DYNAMIC MECHANICAL ANALYSIS OF CARBON NANOTUBE REINFORCED EPOXY COMPOSITES FOR NANO HYBRID STRUCTURES

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Abstract: In this study, laminates of basalt/ glass fiber are fabricated by varying percentages of carbon Nano Tubes (CNTs) which are reinforced with epoxy and investigated mechanical properties and Dynamic mechanical behavior of polymer Nano composites reinforced with various weight percentages of CNT Nano particles. The Nano composite plates were prepared using cold compression moulding technique. Finally, the prepared composite plates were sized in laser cutting according to ASTM standards and subjected to tensile test and DMA test. The mechanical properties such as young's modulus, ultimate tensile strength, % of elongation of material are studied. A series of dynamic mechanical tests were performed for prepared composites over a range of testing temperatures. Test frequency was kept constant. The dynamic mechanical properties of prepared Nano composites were studied by recording storage modulus, loss modulus, tan δ and glass transition temperature (Tg). The graphs like storage modulus vs temperature, loss modulus vs temperature, stiffness vs temperature, tan delta vs temperature, and stress vs temperature are plotted. The loss modulus (E") and damping peaks (Tan δ) for 0.6% CNTs nanocomposite is larger than those of the epoxy. Hence the Experimental results revealed the significant improvement of mechanical properties by filling the carbon nanotubes into epoxy.

Keywords: Dynamic mechanical analysis (DMA), carbon nanotube (CNT), Nanocomposite, Stress Modulus, Loss Modulus, tan δ.

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1.INTRODUCTION

Carbon nanotubes (CNTs) show uncommon properties, they are high warm conductivity and electrical conductivity, low weight to surface region proportion, high mechanical strength and high three layered (3D) adaptability [1-3].
Basalt fabric, glass fiber, mixture of resin, CNTs and hardener (based on required weight %) are used in the fabrication of basalt glass fiber reinforced by CNT. Hand layup technique and hydraulic press is used in fabricating the laminates. The resin mixture is prepared by using magnetic stirrer equipment and the components of mixture are resin and hardener these are mixed in the 10:1 proportion. Dynamic mechanical analysis (DMA) is performed on the specimens. In DMA analysis sinusoidal stress is applied on the specimen and the strain of the specimen is then noted and thus complex modulus can be determined. By varying the temperature or frequency of stress applied on the specimen the variations in the complex modulus are noted and glass transition temperature is determined. It is observed that the Composites which have low concentration of CNTs exhibit improved mechanical properties than the basalt glass fiber without any reinforced materials added. Since CNTs can exhibit electrical properties like high electrical conductivity. The other applications where CNTs reinforced composites are used in [3-7] photovoltaic cells, sensor applications and light emitting diodes (LEDs). [4-9]In electronic applications Conductive and adhesive composite materials are used as a bonding material. [2] Furthermore, In dynamic applications like automobile parts, structural and industrial applications these nanocomposites are used. [10–12] Polymers are low costs and widely available these are used as host material because of adhesive properties. Because of good casting and easy in preparation, EPOXY L-12 can be used in many forms. [5]
Dynamic mechanical analysis (DMA), thermo-mechanical analysis (TMA), differential scanning calorimetry(DSC), dielectric analysis(DEA) are some the techniques which are used to determine thermos-mechanical properties of nano composites. [14, 15] DMA technique is the best suitable for determining the properties of nano composites which are effective for their evaluation. Dynamic mechanical analysis is a technique which is used to determine the elastic and viscous response of the nanocomposites against temperature, time or frequency of stress applied on specimen. The dynamic parameters such as G' denote the elastic part of the sample and thus describes the ability of the sample to bear a load. The loss modulus (G") is equivalent to the dissipation energy and thus significant impact on the viscous behavior of the samples. And damping capability of the sample is denoted by the loss factor tan delta. Further, this dynamic analysis also indicated that G' & G" increases with filler content. [13] Concerning temperature or recurrence, Dynamic mechanical examination decides the capacity modulus (measure of put away energy), misfortune modulus (measure of intensity scattered as intensity), firmness, glass change temperature, and misfortune

