

DYNAMIC MECHANICAL ANALYSIS OF EPOXY NANOCOMPOSITES REINFORCED WITH GRAPHENE AND NANOSILICA IN SINGLE CANTILEVER MODE

Anilkumar P R Assistant Professor, Department of Mechanical Engineering, Sapthagiri College of Engineering, Bangalore (Affiliated to VTU Belagavi)

Dr. L.H. Manjunatha, Professor, School of Mechanical Engineering, REVA University, Bangalore

Dr. T. Venkategowda Associate Professor, Department of Mechanical Engineering, Maharaja Institute of Technology Thandavapura, Mysore (Affiliated to VTU Belagavi)

Abstract

The present research work focused on fabrication and dynamic mechanical behavior of epoxy nanocomposites reinforced with graphene and nano silica. Five different nanocomposites were fabricated using simple compression molding method. Dynamic mechanical analysis is a powerful tool for measuring elastic properties of polymeric materials, the Dynamic Mechanical Analyzer (DMA) measures the changes in mechanical behaviour such as modulus and damping as a function of temperature, time, frequency, stress or a combination. These measurements are very important to know the performance of materials. The nano silica was varied from 0 % to 20 % in the increment of 4% with 6% constant graphene content. The impacts of nano silica content on dynamic mechanical properties were observed. The tests were carried out at different levels starting from room temperature to 150°C. The 16% nano silica content nanocomposite showed excellent dynamic mechanical properties as compared to other composites. The present results indicated that thermal stability and load bearing capacity of the prepared composites were increasing with the addition of nano silica content up to 16%. The obtained dynamic mechanical properties of prepared composites are acceptable and these can be used to make automotive body panels and bumpers.

Keywords: Epoxy, DMA, Storage modulus, Loss Modulus, Damping

Introduction

Now a day's polymer based nanocomposites are important categories of advanced materials because of their unique advantages. Time-dependent response and viscoelastic behavior is one of the most significant features of polymer nanocomposites. Polymer nanocomposites exhibit both elastic and viscous nature when they are subjected to an external load. When these materials are subjected to external load, they undergo elastic deformation and some energy will be stored. When the material is unloaded, this energy will be released and hence the elastic deformation disappears to reach its initial state. Polymer nanocomposites can be prepared by mixing the nanoparticles in terms of nano size with the molten liquid polymer matrix using different techniques. The nanocomposite material exhibits the desired properties, only when the nanoparticles are mixed and distributed homogeneously with the polymer matrix without forming any larger clusters.

Dynamic mechanical analysis (DMA) is routinely used to evaluate the most critical viscoelastic response of polymer materials. In dynamic mechanical analysis (DMA), a sinusoidal force is applied to a specimen to study thermal transitions and molecular motions of viscoelastic material. The DMA results describe the three attributes i.e. storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$) as a function of temperature and frequency. The ratio of E' and E'' is a measure of the damping properties of a material and is known as loss factor. Commonly, damping is measured in terms of loss factor (η) which is a measure of loss of energy per cycle of deformation. The variation of these three attributes can also be seen in glassy and rubbery regions. The storage modulus (E') represents the elastic properties and loss modulus (E'') represents the viscous properties of a polymeric material. As the heating continues, the glass transition (T_g) appears which indicates that the amorphous regions have begun to melt accompanied by the large scale motions in the chains of the amorphous regions. T_g is a temperature at which a material starts to soften.

Fabrication of Nano Composites

A. Materials

A nano particle reinforced polymer nanocomposite is developed using cold compression moulding technique. Graphene and nano silica are the reinforcement materials whereas the epoxy LY-556 is the matrix material. Hardener HY-951 was used with epoxy in the ratio of 10:1 for curing at room temperature. For fabricating the Epoxy-Graphene-nano silica composite plates, a mold box of dimensions 200 mm × 150 mm × 6 mm with a rectangular cavity of 180 mm × 90 mm × 3 mm using two mild steel rectangular plates.

