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Development of a Flexible and Conductive Elastomeric Composite Based on Chloroprene Rubber

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Abstract

Flexible and conductive nanocomposites with enhanced mechanical and dielectric properties have been fabricated from reduced graphene oxide (RGO) reinforced polychloroprene rubber (CR). Composites were prepared on a two-roll mixing mill and the morphological analysis using SEM and TEM showed good dispersibility and exfoliation of RGO layers in the matrix. It imparted a critical role in the enhancement of mechanical and electrical properties of the composites. Composites with 0.9 phr of RGO showed an enhancement in tensile strength, tensile modulus and electrical conductivity as 92\%, 75\%, and 103 \% respectively more than the pristine polychloroprene. RGO formed a good conducting percolating network in the polychloroprene matrix and it led to the formation of a dielectric composite material with enhanced electrical properties.

Keywords: Reduced graphene oxide; chloroprene rubber; Atomic force microscopy; conductivity; dielectric properties.
1. INTRODUCTION

The fields of material science become more attractive and innovative by the invention of graphene and functional graphene materials. The mechanical, thermal, electrical and optical properties of graphene are far excellent and hence it is a promising alternative to conventional fillers. A major challenge in this respect is the permutation of conventional reinforcing fillers like carbon black, silica, clays etc. in the elastomeric vulcanizates by small amount of nanofillers (eg: graphene, CNT, GNP etc) to achieve desirable properties. Graphene oxide (GO) is one of the promising material for the mass production of graphene and the important advantages in this respect are the ease of accessibility of graphite and its processibility in aqueous solution. By the reduction of graphene oxide to reduced graphene oxide, functional groups such as epoxy and hydroxyl groups are removed and the electronic properties are partially restored [1].

Several research reports are available in the field of elastomeric composites with graphene/graphene oxide based materials such as silicon rubber [2], emulsion styrene butadiene-rubber (SBR) [3, 4], solution styrene-butadiene –rubber [5–7] synthetic natural rubber (cis-1, 4-polyisoprene) and epoxidized natural rubber (ENR) [8], nitrile rubber [9] and fluro elastomers [10]. Pressure sensitive composites with high reversible piezo resistive behavior based on silicon rubber and graphite nanosheets were developed by Soltani and Katbab[11]. Recently, Tian et al. [12] developed a high performance dielectric composite by latex compounding of GO/XNBR encapsulated carbon nanosphere hybrids. Kang et al. [13] found that the gas permeability of XNBR/GO composite is significantly lower than that of the neat matrix, but the higher mechanical property and low gas permeability of the composite are correlated to the strong interfacial interactions. Brigandi et al. reviewed the modification of graphene and the fabrication of graphene-based polymer composites and they found that graphene can bring out a dramatic improvement in properties at very low filler content [14]. The utilization of these materials in the composite fabrication with different polymer matrices has been explored. Polymer/graphene and graphene oxide composites show superior mechanical, thermal, gas barrier, electrical, conductive and flame retardant properties compared to those of the neat polymer [15]. The improvement in mechanical and electrical properties of graphene based polymer composites is much better in comparison to that of clay or other carbon filler-based polymer composites [16]. Graphene oxide sheets are more compatible with organic polymers and as a result GO has a considerable attention as reinforcing filler in polymer composites. Like graphene, RGO is electrically
conductive, which makes it suitable for developing conducting composites [17]. The pervaporation performance of CR/RGO membranes has investigated in chloroform/acetone azeotropic mixtures and the permeation rate and separation factor were evaluated [18]. Chloroprene rubber is well known for its high gum vulcanizate strength arising from strain induced crystallization. The uncured rubber also shows storage hardening due to slow crystallization. It has excellent physical properties, weather and heat resistance, and low temperature properties. Owing to the presence of halogen in the rubber molecule, chloroprene resists burning inherently better than any other hydrocarbon rubbers. There are no reports available based on Polychloroprene Rubber/Reduced Graphene Oxide nanocomposites so far and this paper significantly describes the role of RGO in improving its mechanical, thermal and conductive properties with a low loading of 0.9 phr.

2. EXPERIMENTAL

2.1. Materials

Polychloroprene rubber (W) was obtained from DuPont, USA (Mooney viscosity ML 1+4@100°C 47±1). Natural graphite powder was purchased from Sigma Aldrich India with 99% purity and particle size is <20 µm. Other reagents NaOH, NaNO₃, and KMnO₄ of analytical grade and 98% H₂SO₄, 30% H₂O₂ were purchased from Merck Ltd, Mumbai, and are used as received without any further purification.

