EXPERIMENTAL AND NUMERICAL STUDIES ON THE MECHANICAL CHARACTERIZATION OF EPDM/ S-SBR NANO CLAY COMPOSITES

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Abstract: The present work is to investigate the effect of nanoclay loading on the blends of Ethylene-Propylene-Diene Monomer (EPDM)/Silica-Styrene-Butadiene-Rubber (S-SBR) nanocomposites through experimental and Finite Element analysis (FEA) studies. The nanocomposite specimens were prepared according to ASTM standard using open-mill mixer processing. The physical and mechanical properties of EPDM/ S-SBR nanocomposites were determined by measuring the tensile and tear properties, swelling properties, compression set, hardness and abrasion resistance properties. In the tensile tests for tensile strengthening evaluation, the tensile properties of EPDM/ S-SBR nanocomposite specimens have been measured at different temperatures using universal testing machine. From the hardness test, EPDM/S-SBR nanocomposites appears to be a good material for high temperature applications than polymer/clay nanocomposites as per standards of ASTM D-395 and ASTM D-471, compression set and swelling test specimens were prepared for determining the mechanical properties. Hardness, compression set and swelling resistance of nanocomposites increased due to increasing content of nanoclay as well as rebound resilience decreased. The failure modes of fractured surface are observed using Scanning Electron Microscopy (SEM) for EPDM/ S-SBR nanocomposite specimens. From the FEA results, the use of EPDM/ S-SBR nanocomposite specimens provide better results for Von Mises stresses which are much higher than EPDM/ nano-silica composite specimens.

1. Introduction
Nano composite materials are widely used in marine, aerospace and automobile applications due to high strength, stiffness and corrosion resistance of mechanical properties [1]. Many research scholars are investigated to produce the better significant importance and improve their performance for rubber polymer reinforced with nanoclay particles in the recent years. In the polymer Nano composites, the silica fillers produces good mechanical performance, thermal and barrier properties, when few percentage of silica is mixed with nano clay for modifications of mechanical properties [2]. If low nanoclay content in rubber with silica is producing the better mechanical properties and also cost effective additive such as nano clay, silica etc in nano polymer composites [3]. The specific surface area increases and the interaction between the rubber matrix and the reinforcing filler also
consequently increases due to silica filler particles decreases. This increase in matrix-filler interaction improves the properties of the nanocomposites [4-5].

The hydrophilic such as montmorillonite clay is converted into hydrophobic (organophilic) with most polymer matrix for having good compatibility and also most of the polymers materials are hydrophobic [6-9]. Due to organic modifications, the ion exchange between inorganic alkali cations on the clay surface with the desired organic cations and the interlayer cations of the clay are replaced by quaternary ammonium or alkyl phosphonium cations [10-11]. The uniform distribution of the organically modified montmorillonite clay in the rubber matrix enhanced the properties of the nanocomposites effectively. Many research scholars have investigated the clay nanocomposites with thermosetting and thermoplastics polymers for mechanical properties [12-15].

The aim of this study is to investigate the blends of EPDM and Silica-SBR filled with nanoclay for mechanical properties, hardness, compression set and swelling properties. This work is concerned with standard test methods for determining the mechanical behaviour of the nano clay composites as well as developing new materials for use in structural applications.

2. Specimen Preparation for Mechanical Characterization Studies
As per ASTM D-3182 standard, Two roll mixing mill process with standard friction ratio (1:2) used to prepare the EPDM/S-SBR specimens at 80°C. The Compounds of Nano composite Specimens are given in table 1.

| Sample code | Compounds (phr) |
|-------------|----------------|
| Ethylene-Propylene-Diene Monomer | styrene butadiene rubber | Silica | Nano Clay | Zinc oxide | Stearic acid | Dibenzothi azyledi sulfide | Sulphur |
| S_0 | 60 | 20 | 10 | 0 | 4 | 1.5 | 2.25 | 2.5 |
| S_2 | 60 | 20 | 10 | 0 | 4 | 1.5 | 2.25 | 2.5 |
| S_4 | 60 | 20 | 10 | 0 | 4 | 1.5 | 2.25 | 2.5 |
| S_6 | 60 | 20 | 10 | 0 | 4 | 1.5 | 2.25 | 2.5 |
| S_8 | 60 | 20 | 10 | 0 | 4 | 1.5 | 2.25 | 2.5 |
| S_10 | 60 | 20 | 10 | 0 | 4 | 1.5 | 2.25 | 2.5 |

