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Study on Improvement in Tribological and Morphological of f-CNT's with Polymer Composites

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Abstract: In the present paper functionalization and the surface modification of CNT's were examined. CNT's was primarily treated with acid mixtures $\text{HNO}_3/\text{H}_2\text{SO}_4$, to introduce the silane group onto the surface of CNT's and remaining treatments. These groups was used as reaction precursor in the functionalization, were successfully covalently bonded to CNT's. The functionalized CNT's were characterized by (FTIR). Scanning electron microscope (SEM) shows the dispersion of CNT's on the surface of polymers. Dynamic mechanical analysis (DMA) demonstrates both the storage module (E) and temperature (T_g) of the composites. The study shows that dispersion of the carbon nanotubes (CNT's) in Polymer composites the carbon nanotubes are to be functionalized by using acid treatment, silanization, thionilation and amine treatment, Effect of these treatments "enhance the mechanical and tribological properties of polymer composites" by molecular dynamic analysis and motivate for future scope in this direction .

Keywords: Polymers, functionalized carbon nanotubes, morphological, DMA, tribological.

I. INTRODUCTION

Hence the invention of carbon nanotubes (CNT's) by Iijima [1]. It has been turned into a solid possibility for a large scope of utilizations, for example, petrochemical, marine innovation and gas stockpiling. CNTs have novel properties including exceptional firmness and quality. Past inquires about have demonstrated that precisely carbon nanotubes are multiple times more grounded than steel however their thickness is multiple times lower [2]. Broad research has been finished by joining diverse sorts of CNTs into polymer materials to present innovative composites that have high mechanical quality, electrical conductivity and warm. Great interfacial attachment and great scattering among CNTs and the polymer lattice is fundamental to improve the mechanical properties of CNT polymer nano composites [3]. On account of their tendency, CNTs are fundamentally insoluble in most natural and watery solvents just as awful synthetically good with the polymer grid. Therefore, surface improvement of CNTs so as to improve the dissolvability and similarity is one of the fundamental research point in the most recent years. Covalent or non-covalent functionalization of CNTs can upgrade their similarity and solvency, Hong CY[4]. The non-covalent functionalization of CNTs incorporates non covalent covering with surfactants [5]. The principle favorable position of non-covalent functionalization that is the structure and unique characteristics of CNTs are not changed after alteration. Nonetheless, the surfactants, polymer chains and electric acceptor which can be utilized for this strategy are extremely constrained, their scattering is above all and not entirely steady, it is hard to additionally surface alter CNTs with various functionalities.

Polymer-based materials are generally utilized in different parts of designing and logical applications, for example, farming science, modern generation, military, car industry, astronautics, air transportation and day by day life attributable to their astounding thermodynamic and mechanical properties, light quality, low thickness, wear and erosion obstruction and cost, et al. In down to earth and modern applications, polymer-based materials are exposed to various sorts of burden factors, for example, grating wear, consumption, ultra high and low temperature and weight et al. prompting diverse disappointments of polymer lattices [6-8]. Since, so as to escape monetary misfortunes and losses, it is of incredible upgrade to investigate components of disappointments of polymer materials and concentrate how to proficiently improve their tribological and mechanical properties. Over most recent couple of years, broad investigations has been dedicated to finding on the upgrade of tribological and mechanical properties of polymer materials by consolidations of extra fillers, for example, carbon black[9], silica[10], fibers[11] and adjusted ones[12, 13]. Subsequently, prevalent elements of polymer networks fortified by these customary fillers are seriously debilitated and constrained to be broadly connected.

In this examination work the surface alteration and functionalization of CNTs with some natural gatherings, for example, silanization, thionilation and amine treatment was researched. The portrayal of the alteration procedure was described by FT-IR spectroscopy, morphological, DMA and tribological properties of the composites.

II. EXPERIMENTAL PROCEDURE

A. Materials

Acetal (secured from Korea of evaluation K700) is utilized in this examination work of thermoplastic kind polymer. Multi-walled carbon nanotubes (secured from SKYSPRING NANOMATERIALS, USA) are utilized in this investigation work. Compound reagents utilized are Hydrochloric acid (HCl), Sulphuric acid, nitric acid, ethanol and distilled water (procured from LOTUS ENTERPRISES), Thionyl Chloride, Acetone (from COASTAL ENTERPRISES).