RGO was prepared by modified Hummer’s method using NaNO₃, KMnO₄, 30% H₂O₂. The obtained graphite oxide slurry (GO) was vacuum-dried at 50°C for 24 h and thermally reduced at high temperature (400 – 500°C) for obtaining the RGO. CR/RGO composites were compounded according to ASTM D 3182 with the aid of a laboratory two roll mixing mill (150, 300 mm). For all the mixes the nip gap, friction ratio, and the number of passes were kept constant. The total compounding cycle was finished within 15 – 18 min and the friction ratio of the mixing mill was kept at 1:1.25 during the mixing tenures. The samples were prepared by curing on a hydraulic press at the optimum cure time. Samples prepared were designated as CG0, CG1, CG2, CG3, CG4 and CG5 in which the concentration of RGO varies from 0, 0.3, 0.6, 0.9, 1.5 and 2.5 parts per hundred rubbers respectively.
2.2. Characterization of RGO, RGO/CR nanocomposites

Raman Spectroscopy was performed with a Raman spectrometer (Horiba Jobin Yvon LabRam HR system). FTIR was analyzed with Perkin Elmer analyzer at a spectrum range of 400 – 4000 cm\(^{-1}\). XRD spectra of the composites were obtained from Bruker AXS D8 with wavelength 1.5406 Å. TEM analyses of the composites were investigated by means of JEM-2100 HRTEM. The cryocut specimens prepared using an ultra microtome (Leica, Ultracut UCT) and placed on 300 mesh Cu grids (35 mm diameter) for analysis. Thickness of the TEM samples after ultra microtome cutting was around 100 to 130 nm. Atomic force microscopy observations were carried out in air on as-received sample surfaces using Agilent 5500 AFM operated in contact mode. Measurements were carried out using standard triangular silicon nitride cantilevers. Thermogravimetric analysis (TGA) was done using Perkin Elmer STA 6000, Diamond TG/DTA with a heating rate of 10 °C/min from room temperature to 700 °C under nitrogen atmosphere. Differential scanning calorimetry (DSC) measurements were carried out on Mettler Toledo DSC 822e instrument at 10 °C/min under nitrogen atmosphere.

Measurement of mechanical properties was conducted at 25±2 °C according to ASTM standard using a universal testing machine, UTM (Tinius Olsen Universal testing machine H60KT) with a cross head speed of 500 mm/min using dumb–bell shaped specimens. Tear strength was also determined by the same instrument according to ASTM D624.

DC conductivity of the sample was obtained from Keithley electrometer with Delta mode system using 220 V. 2 mm thick samples were used to measure the conductivity of the composites. Dielectric properties were obtained from Precision Impedance analyzer Wayne Kerr 6500B meter with basic measurement accuracy of ± 0.05% and analytical frequency ranges from 5 to 120 MHz.

3. RESULTS AND DISCUSSION

3.1. Characterization of the synthesized RGO

The Raman spectrum of GO and RGO synthesized is shown in Figure 1. Raman spectra of RGO shows characteristic D band, G band and 2D band corresponding to the plane vibration of sp\(^2\) bonded carbon atoms and defection of graphitic layer respectively. 2D band is attributed to the development of graphene structure. It is clear from figure.1 that the conventional G and D band of graphite oxide was raised near 1580 and1350 cm\(^{-1}\) respectively. For RGO, D band (1356 cm\(^{-1}\)
G band (1583 cm\(^{-1}\)) along with 2D band (2634 cm\(^{-1}\)) were raised, which indicates the prominent growth of sp\(^3\) character due to the presence of oxygen containing functional groups [19]. By comparison, a lower density of defects was observed for both GO and RGO on the basis of D/G intensity ratio. Here we got I\(_D/I_G\) ratio of 0.83 for GO and 0.79 for RGO. If the I\(_D/I_G\) ratio decreases, it is expected the consequences of greater graphitization followed by reduction [20]. Moreover, RGO consisting of less number of layers was obtained by reduction. I\(_{2D}/I_G\) ratios were found to be 0.28. However the I\(_{2D}/I_G\) ratios of less than one suggests a multi layered graphene structure in RGO [21].

