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Experimental Study on Manufacture and Analysis of Rubber Nanoclay Mwcnt Composite

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Abstract—**The outstanding properties of Carbon nanotubes have generated scientific and technical interests in the development of nanotube-reinforced polymer composites. Therefore, we investigated a novel mixing approach for achieving a good dispersion of Multiwalled carbon nanotubes (MWCNTs) in a rubber blend with Nanoclay. The initial phase involves Nanoclay material Halloysite (Aluminosilicate nanotube) mixed with a sample of Raw Natural rubber (ISNR-20) in the proportion 1:10 using suitable solvent. The above composition is mixed using Mechanical Stirrer at an elevated temperature for a curing period of 3-4 hours until Rubber-Nanoclay composite is achieved. The Rubber composite and MWCNT's are dispersed in Toluene separately. This process is done to MWCNT's in order to disentangle them from lumps. The final phase is the Melt preparation process which involves the thorough blending of the above solutions at a temperature of 110˚C. It is further sonicated using a mechanical probe sonicator (Branson sonifier), in order to induce an efficient dispersion of nanotubes in Rubber Composite. This process is extended by preparing different composites by dissolving varying proportion of MWCNT's with fixed composition of Rubber-Nanoclay. DMA (Dynamic Mechanical Analysis) test on the obtained Rubber composites showed enhancement in Mechanical and Thermal properties.**

Index Terms—**Carbon Nanotubes, MWCNT's, Raw Rubber, Nanoclay.**

I. INTRODUCTION

Nanocomposites technology is a new era in polymeric materials. While the science of filling thermoplastics and thermosets in order to increase stiffness has been practiced for many years, the need to add large amounts of filler would often increase the stiffness of the thermoplastic while decreasing other desirable properties, particularly impact properties. Most products desire a balance between stiffness and impact, and this need has been met using layered silicate structures at loadings of 10% or less. These nanosilicates or nanoclays enhance mechanical, thermal, dimensional and barrier properties.

The most commonly used clay in the synthesis of polymer nanocomposites is montmorillonite (MMT) which is the major constituent of bentonite. It is well known that filler anisotropy, i.e. large length to diameter ratio (aspect ratio), is especially favorable in matrix reinforcement. Due to unique structure of montmorillonite, the mineral platelet thickness is only one nanometer, although its dimensions in length and width can be measured in hundreds of nanometers, with a majority of platelets in 200–400 nm range after purification. Due to very small size and thickness of the platelets, a single gram of clay contains over a million individual platelets. There is substantial improvement in mechanical and physical properties of nanocomposites and this too at a very low silicate content (3–6 wt %).

Polymer nanocomposites reinforced by relatively small amounts of ultra-fine, nano-particles (most often clay platelets) proved exceptionally promising engineering materials with unexpectedly high stiffness/toughness ratio, gas barrier properties, flame retardence, etc. The real interest in nanotechnology is to create revolutionary properties and functions by tailoring materials and designing devices on the nanometer scale. Natural rubber nanocomposites are proven materials for their high air impermeability, excellent solvent resistance, high modulus etc. The form of rubber *viz.* latex or dry rubber, mainly decides the nature of the clay to be selected for composite preparation. For dry rubber compounding, the clay should be organophilic whereas a hydrophilic layered silicate is preferred for latex addition.

In the first category, the nature of the modifiers of the Nanoclay, the layer distance and the polarity of the polymer, play a crucial role in clay exfoliation and hence the composite properties. In the case of latex, the hydration efficiency of the silicate, aspect ratio of the clay platelets and the polarity of the polymer decide the composite properties. In certain cases the intercalation process can be accelerated by the addition of polar polymers. The factors to be considered in the selection of clay for natural rubber compounding, the effect of modifiers in the vulcanization reaction, characterization of the NR nanocomposites by TEM will be discussed in the proposed talk.

II. EXPERIMENTAL SETUP

A. *Materials:*

The materials used are Raw Rubber product name: ISNR-20) manufactured by Indian Rubber Composites Limited, Kottayam- Halloysite (Aluminosilicate nanotube clay, model No.:PK805), obtained from Pai Kong Nanotechnology Co. Multi walled Carbon Nanotube (MWCNT's) was purchased from ACROS (Somerville, NJ).

B. *Preparation of Rubber-Nanoclay Composite:*

The 4.2 g n-hexadecyltrimethyl-ammonium bromide (intercalative reagent) was dissolved in 250 ml distilled water under a vigorous stirring condition to form a uniformly dispersed solution. Then, 5 g [Al sup.]-

Halloysite was added to the solution and stirred for 24 h, filtered, then washed three times with 400 ml of hot water to remove AlO3. After washing with ethanol (250 ml) to remove residual ammonium salt, the modified clay was dried in a vacuum oven at 80˚C for 24 h.

Nanoclay material Halloysite (Aluminosilicate nanotube) thus purified is mixed with a sample of Raw Natural rubber (ISNR-20) in the proportion 1:10 using Toluene as the solvent[1]. The above composition is mixed using Mechanical Stirrer at an elevated temperature for a curing period of 3-4 hours until Rubber-Nanoclay composite is achieved.