factor (tan δ). In many designing applications the unique burden bearing limits of the materials is fundamental component of many designing applications that as to be examined. Tg is the typical temperature between the lustrous and rubbery stage temperatures of the polymer or composite, when the temperature of composite is continuously expanded then their happens a stage change from polished stage to a fluid or a rubbery stage progressively and as of now glass progress temperature exists.[16] While utilizing the DMA gear, Glass change temperature happens when the misfortune factor accomplishes its most extreme worth. In this trial and error, four different weight % of CNT supported basalt glass fiber are manufactured. 0.2%, 0.4%, 0.6%, and 0.8% are the weight rates of CNTs which are built up in the basalt glass fiber. Hand rest up method and water driven press is utilized in the creation of the Nano composites. As fluctuating the weight % of CNTs in epoxy the mechanical properties like capacity modulus E, misfortune modulus E, Solidifies, Stress and tan δ (damping coefficient) of the still up in the air. In this way the previously mentioned mechanical properties of nanocomposite overlays with various weight rates of CNTs/Slick EXPOY in the L-12 combination are seen by utilizing dynamic mechanical analyzer. The composites are developed with multiple Nano particle reinforced epoxy and perform a comparative analysis of the mechanical properties[16–18].

2. EXPERIMENTAL INVESTIGATIONS

2.1 MATERIALS

The raw materials which are used in the fabrication process are collected from the regional Hyderabad dealers. L-12 epoxy resin which is brought from the market distributors in Hyderabad, India. Density of the L-12 epoxy resin is in between 1.16 and 1.20 g/cm3 and a density of between 10,000 and 12,000 at 25C (cps). K-6 hardener is used in the fabrication of the laminates. Plain basalt fabric of areal density 367 g/m2 is used and the thickness is 0.37 mm and the width is 100 mm. Plain fiber glass fabric with thickness 0.24 mm and width is of 40 inches.

2.2 NANO FILLERS

CNTs Region is 50 + 15 m2, 80 in antae crystal and 200 crystals, with an average 30 nm particle size.

Table 1: properties of CNT

PROPERTIES	VALUE	UNIT
C-PURITY	>95	%
Outer mean diameter	~13	nm
Inner mean diameter	~4	nm
Length	>1	μm
Bulk density	45-95	Kg/m
Elastic modulus	3596	Mpa
Tension at break	72.9	Mpa
Elongation at break	10.3	%
Izod-Impact	103	J/m
PROPERTIES	VALUE	UNIT

2.3 FABRICATION

Basalt fabric, glass fabric, resin, CNTs and hardener are the materials used in the fabrication process of the basalt glass fiber reinforced by CNTs. The first step of the fabrication process is preparing the resin mixture. In preparing resin mixture resin and hardener are mixed in the ratio of 10:1 and the CNTs are mixed as per the required weight percentage. Initially, apply the grease on the lower part of the mold and place the transparent sheet on it. Apply gentle pressure on the transparent sheet to remove air bubbles. Epoxy resin is mixed with hardener in the ratio of 10:1 in a glass jar. Pour the matrix material on the plastic sheet and spread evenly. Place the basalt fabric on the resin and pour some more, so that fiber gets soaked in resin and add the Nano CNT fillets accordingly. Follow the same for the upper mold also, leave the mold for 24 hours for curing.

Fig 1: Fabrication process Step1

Fig 2: Fabrication process Step 2

Fig 3: Fabrication process Step 3

Fig 4: Fabrication process Step 4

Fig 5: fabrication process step 5

2.4 MIXING OF CNT EPOXY

The fluid epoxy sap of 3.42 grams is taken in a little breaker and the measuring glass is set in a stove for 30 minutes at 60°C temperature. Then, at that point, the necessary amount of CNTs (0.2, 0.4, 0.6 and 0.8 weight %) are added into the fluid epoxy blend. Then tenderly mix the answer for 10 minutes. Then, at a temperature of 50°C and for 3 hours the blend is set in a ultrasonic shower. This is to get great dis-collection of CNTs and great scattering in the gum combination. Then for 3 hours and at a tension of 20 mm of Hg the combination of pitch and CNTs is set in a vacuum chamber. Then, hardener of 2.28 grams is added to the epoxy and CNT blend, and the epoxy combination is mixed for 10 minutes. Yet again for 30 minutes at a tension of 20 mm of Hg the gum blend is set in the vacuum chamber. Any air bubbles which are caught during blending process are eliminated. As the substance of CNTs increments above 0.8wt%, the consistency property of the epoxy arrangement gets expanded and prompts the disappointment of the scattering medium. Subsequently, we are utilizing the overlays of CNTs which are under 0.8 wt%. Basalt glass fiber covers with no support are additionally ready for examination.