First eight minutes, Ethylene-Propylene-Diene Monomer was pulverized and mixed with Silica-SBR. After the homogenization of the EPDM/S-SBR rubber mixed with nanoclay and also added properly as per ASTM standard procedure. The electrically heated hydraulic press used to mold the compounded blends under a pressure of 40 MPa at a 180°C and at an optimum curing time for 2 mm thickness of specimens [16]. The nanocomposites containing silica and sulphur system are labeled as S_0 (60/20/10/0 EPDM/SBR/SILICA/NC), S_1 (60/20/10/2 EPDM/SBR/SILICA/NC), S_3 (60/20/10/4 EPDM/SBR/SILICA/NC) and S_5 (60/20/10/6 EPDM/SBR/SILICA/NC), S_8 (60/20/10/8 EPDM/SBR/ SILICA/NC) and S_10 (60/20/10/10 EPDM/SBR/SILICA/NC).

3. Experimental Work for Mechanical Characterization Studies

3.1. Test Procedure for Tensile properties
Tensile test and tear test are used to measure the tensile and tear properties of nanocomposite specimen (600 mm x 430 mm x 380 mm). The specimens (dog bone, dumbbell test-shaped specimens) are mounted on the Instran 3367 Universal testing machine and eXpert 7600 Single Column Testing Machine. According to ASTM standards D3039 and D 412-C, Testing are performed at 23°C and at a crosshead speed of 0.15 mm/min as shown in figure 1.
3.2. Measurements of Swelling properties
The diamond cutting machine is used to cut a sample specimens (25 x 25 x 2 mm$^3$) according to ASTM D471 for Swelling test. After immersion method test, vacuum desiccator used to dry the specimens for 24 hours to obtain uniform absorption. The digital Vernier caliper is used to measure the thickness of the test samples with an accuracy of ± 0.01 mm. The specimens completely immersed in aliphatic (Benzene, Xylene, Toluene, Mesitylene) in glass diffusion bottles for removing the other particles until equilibrium swelling was obtained [5,17].

3.3. Measurements of Compression set
According to ASTM D395, the compression set test is carried out using compression machine. With help of an air circulating oven, the rubber is bulging on the device due to suitable clearance and also when a compressive load is gradually applied at 75°C for 24 hrs. After compression test completed successfully, the test specimen removed suddenly and allowable to cool for 40 min. The electronic digital vernier caliper is used to measure the final thickness of the specimens with 0.01 mm accuracy.

3.4. Test Procedure for Resilience of Rebound Properties
The Pendulum rebound resilience measurement as resiliometer is used to measure the rebound resilience of the nanocomposites according to the ASTM D 1054 and ASTM D 7121. The specimen was free from a given height with corresponding suspension of plunger and the rebound height was determined.

3.5. Test Procedure for Hardness and Abrasion resistance properties
Hardness test is conducted for nano composite specimen under varying loads and time to observe resistance of a materials under penetration. The Shore-A Durometer as per ASTM D-2240 is used to measure the hardness. For each test, the six readings are taken for plotting the graphs.

The Rotary Drum Abrasion Tester (DIN abrader) is used to determine the abrasion resistance of the nanocomposites according to DIN 53516, ASTM D5963 and ISO 4649 based on the volume loss. The test specimens are subjected to loading with help of the starting position of the DIN abrader test at ambient temperature or with the optional drum heating at elevated temperatures. After test, the emery paper of grade 40 (constant force of 10 N and at a constant speed of 0.3 m/s) used to abrade the specimens until abraded distance of 40 m was reached.
4. Results and Discussion

4.1. Mechanical Characterization of EPDM/SBR nanocomposites

The mechanical properties such as elongation, tensile strength, tear strength and 100% Modulus of the nanoclay reinforced EPDM/S-SBR nanocomposites are shown in figures 2-5. From the figures, it is observed that nanoclay loaded Ethylene-Propylene-Diene Monomer / silica-styrene butadiene rubber nanocomposite have marginally improvement in the mechanical properties. The elongation at break of EPDM/S-SBR rubber nanocomposites increases steadily as the content of nanoclay increases as shown in figure 2. When the nanoclay content is mixed up to 8 phr, the tensile strength is increased as shown in figure 3. Further increasing the nanoclay content, gradually decreases the tensile strength. Similarly, the content of nanoclay (mixing ratio) gradually increases in EPDM/S-SBR rubber nanocomposites, the tear strength gradually increases as shown in the Figure 4. The concentration of nanoclay (4-10 phr) gradually increasing in EPDM/S-SBR rubber nanocomposites, the 100% modulus of increased. If the nanoclay is added again in EPDM/S-SBR, the 100% modulus decreases as shown in figure 5. Due to the better dispersion of the nanoclay in the EPDM/S-SBR matrix, the mechanical properties were improved.