First weight the crude MWCNT's to be calcinated so as to evacuate the impurities that are available in it. Taking the crude MWCNTs for calcination treatment at 545°C temperature for 45 minutes. The treated MWCNTs were treated with concentrated hydrochloric corrosive, which is a built up strategy for the evacuation of contaminations. 10gms of MWCNT was set in a round base cup with 500 ml of HCl and 500ml of refined water was included. The blend was mixed utilizing attractive stirrer for 5 hours. After that contaminations are going to drift on the highest point of arrangement. Those are expelled by getting the pH estimation of 6 through pH papers.

B. Preperation of Oxidizing MWCNT's

After the filtration by Hydrochloric acid (hcl), the arrangement is oxidized by refluxing in 1200 ml of a 3:1 blend of 300 ml sulfuric acid, 116 ml nitric acid and 784 ml of refined water in a round bottomed jar with a condenser for 8 hours at 110°C and after that separating with refined water to wash away any acidic buildup from the nanotubes' surfaces. After that contaminations are going to coast on the highest point of arrangement. Those are expelled by getting the pH estimation of 6 through pH papers. The water wash ought to be accomplished for no less than 3 or multiple times with the goal that the acidic buildup transforms into essential. This outcomes that the arrangement transforms into impartial which demonstrates the arrangement is free from a wide range of impurities. After, this treated MWCNTs are separated with filtration set up with a channel paper of 0.22μ and afterward this sanitized MWCNTS. After that acid arrangement is washed with refined water to expel acids from MWCNTs until to accomplish pH estimation of 6. After filtration of MWCNTs all are gathered in a crucible to evacuate dampness content in MWCNTs and crucible is put in a vacuum for 8 hrs. After that MWCNTs resemble strong precious stones and this are fueled to fine particles. This example is given to FT-IR examination to check the bonds arrangement on MWCNTs. After filtration CNTs gathered in a crucible and put in vacuum to evacuate dampness after that it is powdered and an example is given to FT-IR to check oxidation.

C. Preparation of functionalized MWCNTS

In this procedure 1.8gms of MWCNTs are scattered in 2% of silane arrangement which implies 95% of ethanol and 5% of refined water. After that arrangement was mixed for 4 hours with 70°C temperature after that arrangement is washed with refined water to achieve Ph estimation of 6 to transform the acidic arrangement into neutral. After filtration of MWCNTs all are gathered in a crucible cup to expel dampness content in MWCNTs and cup is put in a vacuum for 8 hrs. After that MWCNTs resemble strong gems and this are fueled to fine particles and sent to FT-IR Examination. At long last when contrasted with the oxidation of MWCNT's, the silanization have the better qualities when looked at in the FT-IR examination.

So also similarly the functionalization procedure is conveyed for thionalation and amine treatment moreover. In which the oxidized cnts are taken at that point prepared further with different chemicals depending upon the treatment type. In thionalation process the oxidized cnts are treated with thionyl chloride and refluxed for 24hrs. It is included as 20ml per 1gm of cnts. The remainder of filtration and drying process is as referenced for past procedures.

Amine treatment starts with taking thionalated cnts from above procedure as information. They are treated with EDA (Ethylene diamine) and re-fluxed for 24hrs. Then the remainder of procedure is same as above, at long last given for FTIR examination for checking bond arrangement in the material.

III. RESULTS AND DISCUSSION

A. FT-IR Analysis

Fig .1 demonstrates the FT-IR represents, various functionalization's on MWCNT have been described through FTIR spectra as appeared in the above figure. Fig .(b) demonstrates crests at 3400cm^{-1} (Extending frequency of $-\text{OH}$) and $1380 - 1740\text{cm}^{-1}$ district which can be ascribed to $\text{C}=\text{O}$ present because of oxidation by acid treatment.

When we treated SOCl_2 (thionyl chloride) with acid treated CNT we watched an extra unmistakable crest at 610cm^{-1} which can be credited to $\text{C}-\text{Cl}$ bond vibration which demonstrating the arrangement of $\text{OH}-\text{C}-\text{Cl}$ moiety. Figure (d) is FTIR spectra for the example treated with EDA after thionilated CNT fig(c).