B. Fabrication of Nano Composite

The nanocomposite laminates were fabricated by the following the steps mentioned below:

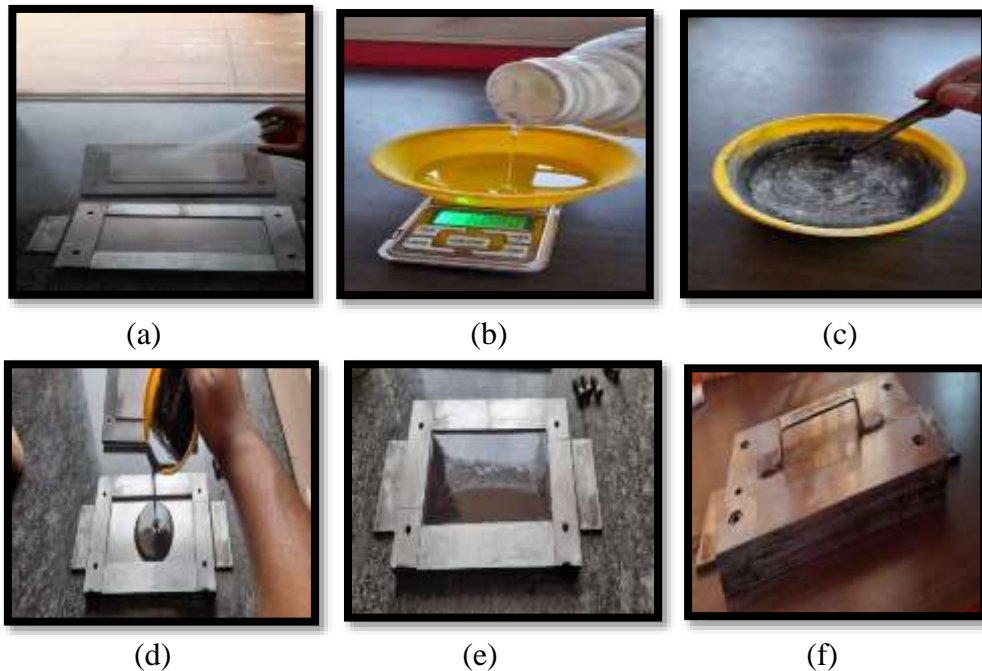


Figure 1. (a) Mold plates; (b) Epoxy-hardener mixing; (c) Mixing of Nano materials with resin mixture; (d) Pouring the mixture into the mold; (e) Mold filled with Resin and nanomaterial; (f) Mold closed after the mixture is poured.

At the beginning, the mould surface was cleaned and polished. Then mould releasing agent (wax) was applied on inner and outer surfaces of the mould. Epoxy resin and room temperature curing hardener are mixed thoroughly. Afterwards, graphene and Nano silica particles are poured into the mold cavity. As the mixture is completely filled into the mold cavity, the upper and lower plates of the mold box are closed by using screws. This closed mold box is allowed to solidify at room temperature up to 24 hrs. Subsequently, the mold plates are opened and composite laminate is ejected from the mold cavity. Same fabrication procedure was used to fabricate different composites for various percentage of nano silica and epoxy resin. Here the graphene content was kept constant. Latterly, the specimens were cut according the ASTM standard.

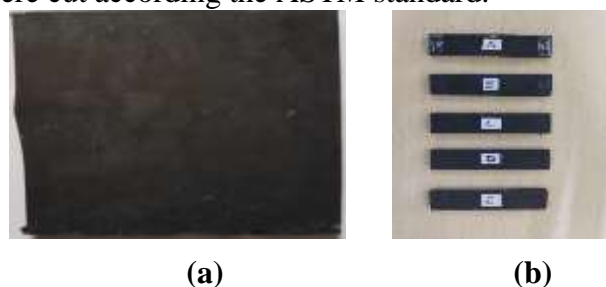


Figure 2. (a) Prepared nano composite; (b) Specimens for DMA test

Table No.1: Composition of Prepared Composite Laminates

Specimen	Epoxy (%)	Graphene (%)	Nano Silica (%)	Specimen
A	94	6	0	A
B	90	6	4	B
C	86	6	8	C
D	82	6	12	D
E	78	6	16	E

