prepared by immersing them in liquid nitrogen and fracturing them, producing a fractured surface morphology.

A durometer (Shore A) was employed for the assessment of hardness of the SWCNTs-EPDM/SBR hybrid composites in accordance with ASTM 2000. In accordance with ASTM 6204, an oscillating disc rheometer (Gibitre Instruments S.R.I., Bergamo, Italy) was used to investigate rheological variables, including viscosity variations, curing behaviours and tan δ. The rheometer comprises upper and lower plates and uses its own heating system to reach the necessary temperature of 190 °C ± 0.5 °C, with an oscillation angle of 0.5%. The sample was cured by inserting it between the heated plates. In the curing process, it was noted that the maximum torque occurred in the last stage of cross-linking. The impact on torque of the gradual rise in cross-linking and curing (cure rate index, CRI) was calculated as

\[ CRI = \frac{100}{(t_{90} - ts2)} \]

where t90 represents the optimal cure time to achieve 90% vulcanisation and ts2 represents the scorch time for raising two units from minimum torque.

A thermogravimetric analyser (Perkin Elmer Diamond TGA, Waltham, MA, USA) was used to characterise the thermal endurance and heat flow response of the material. This experiment was undertaken with a nitrogen flow rate of 300 ml min⁻¹, a heating rate of 10 °C min⁻¹ and a temperature range from 25 °C to 800 °C.

Dynamic mechanical analysis using a DMA 8000 (Perkin Elmer) was employed for the evaluation of storage modulus, loss modulus and hybrid composite damping. This experiment was undertaken at a frequency of 1 Hz in three-point bending mode. The relaxation spectra were in a temperature range between −15 °C and 150 °C with heating rates of 10 °C per min. A universal tensile testing machine (UTM; AG-20 KMXD Plus, Shimadzu, Kyoto, Japan) was employed for testing the tensile properties of the material in accordance with ASTM D412–98A.

3. Results and discussion

3.1. SWCNTs dispersion in EPDM/SBR blended hybrid composites

Figure 1 shows representative SEM micrographs for the cross-sectional fracturing of hybrid composite. The influence of SWCNTs and EPDM/SBR blends in terms of foaming efficiency microstructure can be seen in this figure (neat EPDM (ESC0) and SWCNTs-EPD/SBR blended (ESC1, ESC2, ESC3)). It is clear from the SEM micrographs that there is a fine dispersion of SBR in EPDM caused by the bridge effect of SWCNTs at the interface, increasing the matrices’ interfacial adhesion. This strong interfacial adhesion creates the enhanced rigidity and hardness for the nanocomposites, with reduced material flow-ability, which means that less gas is produced and fewer bubbles formed in the nanocomposite. SEM micrographs show a gradual decrease in microporosity and the formation of sponge structure with increase of SWCNTs content in blending ratio. The reason behind this result was supported by the crosslinking density as the nanocomposite undergoes sulfur vulcanization [17, 18]. This is because sulfur has less affinity with EPDM than it does with the more polar phase of SBR.
3.2. Mechanical characteristics

Figure 2 shows variations in hardness between pure EPDM/SBR nanocomposites and those blended with SWCNTs at a variety of concentrations. As can be seen, nanocomposites become notably harder when SWCNTs are added. Adding 0.2, 0.4 and 0.6 mass% SWCNTs causes respective hardness increases of ∼20%, ∼34% and ∼49% versus an unadulterated EPDM/SBR blend. The hardness is improved because of the nanoscale interface contact between SWCNTs and the EPDM/SBR blended nanocomposite polymeric chains. The interaction between SWCNTs and the rubber matrix creates mechanical interlocking that permits load to be efficiently transferred between the matrix and nanotubes [19, 20].

Additionally, an improvement in tensile strength for the EPDM/SBR blends was noted for every SWCNTs loading fraction (figure 3). The greatest enhancements to ultimate tensile strength (UTS; ∼35%) and elongation at break (EAB; ∼79%) occurred at the highest SWCNTs loading tested: 0.6 mass%. These improvements are attributable to better dispersion of SWCNTs, allowing them to forge physical links with chains in the rubber, enhancing the efficiency of load transfer. Better dispersion improves the interparticle distance for SWCNTs, with better binding with the elastomer causing a thick immobilised polymeric layer to develop across the SWCNTs surface. As the filler-elastomer interfacial interactions are improved with increasing SWCNTs dispersion, slippage of the elastomeric chains on the SWCNTs’ surface ceases. Past this point, they stretch at low strain through slippage and thus become stretched straight polymer chains. These stretched straight polymer chains have the same behaviours as high-strength polymer fibres, showing the same mechanical characteristics of viscoelastic material when stretched, while creating links to the nanofiller particles so that the stress is shared. Additionally, the nanofiller linkage connects via the stretched straight polymer chains, encouraging stress transmission across the elastomeric matrix via nanofiller [21, 22].