2.5 CHARACTERIZATION

In view of ASTM D3039 the malleable test is finished on the basalt glass fiber built up by CNTs. The standard components of the example in light of ASTM D3039 are 250 mm (length) × 15 mm (width) × 3 mm (thickness). The state of the example is rectangular. INSTRON 3382 General Testing Machine of 100 kN load cell (Instron, Norwood, Mama, USA) is utilized in this tractable trial of the examples. The testing strategy for FRP composites, where a clasp on extensometer with a 25mm check length was connected to the example to gauge its prolongation during testing. The crosshead speed was set at 2mm/min, and the information was recorded and dissected utilizing PC programming. Five examples were tried for each FRP composite framework.

Dynamic mechanical investigation (DMA) hardware is utilized to decide the capacity modulus E, misfortune modulus E, Solidness, Stress and damping coefficient tan δ as a component of temperature, recurrence or time. The capacity modulus is comparative with the versatile modulus and is the proportion of energy put away, while the misfortune modulus addresses the gooey properties of polymer composites and it is the proportion of the disseminated energy.

In this study, Dynamic Mechanical Analysis was done on a NETZSCH DMA 242E Artemis setup with dual cantilever mode. It is manufactured by NETZSCH-Gerätebau GmbH (95100 Selb, Germany). It has a controlled force range upto 24 N for measurements of stiff samples. The force resolution can be increased upto 8N. It has a static travel range of 20mm which allows for precise testing of specimens which experience change in their dimensions during DMA. The analyzer also has 30 different sample holders for optimal adjustment of measurement. The sample mass was in the range from 5-10mg. The rate of heating is 10^0C/min over 30 to 600^0C temperature range.

3. RESULTS AND DISCUSSIONS

3.1 TENSILE TEST

The results obtained when tensile test is done on composites are shown in the below table 6.1.From the above table 2, we can get to know the values of Ultimate tensile strength (UTS), Yield strength (YS) and %Elongation for the composites. Firstly, when we compare the values of the Ultimate tensile strength (UTS) of the composites then we can observe that 1.1T is having the highest ultimate tensile strength when compared to remaining experiments as shown in the below fig 6.

Table 2. Tensile test results

Composite	Experiment No.	UTS (N/mm^2)	YS (N/mm^2)	% Elongation
0.2% CNT	1.1T	394.9	376.5	20.7
	1.2T	192.1	167.0	12.2
	1.3T	325.3	284.6	17.7
0.4% CNT	2.1T	384.8	348.8	20.4
	2.2T	241.8	180.5	15.3
	2.3T	301.1	253.0	17.2
0.6% CNT	3.1T	324.9	300.4	19.8
	3.2T	288.8	253.0	17.8
	3.3T	241.1	204.1	20.5
0.8% CNT	4.1T	354.2	298.6	18.9
	4.2T	375.1	312.2	19.1
	4.3T	256.7	220.3	21.1

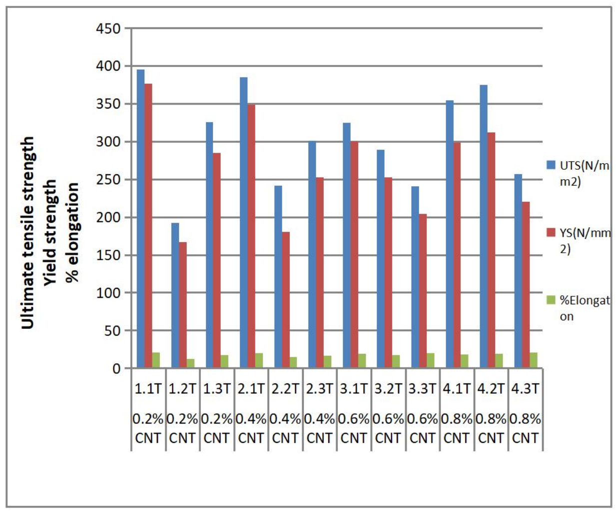

Fig 6: ultimate tensile strength, Yield strength, % elongation vs different wt % of CNT