Dynamic Mechanical Analysis-An Experimental Setup

DMA can be employed to study thermal transitions of viscoelastic materials and the molecular motions associated with the transitions. Dynamic mechanical analysis of the developed nanocomposites was conducted on rectangular cross-section specimens of dimension 35 mm × 13 mm × 3 mm according to ASTM D4065 under single cantilever mode. In single cantilever; one end of specimen is clamped by cantilever. The dynamic properties such as storage modulus, loss modulus, damping factor ($\tan \delta$) and glass transition temperature (T_g) were measured using dynamic mechanical thermal analyzer DMA Q800, TA Instruments (fig 3) The test was carried out from room temperature to 160° at 5° C/min heating rate in a nitrogen flow environment and the frequency of 1 Hz was kept constant. The storage modulus (E') & loss modulus (E'') and the damping factor ($\tan \delta$), as a function of temperature (T), were determined by dynamic mechanical analyser and were plotted versus temperature.



Figure 3. (a) DMA test chamber and system; (b) Single cantilever set up for DMA

Results and Discussions

The dynamic mechanical properties such as storage modulus, loss modulus, damping factor and glass transition temperature are determined for different nano silica content at different temperatures and 1 Hz constant frequency. The table 2 shows the dynamic mechanical properties for the developed nanocomposites and figures 4-8.

Table No.2: E, E', E'', Tan Delta and Tg values for various composites

Composite samples	Storage Modulus (E) (MPa)	Loss Modulus (E'') (MPa)	Tan Delta ($\tan \delta$)	Tg from loss modulus curve (°C)	Tg from Tan δ curve (°C)
A	2906.467	390.4366	0.802812	78.95	90.34
B	3127.78	456.7667	0.7847951	83.35	92.80
C	3223.383	471.5803	0.7706753	86.03	94.19
D	3423.8	482.0328	0.758318	89.82	97.42