The top at 2838 and 2923cm⁻¹ because of extending frequency of – CH aggregate present in EDA. A little mound at 1035cm⁻¹ and 3450cm⁻¹ be credited to – NH bond which emerges in view of EDA. Figure (e) is the FTIR spectra of test treated with APTS after acid treatment of MWCNT. The band at 1021cm⁻¹ can be ascribed to Si-O-Si vibration relate to siloxane units framed amid silanization process.

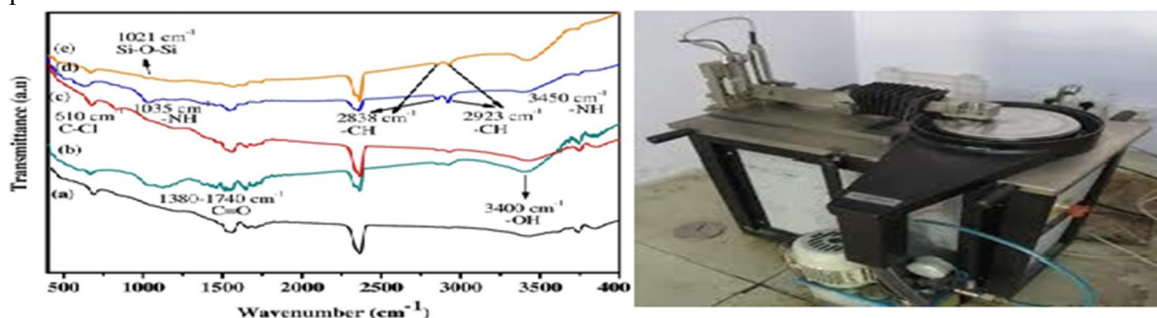


Fig .1 Comparison on functionalization's Fig .2 Wear testing of specimen

Fig .2 shows the pin on disc experiment of measuring friction and sliding wear in pin on disc method. The method tester measures the friction and sliding wear properties of dry or Lubricated surfaces of a variety of bulk materials. The normal load, rotational speed, and the wear track diameter are all is set by the user prior to the pin on disc test. Pin on disc tester consists of a rotating disc of the material to be tested against a stationary sphere, usually made of cemented carbide, referred to as the pin. Although the pin surface can also be wear tested. The pin on disc test method has proved particularly very useful in providing a simple wear on machine components, such as the valve train, particularly in motor sports applications.