The above bar chart is the combination of ultimate tensile strength, Yield strength, % elongation vs samples. 1.1T which is a sample of 0.2 % of CNT laminate have maximum ultimate tensile strength (UTS) of 394.9 N/mm2 and also maximum yield strength (YS) of 376.5 N/mm2. The least % elongation is showed by 1.2T sample of 0.2 % of CNT and the value is 12.2. 4.3T sample shows the highest % elongation that is 21.1 and it is of 0.8 % CNT laminate. For high strength applications 0.2 % CNT composite can be used and it shows less %elongation averagely.

3.2. DYNAMIC MECHANICAL PROPERTIES OF CNTS/NANOCOMPOSITES

CNT [CARBON NANO TUBE] –The paragraph describes Carbon Nano Tubes (CNTs), which are tiny carbon structures with diameters typically measured in nanometers. CNTs possess remarkable electrical conductivity, exceptional tensile strength, and thermal conductivity. Some CNTs act as semiconductors while others have high electrical conductivity.

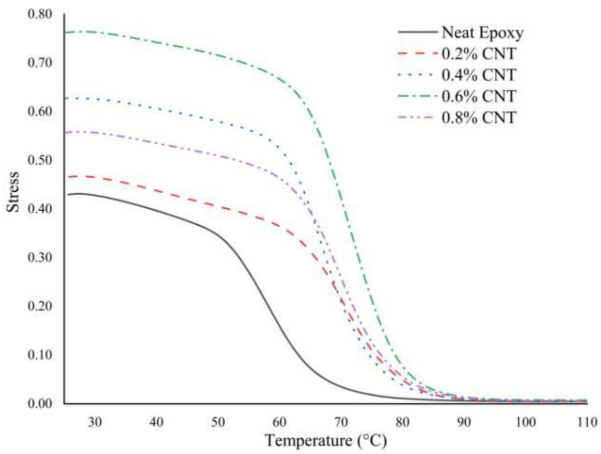

Fig 7. Stress vs Temperature

From the given graphical representation, we can gain information about the stress vs temperature in Carbon Nano Tube. The neat epoxy gradually decreases in CNT. 0.6% CNT is having highest stress than epoxy resin. Epoxy is very less compared with other percentages of graphene. The electrical properties of CNT epoxy composites are less than 1% [low CNT loadings]. It can be observed from the graph that 0.6% CNT can withstand maximum stress compared to 0.2%, 0.4% and 0.8%.

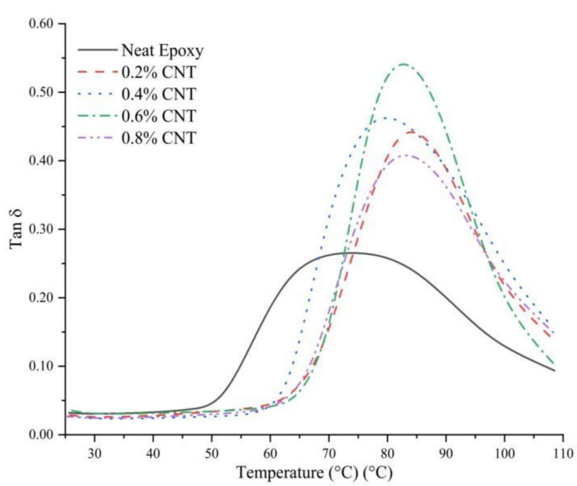

Fig 8. Tan Delta vs Temperature

This figure gives graphical information about Tan Delta vs Temperature in CNT. The neat epoxy resin gradually increases at 50- degree Celsius. There's a vast increase in 0.6 percentage of graphene. The graphical form of the tangent delta from the graph it can be drawn that 0.6% CNT shows the highest tan δ value which is in between 0.5 - 0.6.