E	3299.197	459.1744	0.7430728	93.58	103.01
---	----------	----------	-----------	-------	--------

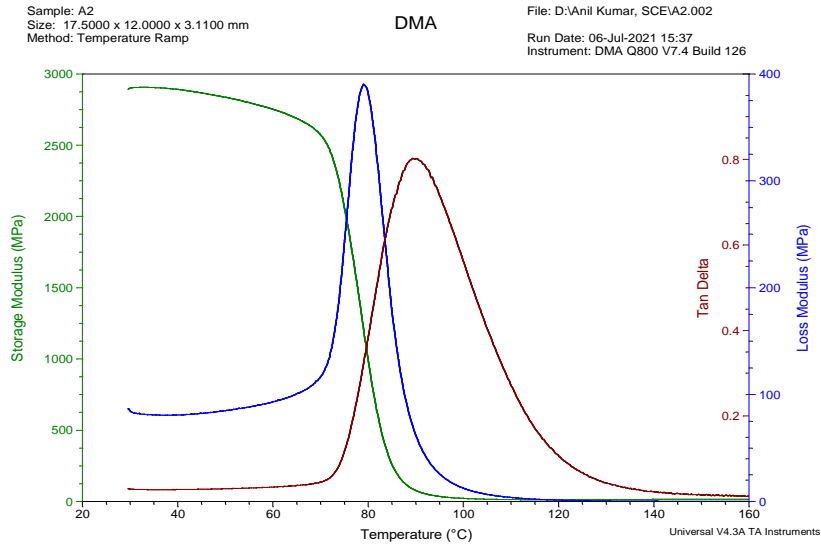


Figure 4. E, E', E'' V/S temperature for composite A

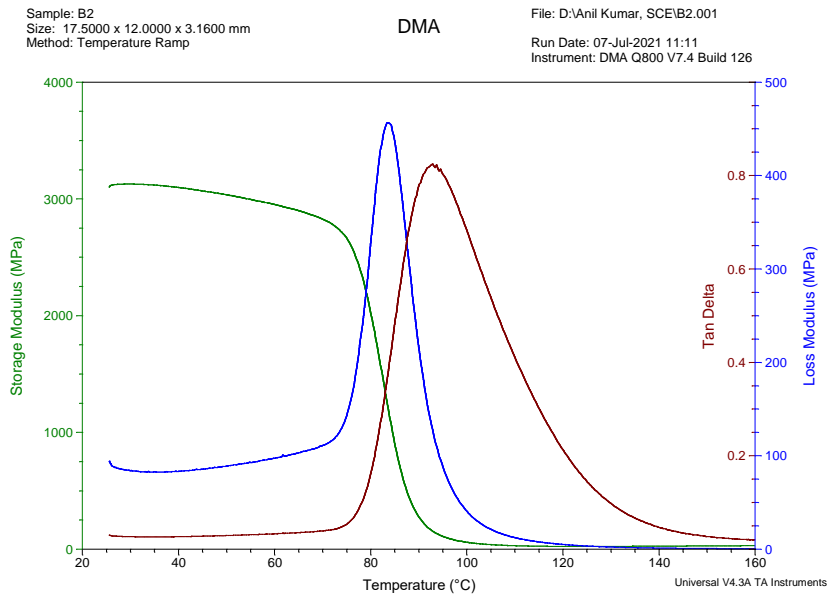


Figure 5. E, E', E'' V/S temperature for composite B

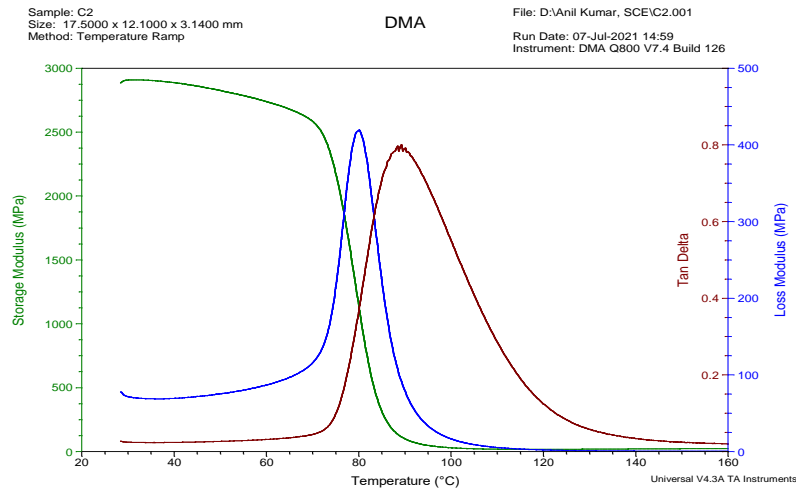


Figure 6. E, E', E'' V/S temperature for composite C

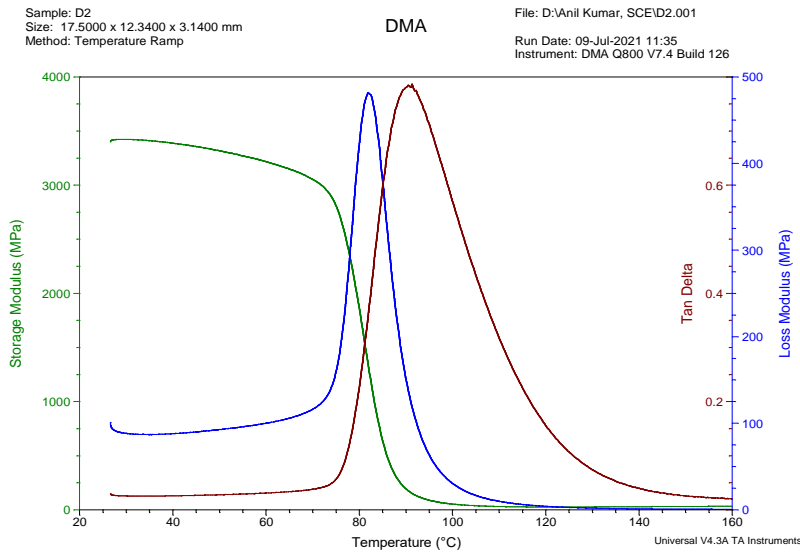


Figure 7. E, E', E'' V/S temperature for composite D

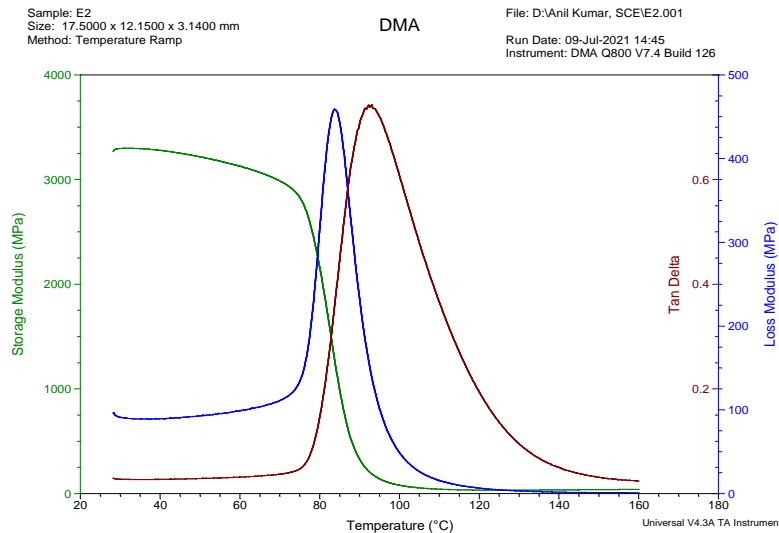


Figure 8. E, E', E'' V/S temperature for composite E

A. Storage Modulus (E)

The change in storage modulus with respect to temperature at constant frequency of 1Hz for various percentage of Nano silica is shown in figure 9. From this figure, it was noticed that at the beginning, the intensity of storage modulus value is constant for all composites. This constant is attributed to the immobility of polymer molecules. On further heating is continued, temperature is increased and the loss modulus starts decreasing because of the stiffness loss in the matrix. The storage modulus was better in specimen D than other composites. This might be due to improved interaction nano silica with the epoxy at higher percentage. This excessive storage modulus indicates better load transfer between epoxy and nano silica. The specimen D has highest storage modulus of 3423.8 MPa as compared all the composites at room temperature and its increment is 17.8% as compared to 0% nano silica. Greater storage modulus value depicts greater elastic behaviour of the composite and efficient stress transfer between Nano silica and epoxy.