Figure 2a shows FTIR spectra of graphene and its functionalities. The presence of different types of oxygen functionalities in GO and RGO was confirmed by the stretching peaks at 3490 cm\(^{-1}\) (O–H stretching vibrations) and 1720 cm\(^{-1}\) (stretching vibrations from C=O). On the other hand, FTIR spectrum of RGO shows a series of new peaks compared to that of graphite. The peak corresponding to 2991 cm\(^{-1}\) is due to the oxygen functionalities in RGO and it is attributed to the C–H stretching vibration. RGO exhibited a very strong C=O (stretching) peak about 1736 cm\(^{-1}\) and to –CH bending vibration at 1540 cm\(^{-1}\). The peak at 1075 cm\(^{-1}\) corresponds to the presence of epoxide group in the RGO. The presence of different types of alkene functionalities like =C–H groups in RGO were confirmed by strong peaks at 953, 798 and 664 cm\(^{-1}\) etc. RGO showed a peak at 3010–3200 cm\(^{-1}\) which attributed to the stretching vibrations of –OH from C–OH, –COOH groups. Thus FT-IR spectrum confirmed the successful oxidation of RGO.

The XRD spectra are shown in Figure. 2b. GO shows a larger interlayer distance (0.87 nm at 2\(\theta\)=10.82º) due to the formation of hydroxyl, epoxy, and carbonyl groups during chemical oxidation [22]. But after reduction, the interlayer distance decreases to 0.37 nm (2\(\theta\)=24.60º) and is very close to the graphite values (2\(\theta\)= 28.53º corresponding to an interlayer distance of 0.35 nm) This is due to the removal of oxygen containing functional groups which is mostly associated with the ring opening of epoxides. This suggests that the conjugated graphene network (ie, sp\(^2\) carbon atoms) is re-established during the reduction of RGO. The flake-like structure of RGO was clearly observed from SEM images (Figure. 3) and these flakes have relatively large surface area.
3.2. Characterization of the CR/ RGO composite

3.2.1. Infrared spectroscopy (FT-IR)

FT-IR is an essential characterization technique used to elucidate the structure of a material at the molecular scale. Figure 4 shows the FT-IR spectrum of chloroprene rubber/RGO composites. The oxygen functionalities in RGO were confirmed by the presence of broad and intense peaks at 3490 cm\(^{-1}\), which is attributed to the strong O–H stretching vibrations of the C–OH groups. But when RGO is incorporated into the CR matrix the intensity of the peak decreased considerably. The peaks at 2850 and 1450 cm\(^{-1}\) are due to the –CH stretching and bending vibrations of the chloroprene rubber. The absorption bands at 1658 and 1712 cm\(^{-1}\) ascribed to the presence of strong stretching and bending vibrations of –C=C and –C=O groups in the composite respectively. The intensity of peak corresponding to –C=O (1712cm\(^{-1}\)) and –C=C (1658cm\(^{-1}\)) groups in the RGO were almost disappeared in the composites. These findings clearly demonstrate the presence of intermolecular interactions between RGO and CR matrix. Hence FTIR is used only to verify the incorporation of RGO into CR matrix. A similar trend was reported in the case of reduced graphene oxide composites [23]. Strong and sharp peaks at 821 and 600 cm\(^{-1}\) were assigned to the presence of alkyl halide like chlorine (C–Cl groups) in chloroprene rubber.

3.2.2. XRD Analysis

From the XRD spectra, the RGO showed a strong and sharp diffraction peak at \(2\theta = 24.7^0\) with interlayer distance of 0.37 nm (Figure. 5). The characteristic peak of RGO is disappeared from the spectra of CR/RGO composites, and is due to the exfoliation of RGO in the CR matrix.

3.2.3. Transmission electron microscopy (TEM)

The well exfoliated and distributed RGO layers in the CR matrix are clearly evident from the TEM images (Figure.6). In the case of CG2 and CG3 (composites loaded with 0.6 and 0.9 phr RGO), the state of dispersion of RGO is very good and a few layers of RGO can be seen in the matrix. But in the case of CG5 (sample loaded with 2.5 phr RGO) the state of dispersion is poor; it depicts the existence of agglomerates in the system. TEM images of the CG3 show a fine dispersion of RGO with layered structure without any agglomeration. The presence of agglomerates becomes obvious as concentrations increased from 1.5phr of RGO. Agglomerated portions were marked in the figure using red circles.
3.3. Mechanical properties of the CR/RGO composites

The stress strain curves of the RGO/CR composites are shown in Figure. 7. The stress–strain curve of the neat CR rubber is compared to that of RGO loaded counterparts. The elongation at break decreases from 498 to 395% with the incorporation of RGO into CR matrix. However, the tensile strength of 12.6 MPa is showed by CG3 sample, which is 92% higher than that of neat sample. This confirms the reinforcing effect of reduced graphene oxide where the filler is well dispersed in the CR rubber matrix and reinforces the matrix even at very low loading. One interesting property of CR rubber is that it crystallizes on stretching and so cured components are strong even without the addition of any reinforcing fillers. Since the double bond between carbon atoms is shielded by the pendant atoms and –CH2 groups, the molecular interlinking is essential for vulcanizing the polymer and is usually affected through the chlorine atom.