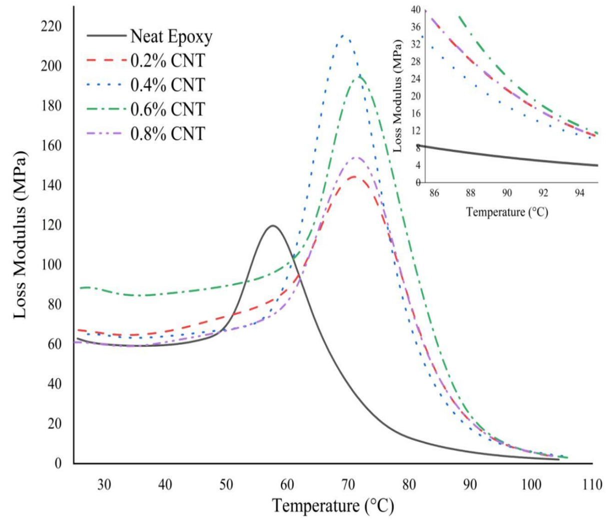

Fig 9. Loss Modulus vs Temperature

The figure gives statistics for loss modulus vs temperature for given percentages of CNT. Dynamic Mechanical Analysis [DMA] is an experimental method to measure the loss modulus. The loss modulus is used to determine the glass transition temperature (Tg). We can see the comparison of 0.2%, 0.4%, 0.6% and 0.8% of CNT with neat epoxy. 0.4% CNT is having highest loss modulus when compared with other weight percentages. Upon heating, loss modulus decreases because less force is required for deformation. Initially as temperature increases loss modulus also increases until maximum is reached then on further increase of temperature results in decreasing the loss modulus.

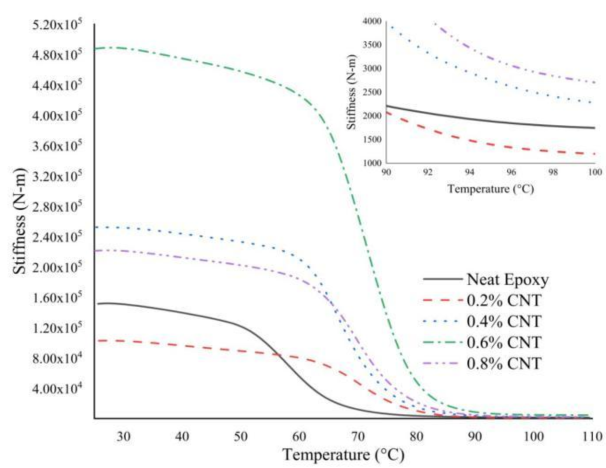

Fig 10. Stiffness vs Temperature

From the above graph, i.e. Stiffness vs Temperature, we get further information about CNT components. CNT's are stiffest materials in terms of tensile strength and elastic modulus. The maximum stiffness is 1.60*10^5 N-m for 0.2% of CNT. It is having the lowest stiffness. As temperature increases, the stiffness is decreasing. 0.6% of CNT is having highest stiffness of 4.80*10^5. It can be observed that up to certain temperature range the stiffness values are decreasing gradually on further increase of temperature leads to drastic decreasing of stiffness.