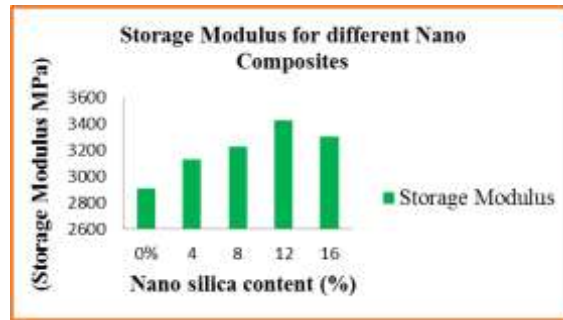


Figure 9. Storage modulus V/S Nanosilica content

B. Loss Modulus (E'')

The change in storage modulus with respect to temperature at constant frequency of 1Hz for various percentage of Nano silica is shown in figure 10. From this figure, it was noticed that at the beginning, the intensity of loss modulus value was constant for all composites. On further heating is continued, temperature is increased and the loss modulus value starts increasing because of mobility of molecules. The results showed that inclusion of Nano silica particles with graphene and epoxy improves the value of loss modulus. Specimen D has shown highest value of loss modulus than other composites. If a composite having a high value of loss modulus value, it depicts more elastic behaviour of nanocomposite and efficient stress transfer between nano particles and epoxy. As the temperature increased, the value of loss modulus starts increasing in the plastic region and declined in the rubbery region after achieving the peak (apex) temperature. The peak temperature of the curve is known as glass transition temperature (T_g). Normally this apex temperature indicates the highest operating temperature of the developed composite. The decline in storage modulus in the rubbery region with the rise in temperature indicates that the composite has turned into viscous phase. As clear from the figures that specimen D has shown the highest value of loss modulus 482.0328 MPa at the peak temperature and 23.5% has been increased as compared to specimen A. The results revealed that specimen D is dynamically stable at temperature and load.

