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Dynamic Mechanical Analysis of Hemp Fiber Reinforced Polymer Matrix Composites

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***Abstract*— Dynamic mechanical analysis (DMA) is a powerful technique for the characterization of the Viscoelastic properties of polymers. DMA measures the modulus (stiffness) and damping (energy dissipation) properties of materials as they are deformed under dynamic stress. These measurements provide quantitative information about the performance of materials. This technique can be used to evaluate a wide variety of materials such as thermoplastics, composites, thermosets, elastomers, films, fibers, coatings and adhesives. DMA is a valuable technique because of its high inherent sensitivity and is the most sensitive thermal analysis technique for the measurement of the glass transition regions, T_g. Secondary relaxation events readily observed by DMA, simply cannot be detected by any other thermal technique.**

In the present work, hemp and glass fiber were used as the reinforcement fiber and General Purpose Resin was used as the matrix for fabrication of Natural and Hybrid fiber reinforced composites (FRP's). It is essential to evaluate the Viscoelastic properties of fabricated composites for improved mechanical and thermal properties. The dynamic moduli, mechanical loss and damping behavior as a function of temperature of the systems were studied using dynamic mechanical analysis (DMA). In the present work DMA was carried out on a fabricated Natural and Hybrid FRP's. The fibers considered are Hemp & Glass, where GP resin is considered as matrix. For DMA analysis loads of 0.5, 0.7 & 1.0 gram with a temperature gradient are considered on both Natural & Hybrid FRP's. The Storage Modulus (E') values are found to be maximum for composites with 1.0 gram fiber loading, indicating that the incorporation of hemp & glass fiber in General Purpose Resin matrix induces reinforcing effects appreciably at higher temperatures. The loss modulus and damping peaks were found to be lowered by the incorporation of fiber. The height of the damping peaks depended on the fiber content.

Keywords— Dynamic Mechanical Analysis (DMA); Glass transition temperature (T_g)

I. INTRODUCTION

Natural fiber has been in a wide use since the evolution of the human race. They had got wide acceptation in communities for their flexibility and strength. Recent trends in the area of fiber rein-forced composites have drawn a string in using these natural fibers as their reinforcement. The natural fiber imparts lower durability and lower strength compared to glass fibers. However, low specific gravity results in a higher specific strength and stiffness than glass. Natural fibers offer good thermal, dielectric and acoustic insulation properties along with ease in processing technique without wearing of tools.

Most of these fibers are produced in developing countries like China, India and Brazil, etc. In a historical perspective composites like straw reinforced walls, bows and chariots made of glued layers played important roles in their survival. Though many of these fibers are limited to their epidemic regions due to high local demand for many years, advent of synthetic fibers have affected the market of natural fibers. After being solely used for their electromagnetic properties, using composites to improve the structural performance of spacecraft and aircraft became popular in last two decades of the previous century. Especially, increased constrains and future environmental road maps, have been stressing automotive industries to decrease their carbon foot prints. These stringent measures have become boon to the lagging natural fiber industries, and thereby creating new challenges for researchers in finding sources of fiber, fabrication techniques and applications of natural fibers. The easy availability of natural fibers and manufacturing have motivated researchers worldwide recently to try locally available inexpensive fibers and to study their feasibility of reinforcement purposes and to what extent they satisfy the required specifications of good reinforced polymer composites [1]. Many studies proved them to be potential contenders to synthetic fiber to some extent. After reviewing the existing literature available on natural fiber composites, various authors had put efforts in designing composites based on the needs of composite industry. There has been a wide variety of literature available on natural fibers such aspen, abaca, bagasse, bamboo, banana, coir, date palm, flax, henequen, isora, jute, kapok, kenaf, oil palm, pine-apple, ramie, and sisal [2–18]. Natural fiber composites provide comparable specific strength with that of synthetic fiber composites, due the low density offered by natural fibers [19]. There are many parameters which affect the performance of a natural fiber-reinforced composite. Chemical modification improves fiber matrix adhesion, their results and effects on the physical properties of composites [20]. Aspect ratio has a considerable effect on composite properties, hence it is important to conserve fiber length as much as possible during composite processing operations [21]. Mechanical properties of the composite vary with various amounts of fiber volume ratio. But there have been a very few papers dealing with Palmyra fiber, the present fiber of our interest [22–24].