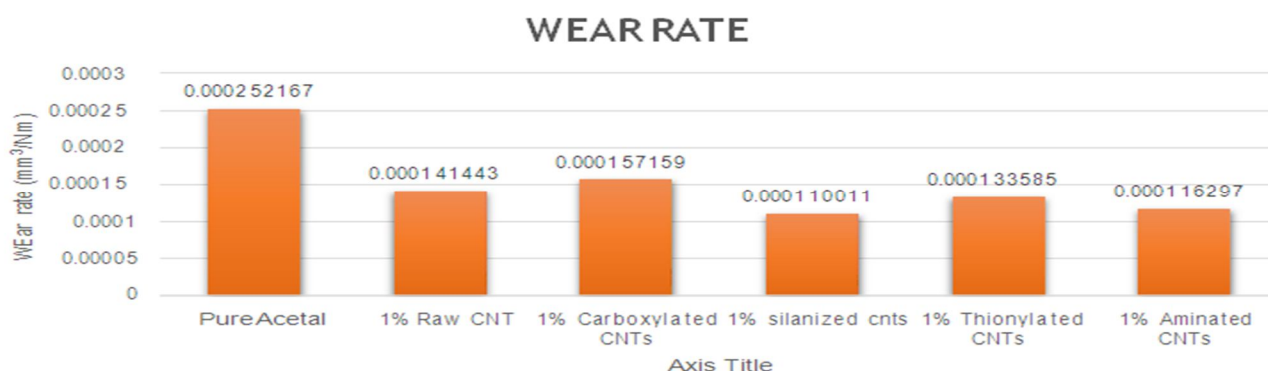


Fig .3 Comparison of Wear rate

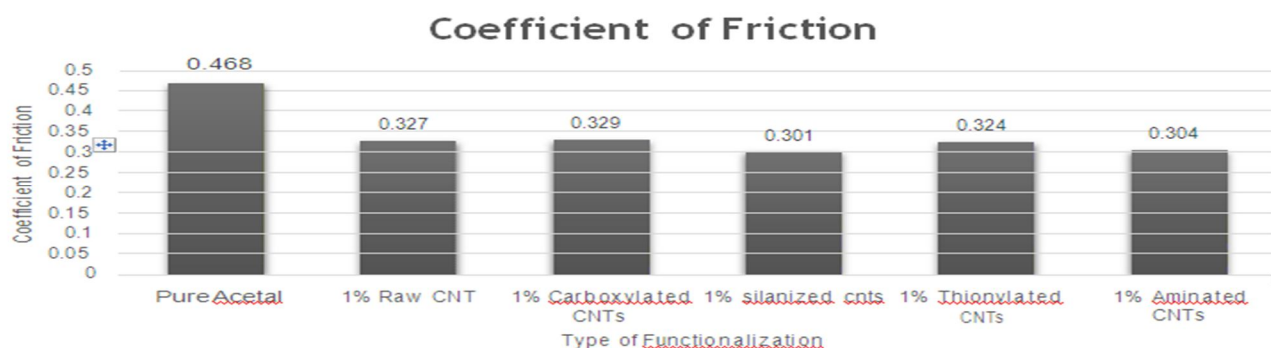


Fig .4 Comparisons in Coefficient of friction

Fig .4 & 5 shows the results from pin on disc testing which was done to study the wear behaviour of the polymer composite materials showed that the wear was less in material acetal with 1% CNT treated with APTS i.e.(Silanization treatment) when compared to other functionalization's. Hence it can be effectively concluded that the usage of polymer material and the addition of functionalized CNT reinforcement (CNT) did considerably improve the polymer composite strength and performance.

B. Wear Track

From the following Fig .5 & 6 wear track images taken from the optical microscope it is clear that tracks are different for different composites. We can clearly say that pure acetal has got more wear tracks when compared to others, i.e. functionalized CNT composites. From our observations it is clear that Silane functionalization has shown much better results than the rest. As we can barely see any wear tracks which indicates that by doing functionalization the wear rate of composite is reduced. Similar such decrease in wear rate can be observed because as there is reduction in wear tracks. Next to Silane are Amine and Thionyl functionalization's which have reduced wear tracks compared to the pure acetal.

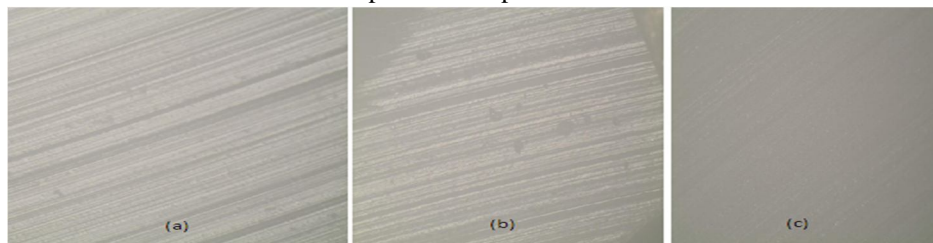


Fig .5 (a) pure acetal(b) Raw CNT composite (c) Silane treated composite

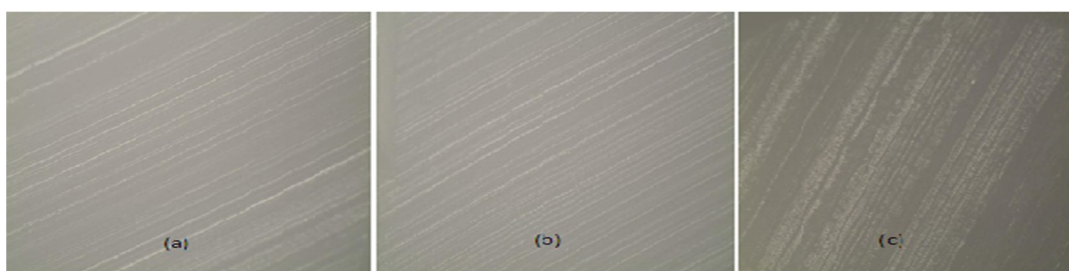


Fig .6 (a) Amine treated (b) Thionyl treated (c) Acid treated composite

C. Morphological study of Polymer/CNFs composites

Fig .7 SEM examination of the fracture surfaces of the pure acetal, acetal with 1% wt fraction of CNT composites which are functionalized with different types of chemicals was performed to investigate the fracture mechanism of the composites. Figure shows SEM images of the fracture surfaces of the four of five composites.