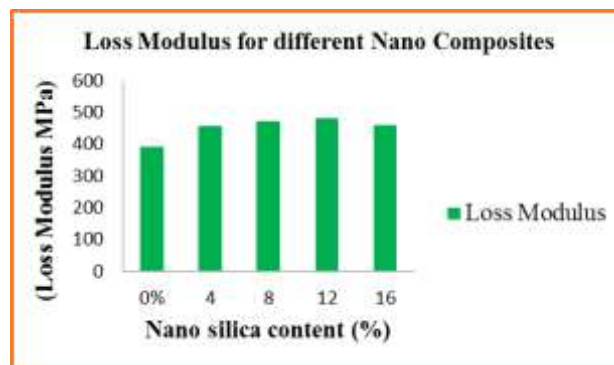


Figure 10. Loss modulus V/S Nanosilica content

C. Damping factor ($Tan \delta$)

Damping factor is used to represent elastic nature of a material. As damping factor increases, elastic nature will increase. A high value of damping factor depicts non-elastic nature of a material. The variation of damping factor at constant frequency of 1Hz for various percentage of Nano silica is shown in figure 11. It was viewed that after the addition of nano silica, the $\tan \delta$ peak has decreased as compared to 0% Nano silica. The highest value of peak was shown by specimen E and its value is 0.7430728 and its decrement is 7.4% in comparison to 0% Nano silica. This decrease could be attributed to the decrease in mobility of polymer molecules. The peak of the $\tan \delta$ follows the order $A > B > C > D > E$. Least value of damping indicates good load bearing capacity of a composite material. This fact may be because of strong adhesion of nano silica with the epoxy.

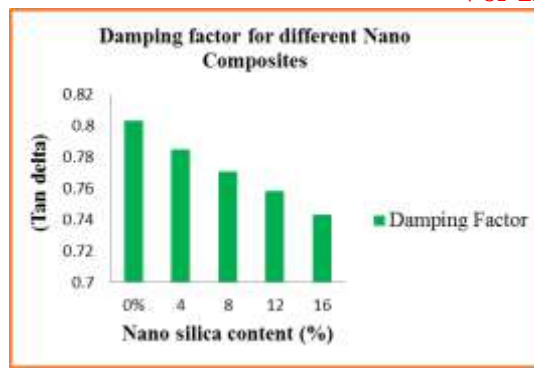


Figure 11. Tan delta modulus V/S Nanosilica content

D. Glass Transition Temperature (T_g)

Glass transition temperature is a point at which the polymer composite turned from a glassy to rubbery form. Higher value of T_g depicts the higher thermal stability of a polymer composite. Polymer composite starts to become soften and drops its property after it has been reached T_g due to the breakdown of cross linking between reinforcement and matrix molecules as further temperature is increased. There are many methods for T_g determination. In this research, T_g values were obtained from two different ways: Apex of loss modulus and of $\tan \delta$ curves respectively. The results are summarized in table 1. On comparison of two ways; the T_g temperatures obtained from the $\tan \delta$ curves (apex values) are slightly more than the temperatures obtained from loss modulus curves (apex values). As clear from the table 1 that T_g has increased after the addition of Nano silica. This indicates that elastic behaviour of developed nanocomposite has increased. The increase in T_g value after the addition of nano silica from 0% to 20% is 18.53% from the apex of loss modulus curves and 14.63% from the apex of $\tan \delta$ curves. Specimen E has shown the highest value of T_g . The highest T_g value denotes the superior structural stability due to the restriction of movement of polymer molecules.