Figure 2. Nanoclay loading Vs Elongation of the EPDM/S-SBR nanocomposites.

Figure 3. Nanoclay loading Vs Tensile strength of the EPDM/S-SBR nanocomposites.

Figure 4. Nanoclay loading Vs Tear Strength of the EPDM/S-SBR nanocomposites.

Figure 5. Nanoclay loading Vs 100% Modulus of the EPDM/S-SBR nanocomposites.
4.2. Swelling properties
The nanoclay with silica particles are used in different applications such as automobile and barrier applications and also their lifetime extended due to chemical environments for improvement of mechanical properties [5]. The EPDM/S-SBR nanocomposites is analyzed due to nanoclay loading and nature of penetrants on mole percent. Figure 6 shows the mole percent uptake of aliphatic penetrants such as Benzene, Xylene, Toluene, Mesitylene by nanoclay loading. Due to uptake of benzene, there are significant changes on swelling properties for the nanoclay filled composites compared to unfilled compounds. It is observed that when the EPDM/S-SBR blend are exposed to the uptake of aliphatic penetrants with highest equilibrium uptake depends on the free space available in the polymer matrix. It can be seen from the figure 6, the % of mole content Benzene > Xylene > Toluene > Mesitylene is mixed with EPDM/S-SBR nanocomposites for improvement of mechanical strength. Due to better matrix filler interaction, there is a significant improvement in the strength of the EPDM/S-SBR nanocomposite from the swelling study. Depends on the penetrant molecule, the solvent penetrant and molecular mass are inversely related.

![Figure 6. Nano clay loading Vs % of mole content for penetration of EPDM/S-SBR nanocomposites.](image)

4.3. Compression Properties
The EPDM/S-SBR nanocomposites filled with nanoclay filler at 70°C for 24 hours used for compression test. As nanoclay loading increases upto 8 phr, the compression set increases and gradually decreases due to crosslinking density and mobility of the long rubber chains. If the compression strength is decreased, the EPDM/S-SBR nanocomposite as a better matrix filler material and also to be useful for structural applications as shown in figure 7. It is observed that the better retainable elastic properties produced due to lower compression set. Due to better matrix filler interaction, there is a significant improvement in the strength of the EPDM/S-SBR nanocomposite using compression test.

4.4. Rebound resilience
The six types nano clay specimens of EPDM/S-SBR rubber nanocomposite are tested and values are plotted as shown in figure 8. If the nanoclay content increased, the rebound resilience of nanocomposites gradually decreases. Due to better rubber-filler interaction, the decreasing tendency to be found from the figure 8. Whenever, the filler particles are increased in the rubber matrix, it is observed that the elasticity of the rubber chains and lower rebound resilience properties. The increasing incorporation of the nanofillers into rubber matrix, this can lead to increasing the hardness as well as reducing the rebound resilience, predominantly with more reinforcing filler.
4.5. Hardness and Abrasion Resistance properties

The effect of nanoclay loading on the hardness of EPDM/S-SBR rubber nanocomposites as presented in figure 9. If the nanoclay content increased, the hardness of EPDM/S-SBR rubber nanocomposites gradually increases. It is observed that nanoclay improved the stiffness of the rubber nanocomposites due to better rubber-filler interaction and also higher crosslink density.

The effect of nanoclay loading on the abrasion resistance study of EPDM/S-SBR rubber nanocomposites as presented in figure 10. The cylindrical shaped nanoclay filled EPDM/S-SBR nanocomposites specimens (diameter: 8 ± 0.1 mm and thickness: 10 ± 0.1 mm) is used for abrasion resistance study. If the nanoclay content increased, the abrasion resistance of EPDM/S-SBR rubber nanocomposites gradually increases. Due to the better rubber-filler interfacial adhesion and greater surface area, the improvement of hardness and abrasion properties may be resulting.
4.6. Morphology study by using Scanning Electron Microscopy (SEM)

Scanning electron microscopy have acceleration voltage of 30 kV, coated with a gold layer and used to find the microscopic evidence for the assumed assignment of failure modes. Experimental analysis of failure modes in EPDM/S-SBR nano composite specimens is a complex research subject, especially because the surface failure modes phenomena are superposed during mechanical testing. The fractured surface of the nanoclay filled EPDM/S-SBR rubber blends are investigated using SEM images as shown in the figures 11 (a-d). From the Figure 11 (a), it is clearly show that the tensile fracture surface having lesser tear line with lesser rougher surface and low energy/loaded needed to break the nanocomposites compared with figures 11 (b-d). The SEM observation shows the fractured surfaces such as many tear line, matrix crack lines, crack initiation and crack propagation as shown in the figures 11 (b), (c) and (d) due to high loading of nanoclay tends to form the agglomeration which contributed to poor dispersion.