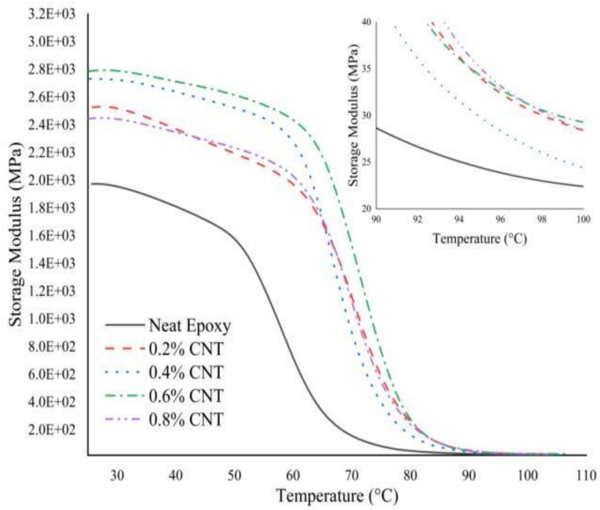

Fig 11. Storage Modulus vs Temperature

Above diagram of capacity modulus versus temperature bends of unadulterated epoxy and nanocomposite with various CNTs/Flawless epoxy proportions (0.2, 0.4, 0.6 and 0.8 mg wt. %) are gotten from DMA analyser at 1-Hz recurrence and is displayed in Fig. 11. It is seen that the capacity modulus relies upon both temperature and the level of CNTs in the epoxy blend. At any temperature, storage modulus values of CNTs reinforced basalt glass fiber laminates have greater values than the storage modulus value of the basalt glass fiber with any reinforcement. It explains that the stiffness of a nanocomposite increases when there is a decrease in epoxy content, resulting in higher E values as shown in Figure 11. Additionally, at temperatures up to 90°C, the storage modulus values of basalt glass fiber laminates increase with an increase in CNTs/NEAT EPOXY ratio up to 0.6 weight %.

The above graph gives data about the Storage modulus vs Temperature. Storage Modulus is the measure of energy stored by the material during its deformation. It represents the elastic behavior of the composite material. The paragraph states that as the temperature increases, the interfacial region between Carbon Nano Tubes (CNTs) and polymer matrix degrades, leading to an amplification of the storage modulus of the basalt glass fiber. 0.6% of CNT is having the highest storage modulus.

4. CONCLUSION

Basalt glass fiber laminates with different CNTs/NEAT EPOXY ratios (0.2, 0.4, 0.6 and 0.8 wt. %) are fabricated by using a high intensity ultrasonic liquid processor technique and by using hydraulic press. Epoxy resin mixture is prepared by using magnetic stirrer method. Thermo-Mechanical properties of the prepared basalt glass fiber laminates are observed by using DMA analyser. The mechanical properties of basalt glass fiber built up by CNTs overlays are estimated at various temperatures. The properties estimated incorporate storage modulus, loss modulus and glass transition temperature (Tg), and damping coefficient tan δ. The estimations were taken utilizing a Dynamic mechanical analyzer at a recurrence of 1 Hz. The power of the tan δ top for 0.6% CNT nanocomposites is bigger than those of the other weight rates of CNTs. The impact of expanding CNTs/Perfect EPOXY proportion on the mechanical properties of a nanocomposite material. The benefit of damping coefficient tan δ diminishes steadily as the CNTs/Perfect EPOXY proportion increments from 0.6%, while the Tg worth of the nanocomposite shifts towards a higher temperature range. Specifically, the Tg value of the nanocomposite increases from 80 ◦C to 90 ◦C as the CNTs ratio increases from 0 to 0.6 wt%. 0.6% CNT shows the highest storage modulus, stiffness, tan δ valve and stress. 0.4% CNT shows the highest loss modulus. Based on the above information it can be derived that the properties like storage modulus, stiffness, tan δ values and stress increases up to 0 - 0.6% CNTs with respect to temperature. For loss modulus increases up to 0.4% CNT then decreases as temperature increases. 0.6% CNT is best suitable for optimum thermomechanical applications.

REFERENCES

1) Amraei, Jafar, Jafar E. Jam, Behrouz Arab, and Roohollah D. Firouz Abadi. "Modeling the interphase region in carbon nanotube reinforced polymer nanocomposites." Polymer Composites 40, no. S2 (2019): E1219-E1234.

2) Ramana, G. Venkata, Balaji Padya, R. Naresh Kumar, K. V. P. Prabhakar, and P. K. Jain. "Mechanical properties of multi-walled carbon nanotubes reinforced polymer nanocomposites." (2010).

3) Li, Yuan, Yaolu Liu, Xianghe Peng, Cheng Yan, Sen Liu, and Ning Hu. "Pull-out simulations on interfacial properties of carbon nanotube-reinforced polymer nanocomposites." Computational Materials Science 50, no. 6 (2011): 1854-1860.

4) Allaoui, Aïssa, Shuo Bai, Hui-Ming Cheng, and J. B. Bai. "Mechanical and electrical properties of a MWNT/epoxy composite." Composites science and technology 62, no. 15 (2002): 1993-1998.