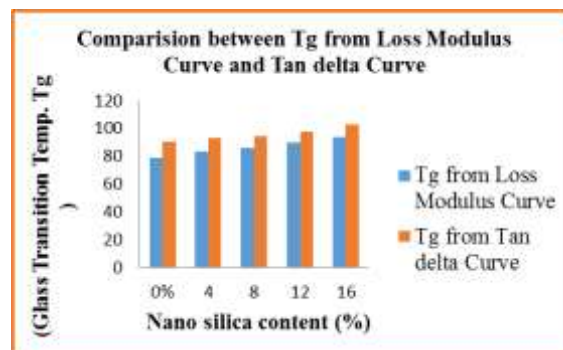


Figure 12. Glass transition temperature V/S Nanosilica content

Conclusions

- The results of DMA clearly show the improvement in storage modulus and loss modulus after the addition of nano silica with the graphene and epoxy. Composite D has shown the highest value of storage and loss modulus than other composites. Different nano silica content improved the viscoelastic behaviour of composite
- Reduction in damping factor was observed with the increase in nano silica content. The specimen E has shown the least value of damping factor.
- The incorporation of nano silica has improved the glass transition temperature of developed composites as compared to 0% nano silica content composite.
- Finally, it was noticed that nano silica particles have tough adhesion with the graphene and epoxy. Results imply that specimen D (16% nano silica) is the better composite and can be used in automotive and aerospace applications.

References

1. Chuan Her, Kuan-Yu Lin, “Dynamic mechanical analysis of carbon nanotube reinforced nanocomposites,” *J Appl Biomater Funct Mater*, 2017; Vol. 15, pp. 13-18, 2017.
2. William K. Goertzen, M.R. Kessler, “Dynamic mechanical analysis of fumed silica/cyanate ester nanocomposites,” *Science direct, Elsevier*, Vol. 39, pp. 761–768, 2008.
3. B. Surajarusarn, S. Hajjar-Garreau, G. Schrodj, K. Mougin, T. Amornsakchai, “Comparative study of pineapple leaf microfiber and aramid fiber reinforced natural rubbers using dynamic mechanical analysis,” *Polym. Test*, Vol. 82 pp. 1-8, 2020.
4. T.K. Timothy, Albert U. Ude, Chinnasamy Vivekanandhan, “Concise review on the mechanical characteristics of hybrid natural fibres with filler content,” *Mater. Sci*, Vol. 7, pp. 650–664, 2020.
5. CH Gangadhara Rao, A Nageswara Rao, “Mechanical dynamic analysis of banana fiber reinforced polymer matrix composites,” *International, Journal of Applied Research*, Vol. 1 pp. 396–400, 2015.
6. H. Asadian, K. Shelesh-Nezhad, “Simulation of dynamic mechanical and viscoelastic behavior in polymer/clay nanocomposites,” *Wiley*, Vol. 41, pp. 817–823, 2020.
7. S. Patra, P.M. Ajayan, T.N. Narayanan, ‘Dynamic mechanical analysis in materials science,’ *The Novice’s Tale, Oxford Open Materials Science*, vol.1, pp. 1-12, 2021).
8. A. Manral, P.K. Bajpai, “Static and dynamic mechanical analysis of geometrically different kenaf/PLA green composite laminates,” *Wiley*, Vol. 41, pp. 691–706, 2020.
9. M. Rajesh, J. Pitchaimani, “Mechanical and dynamic mechanical behaviour of novel glass natural fibre intra-ply woven polyester composites,” *Indian Acad. Sci*, Vol.42, pp. 1215–1223, 2017.
10. O.G. Echiye, G.B. Abugh, Ashwe, Nyior., Humphery.A., Iortyer, “Dynamic mechanical analysis of Ukam (*Cochlosperum Planchonii*) fibres reinforced polyester composites,” *Int. J. Sci. Eng. Res*, Vol. 9, pp. 442–453, 2018.