![Figure 11. Tensile fractured surfaces of EPDM/S-SBR nanocomposites (a) S₂ (b) S₆ and (c) S₈ (d) S₁₀.](image)

5. Modelling and analysing of test specimen using FEA ANSYS

The 3D models of nano composite test specimen are used to carry out the static analysis based on the Von-mises stresses yield criterion using FEA ANSYS software. FEA software has been utilized to investigate the stress distributions on nano composite specimens under tensile loading [18].

5.1. Meshing of the model

The nano composite laminates of FEA model as per ASTM standard are developed using Layered 46, a three dimensional brick element. The nano layer is modelled using SOLID 45 and finer mesh is used for FEA model. The meshing of the EPDM/S-SBR nano composite specimens in a more sensitive manner by dividing it into small elements as shown in figures 12 (a) and 12 (b). The analysis for nano composite specimens are performed by applying a tensile load of 12 KN at the end, which was free to move in the longitudinal direction only (UY=UZ=0). Figure 12 shows the meshed model of EPDM/S-SBR nanocomposite test specimen and EPDM/ nano-silica composite specimens, which contains 16040 nodes and 7899 elements.

5.2. FEA Results

5.2.1. EPDM/S-SBR nanocomposite specimens The Total deformation and equivalent stress are analysed using FEA ANSYS as shown in the figures 13 (a) and (b). From FEA results, it is observed that the maximum deformation occurred is 1.0902 mm and Maximum equivalent stress induced is 1.3056 GPa for EPDM/S-SBR nanocomposite specimens. In the specimens, peel or maximum shear stresses occurred at the edge of specimen and maximum shear stress is 0.3 GPa are shown in the figure 14.
Figure 12. Meshed model of test specimen (a) EPDM/S-SBR nanocomposite specimens.

Figure 12. Meshed model of test specimen (b) EPDM/ nano-silica composite specimens.

Figure 13. (a) Total deformation of test specimen.

Figure 13. (b) Equivalent stress of test specimen.

Figure 14. EPDM/SBR Shear stress of test specimen.
5.2.2. **EPDM/ nano-silica composite specimens** From FEA results, it is observed that the maximum deformation occurred is 0.902 mm and Maximum equivalent stress induced is 1.2308 GPa for EPDM/ nano silica composite test specimen as shown in the figures 15 (a) and (b). In the specimens, peel or maximum shear stresses occurred at the edge of specimen and maximum shear stress is 2.8 GPa as shown in the figure 16. The results of deformation, equivalent stresses and shear stresses for various cases clearly explains that maximum deflection occurred at free end and the maximum resisting forces created at fixed end, which are fulfill and comes under the basic strength of materials theory. The numerical simulation of nanocomposites values of equivalent stress and maximum deformation are compared with experimental data as shown in Table 2. The FEA simulation results are within permissible limit and also suggested the application of EPDM/S-SBR nanocomposite as a strengthening materials to be useful for aerospace and automobile structural components.

![Figure 15.](image1.png)

(a) Total deformation of test specimen and (b) Equivalent stress of test specimen.

![Figure 16.](image2.png)

Figure 16. EPDM/ nano-silica Shear stress of test specimen.
Table 2. Experimental Results compared with FEA.

| Specimens                  | Experimental results | FEA results          |
|----------------------------|----------------------|----------------------|
|                            | Max. Deformation     | Max. Tensile Strength| Max. Deformation   | Max. Tensile Strength |
| EPDM/S-SBR nanocomposite   | 0.545mm              | 1.15 GPa             | 1.0902 mm          | 1.3056 GPa            |

6. Conclusions
The objective of this study is to investigate the mechanical characterization on ethylene-propylene-diene monomer/silica-styrene butadiene rubber (EPDM/S-SBR) blend nanocomposites using experimental and FEA.

(i) Due to the better dispersion of the nanoclay with silica particles in the EPDM/S-SBR matrix, the mechanical properties are improved.

(ii) If the improvement in properties of mole content Benzene > Xylene > Toluene > Mesitylene, and also mixed with EPDM/S-SBR nanocomposites for increasing the mechanical strength using standard testing.