5) Biercuk, M. J., Mark C. Llaguno, M. Radosavljevic, J. K. Hyun, Alan T. Johnson, and John E. Fischer. "Carbon nanotube composites for thermal management." Applied physics letters 80, no. 15 (2002): 2767-2769.

6) Meng, H., G. X. Sui, P. F. Fang, and R. Yang. "Effects of acid-and diamine-modified MWNTs on the mechanical properties and crystallization behavior of polyamide 6." Polymer 49, no. 2 (2008): 610-620.

7) Zou, Wei, Zhong-jie Du, Yan-xin Liu, Xiaoping Yang, Hang-quan Li, and Chen Zhang. "Functionalization of MWNTs using polyacryloyl chloride and the properties of CNT–epoxy matrix nanocomposites." Composites Science and Technology 68, no. 15-16 (2008): 3259-3264.

8) Shi, Jia-Hua, Bing-Xing Yang, and Suat Hong Goh. "Covalent functionalization of multiwalled carbon nanotubes with poly (styrene-co-acrylonitrile) by reactive melt blending." European polymer journal 45, no. 4 (2009): 1002-1008.

9) Lee, Jea Uk, June Huh, Keon Hyeong Kim, Cheolmin Park, and Won Ho Jo. "Aqueous suspension of carbon nanotubes via non-covalent functionalization with oligothiophene-terminated poly (ethylene glycol)." Carbon 45, no. 5 (2007): 1051-1057.

10) Wang, Mian, K. P. Pramoda, and Suat Hong Goh. "Enhancement of interfacial adhesion and dynamic mechanical properties of poly (methyl methacrylate)/multiwalled carbon nanotube composites with amine-terminated poly (ethylene oxide)." Carbon 44, no. 4 (2006): 613-617.

11) Park, Sungjin, Jung Oh Huh, Nam Gwang Kim, Sung Min Kang, Kyung-Bok Lee, Seok Pyo Hong, Youngkyu Do, and Insung S. Choi. "Photophysical properties of noncovalently functionalized multi-walled carbon nanotubes with poly-para-hydroxystyrene." Carbon 46, no. 4 (2008): 714-716.

12) Xue, Chao-Hua, Ren-Jia Zhou, Min-Min Shi, Yan Gao, Gang Wu, Xiao-Bin Zhang, Hong-Zheng Chen, and Mang Wang. "The preparation of highly water-soluble multi-walled carbon nanotubes by irreversible noncovalent functionalization with a pyrene-carrying polymer." Nanotechnology 19, no. 21 (2008): 215604.

13) Nagamadhu, M.; Vijay Kumar, S.; Ravi Kumar, S.; Suraj, R.; Mohan Kumar, G.C. (2020). Dynamic Mechanical Analysis and Thermal Stability of Neem Wood Veneer Plastic Composites. Materials Today: Proceedings, 24(), 2265–2273.

14) Shen, Jianfeng, Weishi Huang, Liping Wu, Yizhe Hu, and Mingxin Ye. "The reinforcement role of different amino-functionalized multi-walled carbon nanotubes in epoxy nanocomposites." Composites Science and Technology 67, no. 15-16 (2007): 3041-3050.

15) Liu, Lei, and Jaime C. Grunlan. "Clay assisted dispersion of carbon nanotubes in conductive epoxy nanocomposites." Advanced Functional Materials 17, no. 14 (2007): 2343-2348.

16) T. Malyadri , Dr J. Suresh Kumar, Investigation Of Mechanical Properties For Basalt Fiber Compoite With Silica Nano Particles, International Journal of Emerging Technologies and Innovative Research, 2022, 9, 2349–5162.

17) T. Malyadri , Dr J. Suresh Kumar, Investigation Of Mechanical Properties For Basalt Fiber Composite With CNT Nano Particles, International Journal of Emerging Technologies and Innovative Research, 2022, 9, 2349–5162.

18) T. Malyadri , Dr J. Suresh Kumar, Investigation Of Mechanical Properties For Basalt Fiber Composite With Graphene Nano Parti- cles, International Journal of Emerging Technologies and Innovative Research, 2022, 9, 2349–5162.