Figure 4 shows a plot of load versus compression. Compression sites for applied external loads are provided by the sponge structure inside the SWCNTs-reinforced EPDM/SBR blend nanocomposites. The SWCNTs-EPDM/SBR blend sponge nanocomposite specimen experiences a 20% reduction in compressive strain when 0.6 mass% SWCNTs is incorporated into the EPDM/SBR polymeric matrix. SWCNTs cause a reduction in bubble formation for the nanocomposite, which in turn causes a reduction in compressive strain. This is because the SWCNTs are evenly dispersed through the polymeric blend, cross-linking with the polymeric chains. At the start of the plateau region, buckled cell walls start to come into contact, rapidly enhancing stress until the sponge structures densify completely [23].

Figure 5 depicts impact energy absorption. Freefall absorption efficiency is clearly impacted by the blended sponge effect caused by even dispersion of SWCNTs within EPDM/SBR. The applied impact is dampened because of the foamy structures created in the composite during fabrication. Energy absorption decreases with increasing SWCNTs content, showing a substantial reduction of 12% at the highest SWCNTs content tested (0.6 mass%). Uniform distribution of SWCNTs through the blend reduces bubble formation in the nanocomposite, as can be seen from SEM micrographs. This can be attributed to the strong network structure and bridging of
Figure 3. (a) Stress- Strain Curve of designed nanocomposites, (b) Variation in tensile strength of EPDM-based nanocomposites with various SWCNTs loadings, and (c) Variation in elongation at break for EPDM-based nanocomposites with various SWCNTs loadings.

Figure 4. Variation in compressive strain for SWCNTs-EPDM/SBR blended nanocomposites.
SWCNTs, filling the nanoscale spaces in the polymeric chain and causing reductions in damping efficiency/energy absorption in the blended sponge nanocomposite [24].

### 3.3. Dynamic mechanical behavior

Dynamic mechanical analysis (DMA) measures variations in nanocomposite stiffness as a function of temperature for various levels of SWCNTs content in the EPDM/SBR blend. Figure 6 shows temperature-dependence for storage modulus for the neat blend and for blends with SWCNTs added. At room temperature, storage modulus for the neat blend is approximately 0.6 MPa. Addition of 0.2, 0.4 and 0.6 mass% SWCNTs improves the storage modulus in comparison with the neat blend by 20%, 55% and 67%, respectively. This effect is attributable to the enhanced stiffness caused by the addition of SWCNTs and secondarily to improved dispersion. The SWCNTs work as compatibilisers and boost the interfacial interactions of the blend with the CNTs, engendering more substantial stress transfers. The same kind of storage modulus improvement is seen at 100 °C.

Results demonstrate that storage modulus is lowered as temperatures increase. Because of the energy dissipation that involves supportive movement for polymeric chains, this storage modulus trend is a marked one in the temperature range −25 °C to 50 °C. These findings are attributable to the polymeric chains’ arrangement in a stronger percolated network because of the more uniform distribution within SWCNTs-EPDM/SBR blend sponge nanocomposites.

The damping properties of the composites are affected by the dissipation of unrecoverable oscillation energy in each cycle. The loss modulus as a function of temperature for the blend reinforced with SWCNTs is shown in figure 7. When the level of SWCNTs in the blend matrix is increased, the loss modulus and thus damping characteristics decrease. This pattern is due to the increase in the blend’s cross-linking density with SWCNTs addition. The behaviour of the loss modulus curve shows a decrease in loss modulus as temperature rises, especially for temperatures between −25 °C and 50 °C [25]. As the temperature increases, the polymer chains become more mobile. When 200 Hz frequencies are applied, polymer chains show reduced ability to remain in their original positions, creating enhanced loss modulus for the nanocomposite. A 27% loss modulus reduction is observed for the highest SWCNTs content tested (0.6 mass%). SWCNTs networking inside polymeric chains causes improved molecular and intermolecular interaction. This finding is attributable to the improved
**Figure 6.** Variation in storage modulus of SWCNTs-EPDM/SBR blended composites.

**Figure 7.** Effect of SWCNTs loading on loss modulus of EPDM/SBR blended nanocomposites.
distribution of SWCNTs throughout the hosting elastomeric matrix, causing an increase in hindrance for chain segmental movement.