(iii) From the FEA results, the use of EPDM/S-SBR nanocomposite specimens provide better results for Von Mises stresses which are much higher than EPDM/nano-silica composite specimens.

(iv) The FEA simulation results are within permissible limit and also suggested the application of EPDM/S-SBR nanocomposite as a strengthening materials to be useful for aerospace and automobile structural components.

7. References
[1] Man-wai Ho, Chun-ki Lam, Kin-tak Lau, Dickson H.L. Ng and David Hui 2006 Mechanical properties of epoxy-based composites using nanoclays Compos Struct. 75 415-21
[2] Mostafa, A. Abouel-Kasem, M. R. Bayoumy, and M. G. El-Sebaie 2010 Rubber-Filler Interactions and Its Effect in Rheological and Mechanical Properties of Filled Compounds Journal of Testing and Evaluation 38 3
[3] Vasanth Chakravarthy Shunmugasamy, Chongchen Xiang and Nikhil Gupta 2015 Clay/Polymer Nanocomposites: Processing, Properties, and Applications Hybrid and Hierarchical Composite Materials 6 161–194.
[4] Abdol majid Alipour 2012 Fabrication and Characterization of Nanostructured Polymer Composites Prepared by Melt Compounding International Journal of Bioscience, Biochemistry and Bioinformatics 2 2
[5] S.Vishvanathperumal and S.Gopalakannan 2019 Effects of the Nanoclay and Crosslinking Systems on the Mechanical properties of Ethylene-propylene-diene Monomer/styrene Butadiene Rubber Blends Nano composite Silicon 11,117–135
[6] Asma Yasmin, Jandro L. Abot and Issac M. Daniel 2003 Processing of clay/epoxy nanocomposites by shear mixing Scripta Mater. 49 81-86
[7] N. Sheng, M.C. Boyce, D.M. Parks, G.C. Rutledge, J.I. Abes and R.E. Cohen 2004 Multiscale micromechanical modeling of polymer/clay nanocomposites and the effective clay particle Polymer 45 487-506
[8] Jyi-Jiin Luo and Isaac M. Daniel 2003 Characterization and modeling of mechanical behavior of polymer/clay nanocomposites Compos. Sci. Technol. 63 1607-1616
[9] Naveed A. Siddiqui, Ricky S.C. Woo, Jyang-Kyo Kim, Christopher C.K. Leung and Arshad Munir 2007, Mode 1 interlaminar fracture behavior and mechanical properties of CFRP with nanoclay-filled epoxy matrix Compos Part A: Appl. Sci. Manuf. 38 449-60
[10] Mostafa, A., Aboul-Kasem, A., Bayoumi, M. R and El-Sebaie M. G 2009 Insight into the Effect of CB Loading on Tension, Compression, Hardness and Abrasion Properties of SBR and NBR Filled Compounds Mater. Des. 30 1785–1791.

[11] Hamza, S. S 1998 Effect of Aging and Carbon Black on the Mechanical Properties of EPDM Rubber Polym. Test. 17 131–137.

[12] Nunes, R. C. R., Fonseca, J. L. C. and Pereira M. R 2000 Polymer-Filler Interactions and Mechanical Properties of a Polyurethane Elastomer Polym. Test. 19 93–103.

[13] Zoran S., Young J., Ivan J., Sergei M., Natalia Y., Dale W., and Jan I 2004 Effect of Silica Nanoparticles on Morphology of Segmented Polyurethanes Polymer 45 4285–4295.

[14] Chang Y, Yang Y, Ryu S and Nah C 2002 Preparation and properties of EPDM/organomontmorillonite hybrid nanocomposites Polym. Int. 51 319-324.

[15] Kok, C.M., Yee and V.H 1986 The effects of crosslink density and crosslink type on the tensile and tear strengths of NR, SBR and EPDM gum vulcanizates Eur. Polym. J. 22 341-352.

[16] Chun-ki Lam, Hoi-yen Cheung, Kin-tak Lau, Li-min Zhou, Man-wai Ho and David Hui 2005 Cluster size effect in hardness of nanoclay/epoxy composites Compos Part B: Eng 36 263-269

[17] Liliane Bokobza 2017 Mechanical and Electrical Properties of Elastomer Nanocomposites Based on Different Carbon Nanomaterials Journal of Carbon Research 3 10.

[18] Rajkumar G, Senthil kumar M, Mohamed Bak K and Vijayanandh R 2019 Mechanical characterization of carbon fiber reinforced epoxy with carbon nanotubes International Journal of Mechanical and Production Engineering Research and Development 9 243-255.