CR/RGO composites exhibit higher mechanical properties such as tensile strength, tear strength and hardness than pure chloroprene rubber due to its good compatibility and uniform distribution of exfoliated RGO within the rubber matrix (Table 1). Here tensile strength increases with filler loading and reaches an optimum value and then decreases. Composite with 0.9 phr of RGO (CG3) shows a good increment in tensile strength, tensile modulus and tear strength as 92%, 75%, and 12% respectively when compared to pristine polychloroprene. At the same time, further addition of RGO (CG5) reduces the mechanical properties due to the formation of stress concentration centers in the composites. Even at very low loading the surface area of RGO is high, which causes maximum contact points within the chloroprene rubber molecules, so it enable maximum possible interaction with the matrix. A similar trend is observed in the case of hardness of the composites which increases gradually with filler loading, and reaches a maximum and then decreases.

It is further confirmed by the surface roughness values of the CR/RGO composites obtained from AFM analysis. Figure.8 illustrate the surface topographical features of CG3 and CG5 composites in 2D and 3D view. For CG3 sample the distribution and dimensions of the heaps or hills are almost homogeneous, which indicates a good dispersion of RGO in the polymeric matrix. Surface roughness parameters including root mean square roughness (Rq) and average surface roughness (Ra) of CG3 are 0.13µm and 0.19 µm respectively and CG5 are 0.23µm and 0.31 respectively. This increase in surface roughness value for the CG5 sample compared to CG3 can be attributed to the aggregation of RGO in the CR matrix. The improvement in mechanical properties compared to the gum samples can be correlated with both AFM and SEM analysis.
Fracture surface of the filled CR vulcanisates is shown in Figure. 9 The SEM micrograph obtained from the CR vulcanisates showed a smooth surface with closely enclosed small particles. These particles are likely to be the metal oxides viz. ZnO and MgO, which were used in the curing of rubber. The smoothness of the fractured surface indicates uniform failure of the CR matrix without any mechanically weaker region for crack initiation. The SEM morphology also indicates that CR matrix has a good reinforcement with RGO, and the particles are homogeneously distributed throughout the matrix having good compatibility. There is no crack initiation observed in the interface. At higher filler loading, inadequate stress transfer occurred due to the agglomeration of filler particle in the polymer matrix and hence mechanical properties decreased, as it is evidenced from the AFM results. This promising mechanical strength of RGO offers the development of affordable and high quality composites.

3.4. Thermal analysis

3.4.1. Thermogravimetric analysis

The thermal stability of the composite was examined by thermogravimetric analysis (TGA) under nitrogen atmosphere. Figure. 10 show the TGA thermograms of the CR/RGO composites respectively. The nanocomposites were found to be stable up to a temperature of ~300 °C after that the CR composite undergoes two stages of thermal decomposition. From figure. 10, it is clear that there are two weight loss decomposition stages in the CR composite. Firstly, the elimination of moisture occurred corresponding to the minor weight loss around 150 °C. The major weight loss occurred at a temperature of nearly 315 °C due to the decomposition of the polymer network and then followed by slow step-by-step degradation up to 700 °C. The degradation is mainly due to the dehydrochlorination starting from 260 °C and reaches a maximum temperature of around 315 °C. This sudden degradation was studied by Kaisersberger et al. [24] and it might be due to an auto catalytic pyrolysis of chloroprene chain. The second stage degradation might be due to the evolution of some plasticizer used in the compounding of chloroprene rubber which starts around 360 °C and proceeds up to 500 °C, after which a slow and steady degradation is found until 700 °C. The thermograms showed that all the composites leave a higher amount of residue. Among all these composites, chloroprene loaded with 0.9 phr RGO (CG3) shows better thermal properties. In case of CG1, it contains only 0.3 phr of RGO and thereby the thermal stability is not improved as much as compared to other nanocomposites with more filler content. It might be due to the insufficient amount of the filler material to
enhance the thermal behavior of the CR composites. But with higher filler loading, the thermal stability of the CR/RGO composites increases and thereby it reduces the permeability of the volatile degradation products. It results into an improved heat insulation of the material.