Tangent Delta ($\tan \delta$) measures viscous dissipation and elastic recovery for the blends with SWCNTs added, as shown in figure 8. Tangent Delta is employed as an indirect measure for the cross-linking density in the polymeric nanocomposite sample. It is clear that this value increases as the SWCNTs content in the blend increases. This effect is augmented by 66% and by 70%, respectively, at the onset and maximum cross-linking stages for the highest SWCNTs content tested (0.6 mass%). Interfacial adhesion and interaction are enhanced, causing a lowering of the elastic recovery for the blend, which becomes a viscoelastic blended nanocomposite [26–28].

3.4. Rheological characteristics
Composites undergo permanent cross-linking during curing/vulcanisation. The rheological profile of designed nanocomposites is depicted in figure 9. Neat EPDM (ESC0) takes a longer time to cure, but when SWCNTs are added, the composite vulcanisation rate improves, and curing takes a shorter time (figure 10). Adding SWCNTs to the polymeric blend alters the structure of the material, with a percolated nanotube network forming among the polymeric chains in the SBR and EPDM matrices. This effect restricts the motion/flowability for polymeric chains, with the SWCNTs behaving as physical cross-linkers, accelerating the cross-linking/vulcanisation of the blend.

In vulcanisation with SWCNTs added, the rubber polymer chains gain some cross-links and decay links and some intermolecular cross-links between polymer chains. Figure 11 illustrates variations between onset/maximum torque with rheometry as SWCNTs content increases and sulphur vulcanisation takes place within the blend. When curing begins, torque values have a negligible influence because at this stage the nanocomposite is in a liquid form. At the maximum cross-linking stage, 40% depreciation occurs, attributable to the increased cross-link density caused by the SWCNTs influence on the blend’s curing reaction. Furthermore, the nanoscale interlocking of SWCNTs with the EPDM and SBR polymeric chains makes the two separate phases of the polymeric system more compatible.
3.5. Thermal properties

Thermograms obtained for a variety of SWCNTs loadings in rubber blends of EPDM/SBR are shown in figure 12. Each rubber blend demonstrates a two-stage thermal decomposition. Initial thermal degradation occurs at temperature ranges of 200 °C to 450 °C, as wax, aromatic oil and other processing elements evaporate. Thermal oxidation is next experienced at a temperature range between 450 °C and 520 °C, caused by the pyrolysis of the blend. As predicted, the introduction of SWCNTs to the blend retards the blend’s degradation. This increased thermal stability for the constructed nanocomposites is due to the restriction of thermal motion of the molecular polymer chains when heated. This restriction is due in turn to the nanotubes’ strong thermal...
Figure 11. Variation in Tangent Delta of the SWCNTs-EPDM/SBR blended nanocomposites.

Figure 12. Thermal decomposition analysis of SWCNTs-EPDM/SBR blended nanocomposites.
endurance and nanoscale interactions [29]. Thermal stability improves by up to 4% at 600 °C for the highest SWCNTs content tested (0.6 mass%) [30–32].

Figure 13 shows the boosting of heat absorption capabilities with increasing SWCNTs content in the blend. This thermal stability increase with added SWCNTs was also observed by Chen et al [33]. As the level of nanotubes in the blend increases, heat quenching capability also improves. Peak melting temperature increases with increasing SWCNTs content in the polymer matrix. This finding demonstrates enhancement of the blend’s thermal stability, as shown in the TGA thermograms [34, 35].

4. Conclusion

Progressive incorporation of SWCNTs into a blended polymeric nanocomposite causes remarkable enhancements in rheology response, viscoelastic behaviours and thermomechanical performance, in this case with an EPDM/SBR nanocomposite. Dynamic mechanical analysis shows that the incorporation of SWCNTs in an EPDM/SBR blend sponge polymeric system causes significant enhancement to storage modulus and a decline in loss modulus. Nanotube-reinforced blends also show remarkable improvements in both endothermic capabilities and thermal stability. Rheology examination shows that incorporating SWCNTs into the blend leads to an increase in Tangent Delta and a reduction of torque in the blend. Improvements are also observed in EAB, UTS and Shore A rubber hardness. Deteriorations with increasing filler-to-matrix ratio are observed in compressive strength and energy absorption efficiency. All of these effects are due to the superior mechanical strength, even dispersion and long-lasting bonding between matrix and filler possessed or engendered by SWCNTs.

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Conflict of interest

The authors declare that there is no conflict of interest.

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