In case of pure acetal fractured surfaces are smooth where as in the case CNT reinforced acetal fractured surfaces were found to be rough. In case of 1% CNT reinforced acetal CNTs were sparsely found. In case of Silane treated and Amine treated CNTs, CNTs were distributed uniformly, which might be resulted in improvement of mechanical properties. In case of raw CNT's CNT pullouts have been observed. These pullouts will act as stress concentrators and might resulted in decrease of mechanical properties at CNTs.

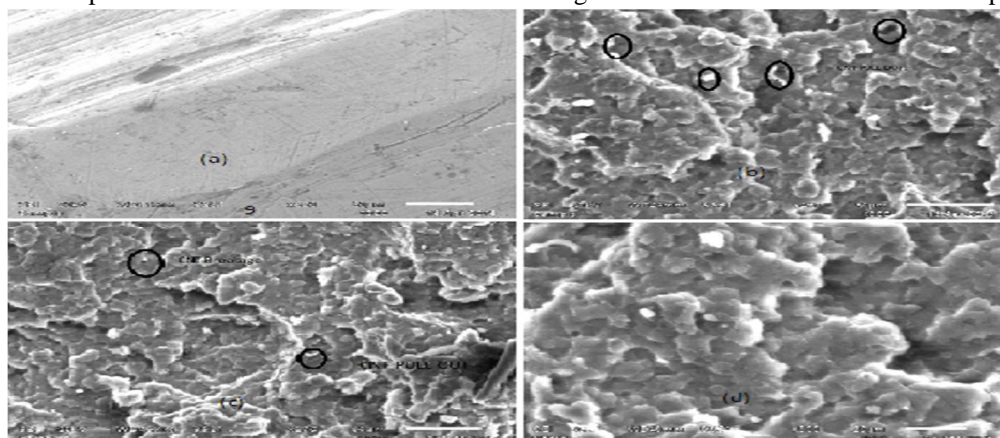


Fig .7 SEM images of fracture surfaces (a) Pure acetal (b) Raw CNT (c) Amine treated CNT (d)Silane Treated CNT

In case of raw cnts, more CNT pull outs are observed. In case of silane and amine functionalization no of pull outs are less and cnt breakage is observed, which might have lead to enhancement of properties.

D. Dynamic mechanical analysis (DMA)

Fig .8 Demonstrates the most fundamental part of the DMA, an oscillator for applying cyclic powers to the test example? The example is mounted on a stage which is encased in the heater furnace. A few example stages are accessible for different testing modes. For example, the Perkin Elmer DMA 7 utilized in this examination is furnished with a three-point bowing stage just as a stage for performing cantilever type tests. By utilizing hacksaw cutting edge test of size 40mm×12mm×3.0mm, with is readied.

In a commonplace DMA damping test, a sinusoidal information drive from the oscillator twists the test example. The stage slack of the sinusoidal strain reaction is determined utilizing fourier investigation. Damping results processed by a PC program is introduced as $\tan\delta$ as a component of temperature or recurrence. Operational frequencies of DMAs are more often than not somewhere in the range of 0.001Hz and 100Hz. DMAs can go as high as 1000⁰ C, just as work at below zero temperatures (down to – 170⁰ C) when an appropriate cooling liquid, for example, fluid nitrogen is utilized.



Fig .8 DMA Setup

From the graphs Fig .9&10 plotted below we say that thee storage modulus of all the composite's decreases with the increase in temperature. Initially within the composites comparison we can say that there is increase in values of storage modulus as functionalized CNT's are induced into the material. Clearly we can say that silane treatment has better offerings when compared to pure acetal. Next to it Amine and Thionylation treatment has shown better results. Similary from the Tan delta graph plotted above we can see that the values of Functionalized CNT treated material has decreased in comparison to the pure acetal. Which indicates that CNT addition to material has increased in stiffness of material so the corresponding tan delta values i.e. damping nature is decreased in the material.