3.4.2. Differential Scanning Calorimetric (DSC) Analysis

DSC heating curves of CR/RGO composites are given in Figure. 11. It is noted that when we are incorporating RGO, Tg of the polymer is shifted to the high temperature region. The Tg values are related to the mobility of polymer chains. Typically, the addition of filler increases the Tg by decreasing the mobility of the chains close to the surface of RGO. From figure, it is clear that single Tg is observed in all the systems studied. The glass transition (Tg) temperature of pure CR was observed at -42°C and the incorporation of reduced graphene oxide increase the Tg values of the composite to –41.61, –41.01, –39.53, –40 and –40.53°C with 0.3, 0.6, 0.9, 1.5 and 2.5 phr RGO respectively. All the composites showed a slight increment in the Tg value than that of pure CR. This is due to the effective exfoliation of RGO and the interfacial interaction between the RGO and the CR matrix. This might be due to the stiffening of rubber matrix by reduced graphene oxide with its better dispersion and distribution.

3.5. Dielectric properties of the composites

Figure.12a shows the dielectric permittivity (ε') of the RGO/CR composites as a function of frequency at room temperature. As expected, dielectric properties of the RGO/CR composites show a typical percolation behavior as the concentration of RGO increases. At low RGO content, the dielectric permittivity of the composites shows a gradual decreasing trend with increasing frequencies. Permittivity of a composite is determined by the number of orientable dipoles within the system, which has the ability to orient under an applied electric field. When the frequency of the applied voltage is increased, the larger polymer chains will orient themselves. So the effective permittivity of the elastomer will decrease with increasing frequency in a measured range. When RGO loading reaches about 0.9 phr, there is a percolation transition from insulator to conductor and thereby dielectric permittivity increases.

The increment in dielectric permittivity of conductive filler–polymer composites near to percolation threshold is due to the micro capacitor effect [25]. In the present work, conductive RGO is incorporated into a rubber matrix and it generates number of micro capacitor structures throughout the composites, which significantly increases the intensity of electric field. This
increased intensity further promotes the movement and accumulation of charge carriers at the interface between the RGO and CR matrix. This is known as Maxwell-Wagner-Sillars polarization [26] and is an important reason for the improvement of dielectric permittivity of the CR composites.

Figure 12b demonstrates the frequency dependence of AC conductivity of the composites with different RGO loadings. For the composites with low RGO content, the conductivity curves showed a strong dependence on frequency owing to their insulating nature. As the amount of filler increases, the AC conductivity of the composite increases simultaneously with frequency. This is due to the formation of filler networks in the polymer on higher loading. This creates a highly conductive path in the matrix which leads to an increased AC conductivity of the composites.

3.6. Electrical conductivity
Table 2 represents the variation of DC conductivity of the CR/RGO composites with filler concentration. Electrical properties of composites strongly depend on filler particle distribution through the polymer matrix. At lower filler loading the conductivity of the composite is higher than that of base polymer, because filler particles are isolated from each other by an insulating rubber matrix. When the concentration of filler in the rubber matrix increases then the filler particles begin to contact with each other and form a continuous network throughout matrix. This creates an easy path for the electrons to flow [27]. At higher filler loading this continuous network reduces the easy flow of electrons through it due to agglomeration and thereby electrical conductivity decreases.

4. CONCLUSION
In summary, the developed RGO/CR composites showed improved mechanical, thermal and electrical properties. The exfoliation and dispersion of RGO in the CR matrix was confirmed by FTIR, XRD and TEM analysis. Tg values showed good improvement when compared to neat polymer. The increase in mechanical and glass transition properties attributed to the better interaction between the RGO layers and the CR rubber. Dielectric properties of the composites showed typical percolation transition when increasing the amount of RGO. The AC conductivity of the composites increases almost linearly with the increase of frequency. The variation of DC
conductivity with concentration of filler in CR/RGO composites also confirmed good conductivity enhancement even at low RGO loading.

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**REFERENCES**

List of Tables

Table 1. Mechanical properties of CR/RGO Composites

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation at break Eb (%)</th>
<th>Tensile Modulus (MPa)</th>
<th>Tear Strength (N/mm)</th>
<th>Hardness (Shore A)</th>
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<tr>
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<td>498</td>
<td>1.77</td>
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<tr>
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<td>2.96</td>
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<tr>
<td>CG3</td>
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<tr>
<td>CG4</td>
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<tr>
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<td>46.00</td>
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Table 2. DC electrical conductivity of CR/ RGO composites

<table>
<thead>
<tr>
<th>Sample name</th>
<th>DC Conductivity [S/cm]</th>
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<td>CG4</td>
<td>5.74</td>
</tr>
<tr>
<td>CG5</td>
<td>5.00</td>
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</table>
Graphical Abstract
Highlights of the present work

- RGO reinforced Chloroprene rubber nanocomposite were prepared by mill mixing method
- Exfoliation of graphitic layers leads to better interaction between RGO and CR
- Overall improvement in mechanical and thermal properties
- Morphological studies confirms the improvement in characteristic properties
- 103% of improvement in electrical conductivity