C. Preparation of Specimen:

The carbon nanotubes were added to Rubber-Nanoclay Composite as filler. The preparation of the nanocomposites was carried out by using a solvent casting method using toluene as a solvent. The added amounts of the carbon nanotubes were 1, 3, 5, 7 and 10 wt % of 10 grams of the total weight.

The process of making natural rubber/nanotubes as nanompcosite material divided into the four following processes.

Dispersion of Nanotubes:

This phase involves the dissolution/dispersion of CNTs in a solvent (in this case, toluene) in order to disentangle the nanotubes that typically tend to cling together and form lumps, which become very difficult to process. For this, a certain quantity of carbon nanotubes or nanofibers was added to a specific amount of toluene solution after carefully weighing (in order to maintain a specific weight ratio of nanotubes in the solution). This solution was further sonicated using a mechanical probe sonicator (Branson sonifier), capable of vibrating at ultrasonic frequencies in order to induce an efficient dispersion of nanotubes or nanofibers. For this study, different CNT solutions were prepared (containing CNTs in various weight ratios):

1) 1 wt% CNTs containing in 10ml of toluene solution
2) 3 wt% CNTs in 10ml of toluene solution
3) 5 wt% of CNTs in 10ml of toluene solution
4) 7 wt% CNTs in 10ml of toluene solution
5) 10 wt % CNTs in 10ml of toluene solution[2].

Dissolution of the Rubber

This stage involves the dissolution of the Rubber-Nanoclay composite in a suitable organic solvent (toluene). A specific amount of rubber composite (in this case, 10 gms) weighed using a balance was added to a certain quantity of organic solvent (500 ml of toluene) thereby maintaining a desired rubber weight ratio. This mixture was stirred and kept for certain duration of time until the rubber became uniformly dissolved in the solvent.

Mixing of Rubber with Nanotube Solution

This is the final step in the melt preparation process and basically involves thorough mixing of the solutions prepared in the first and second stages, resulting in a solution that consists of a good blend of nanotubes in the rubber composite.

Pressing and Testing the Sample

The Rubber Nanoclay composite material (rubber with CNTs) was pressed using hot press and cut into standard shapes. The samples were then characterized and mechanical properties measured.

III. RESULTS AND DISCUSSION

A. Effect of CNTs on Mechnanical properties of Natural Rubber

The stress curve of different percentages of pure carbon nanotube (1, 3, 5, 7 and 10 wt % of CNT's) with ISNR-20 is presented. The tensile strength radically increases as the amount of CNT's concentration increases[4]. The general tendency is that the stress level is increased by the addition of CNT's which plays the role of reinforcement. From these results, it is deduced that the reinforcing effect of CNT's is very marked. As the CNT content in the rubber composites increases, the stress level gradually increases but at the same time the strain of the nanocomposites decreases[3].

The increased level of stress was due to the interaction between the CNTs and the rubber composite. A good interface between the CNT's and the rubber composite is very important for a material to withstand the stress. As described above CNT's are extremely strong materials compared to other types of fillers, thus making them good candidates as nanofillers. Under load, the matrix distributes the force to the CNT's which carry most of the applied load.

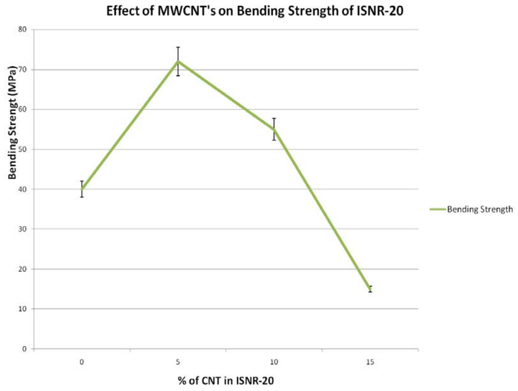


Figure 1. Bending stress of ISNR-20 with different percentage of CNTs.

B. Effect of CNTs on the Young's modulus of ISNR-20.

The same phenomenon was observed for Young's Modulus. The young's modulus of the composites normalized with that of the pure matrix is presented in figure 3. The result indicated that the Young's Modulus increased with an increase in the amount of the CNTs in the formulation. However, at 1 and 3 wt % of CNTs, the increment of the modulus is not as high as that of the tensile strength. The same value of the modulus and the tensile strength were observed at 5 wt % of CNTs. While at 7 and 10 wt % the modulus was higher than the tensile strength[5].