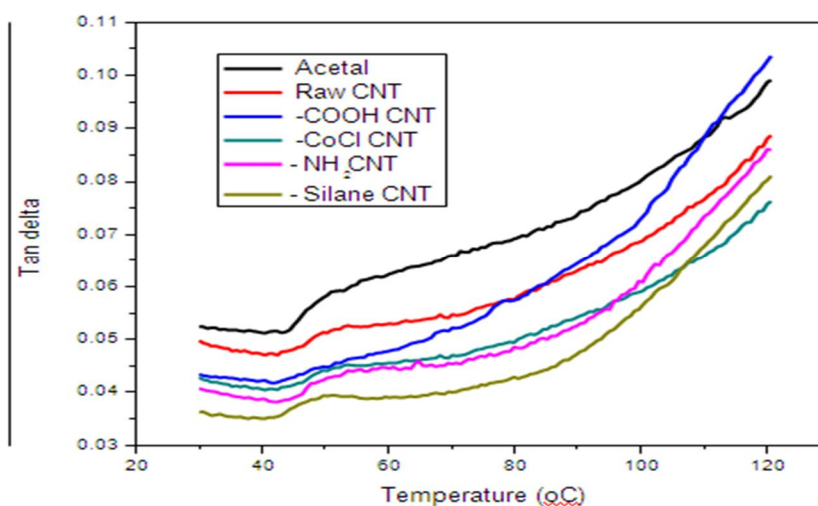


Fig .9 comparison of tan delta

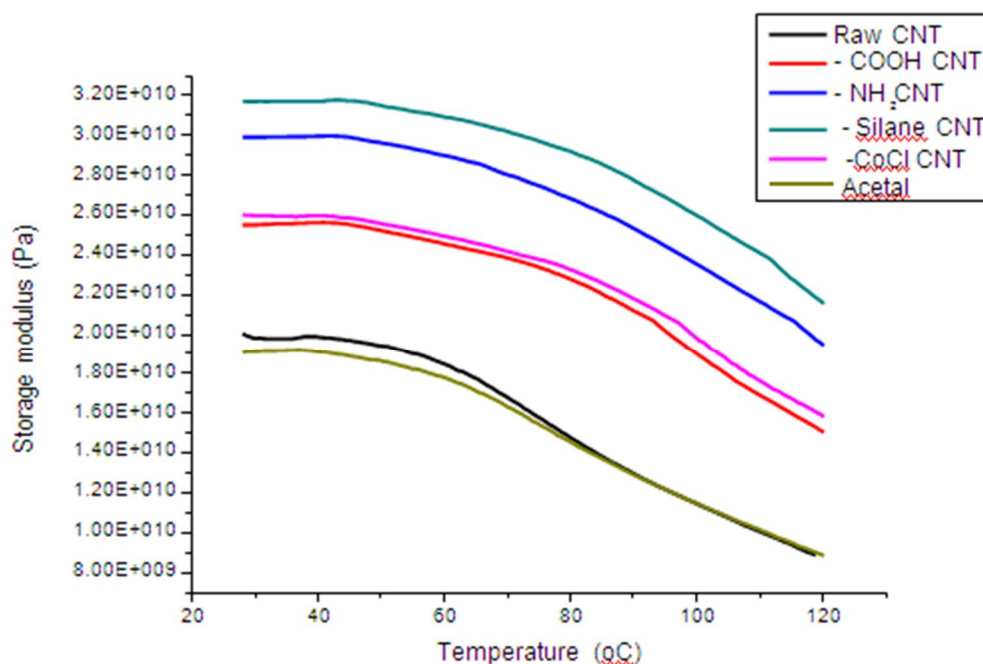


Fig .10 Comparison of Storage modulus

IV. CONCLUSION

The impacts of surface change of CNTs, the information got subsequent to advancing the yield parameters by watching the functionalization's of CNT's at 1% weight part, we reach these following resolutions. There by dispersion of functionalized CNTs in polymer gives predominant mechanical properties. The Silane treated MWCNTs dispersed in a composite improving the Tribology properties and furthermore increment damping proportion which isn't as much shown by different composites. However, Amine and Thionyl treated composites remain alongside it with impressive contrast. Morphological investigations have uncovered that silane-functionalized MWCNTs and other functionalization's advances great bond among CNT and polymer matrix by changing the matrix properties and henceforth, the properties of composite increments. It is seen that the silane treated MWCNTs blended in the composites prompts increment of the mechanical properties just as damping proportion. Functionalization by Silane treatment and other functionalization's are to accomplish the required bond on the MWCNTs. Likewise as the hardness of the material increment with the relative blending or sonicating the CNT's to the composites.

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