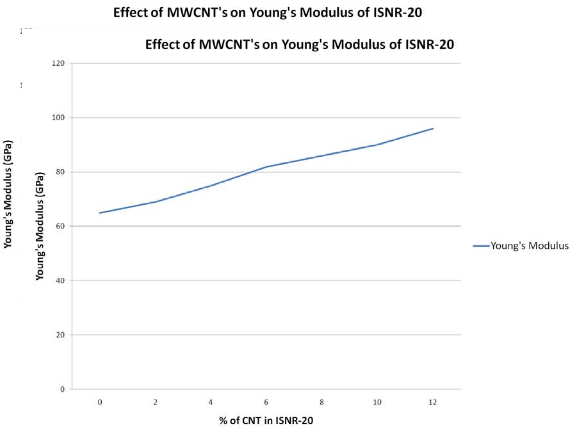


Figure 2. Young Modulus of ISNR-20 at different percentage of CNT's.

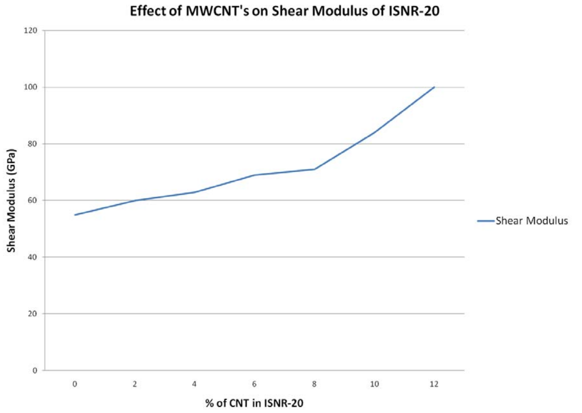
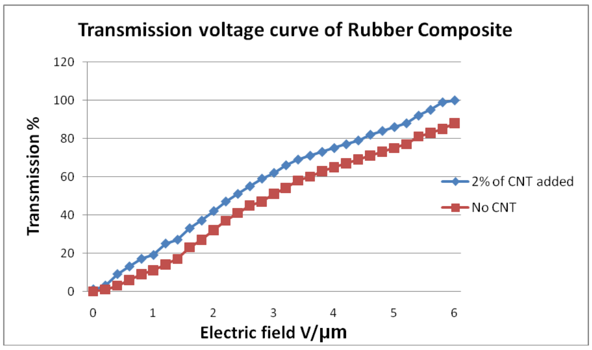


Figure 3. Shear Modulus of ISNR-20 at different percentage of CNT's.

C. Effect of CNTs on the Young's modulus of ISNR-20.

The composite is placed between the Electric plate and the current is placed. The transmissiblity capacity was analysed. The transmissiblity capacity was far superior at 2% of MWCNT content. In the experiment, not all films returned to the original scattering state after field removal. The rise time was inversely proportional to the applied voltage, while the decay time was voltage independent. Both the rise and decay times of the Rubber Composite with MWCNT's were faster than Composite without it.



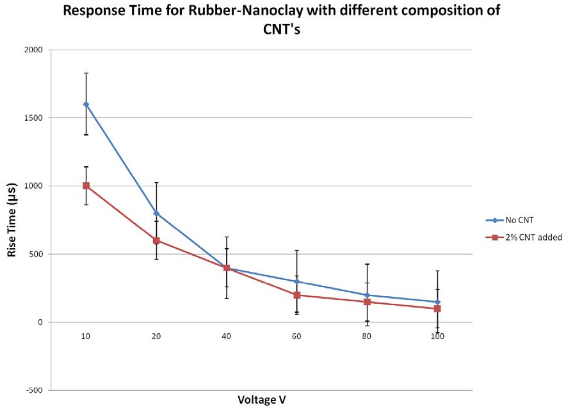


Figure 4. Dielectric properties of ISNR-20 at different percentage of CNT's.

IV. CONCLUSION

In summary, we have demonstrated the successful fabrication of Nanocomposite consisting of ISNR-20 Nanoclay composite with 1-10 wt % Multi walled carbon nanotubes (MWCNTs). Carbon nanotubes were applied for interface nano-reinforcement in advanced commercial carbon/rubber composite and this is the first attempt such a work is reported. The preparation of the nanocomposites was carried out by a solvent casting method using toluene as a solvent. It is clear from the figure that the maximum stress of pure ISNR-20 is 0.2839 MPa. When 1wt % of CNTs was added to the rubber the stress level for the Nanocomposite material increased from 0.2839 MPa to 0.56413 MPa. Addition of the wt % CNTs to the natural rubber increased the stress level gradually as shown in figure 2. At 10 wt % of CNT's the stress value obtained reached 2.55 MPa which is 9 times that of pure Natural rubber. The result indicates that, by increasing the amount of CNTs added into the rubber composite the ductility decreased and the material become stronger and tougher but at the same time more brittle. The clear trend observed here is that as nanotube load increases, the fiber breaking strain decreases. It also shows that the highest strain value was obtained for the Nanocomposite at 1wt % of the CNTs. This composite at this percentage is more ductile and more elastic compared to other percentages of CNTs. The strain value at 1wt % was almost the same as for pure rubber. Minimum strain value was obtained at 10 wt % of CNTs; the strain value decreased almost 2.5 times i.e. 2.94 compared to pure rubber which was 7.34.

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