The Hemp fiber is a tall and erect palm, and can live 100 years or more and reach a height of 30 m, with a canopy of large, fan-shaped leaves several dozen found spreading 3 m across. Each and every part of the tree is a notable socio-economic value for people in Southern India. The mid-ribs of the leaves and the fibers from their stalks are used in making industrial brushes and brooms. The fiber has good resistance to friction and heat, and will withstand many chemicals and solvents. The density of Palmyra (0.7 g/cm^3) is least among all known natural fibers that are being commercially used in manufacturing natural fiber composites. This is highly favorable property from the view point of light-weightiness. The present composite can find extensive application in non-structural, low-performance uses. Hence, the hemp fibers are most suitable for making reinforcement in green composite materials.

II. EXPERIMENTAL PROCEDURE

A. Preparation of die

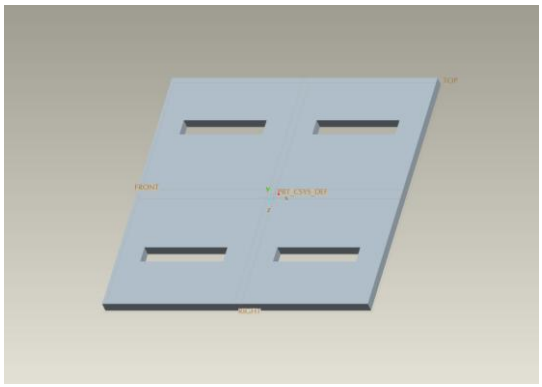

Fig. 1 Female Die

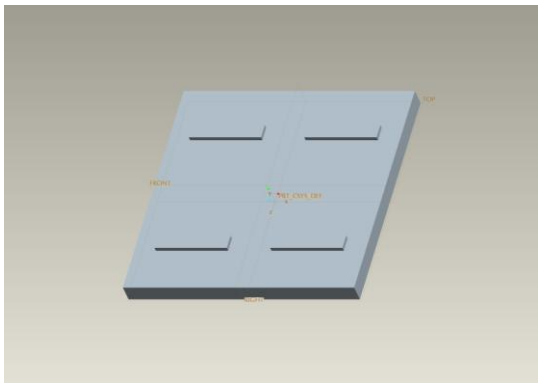

Fig.2. Male Die

B. Materials

Fibers	-	hemp fiber, glass fiber
Matrix	-	General purpose resin (GPR)
Accelerator	-	Cobalt naphthenate
Hardener	-	Methyl ethyl ketone peroxide

C. Preparation of composite

Firstly, the hemp fiber was taken and it was neatly separated by hand and the fibers were cut to uniform size of 10 mm length. After cutting the fiber, 0.5 gram, 0.7 gram, 1.0 gram of the fiber was measured using digital weighing machine. 0.5 gram of the fiber was taken and evenly arranged in a die measuring 50 x 10 x 3 mm^3 and pressed into a mat. This Composite sample was prepared by impregnating the fiber with the GP resin to which 0.9 volume percent cobalt naphthenate (accelerator) and 1 % methyl ethyl ketone peroxide (hardener) was added. The fiber was kept in the closed die and cured for 3 to 5 hours. Finally we get the natural fiber composite with required dimensions (50 x 10 x 3 mm^3).

In the similar process, we prepared the remaining samples with 0.7gram, 1.0gram fiber weight content and also we prepared the sandwich composites (hemp + glass fiber) with 0.5gram, 0.7gram, and 1.0gram weight content.

D. Experimental conditions

Experiments are conducted by applying a small cyclic deformation to a sample over a wide temperature range. The following experimental conditions were used to analyze the polymeric sample.

Instrument	-	Diamond DMA
Heating rate	-	5 ^{0}C/min
Temperature range	-	120 ^{0}C to 150 ^{0}C
Frequencies	-	0.5, 1, 2, 5, and 10 Hz
Maximum load range	-	up to 18 N
Deformation mode	-	Three point bending
Sample dimensions	-	Length (l)-50 mm, Width (w)-10 mm, Thickness (t)-3 mm.
Deformation amplitude	-	40 µm
Cooling	-	Liquid nitrogen with Automated Cooling

Accessory

E. Test procedure

In this test, the sample is clamped inside an environmental chamber and cooled to -120°C. Frequency, amplitude, and a temperature range appropriate for the material are input. A small deformation is then applied, and the resulting force is measured. When the deformation is complete, the temperature is raised by 5-10°C, and the deformation is repeated. For composites, testing continues up to 150°C, at which time the composite starts to degrade or melt. Sample DMA data is shown in fig.3 on the graph below:

From the force response of the sample it's possible to calculate the modulus (stiffness) and dissipative (tan δ) characteristics of the composites. Typically, experiments are conducted by varying temperature at a constant deformation frequency (1 Hz); other frequencies can also be used to simulate conditions more like field applications. Experiment can be conducted for 3-point bending.

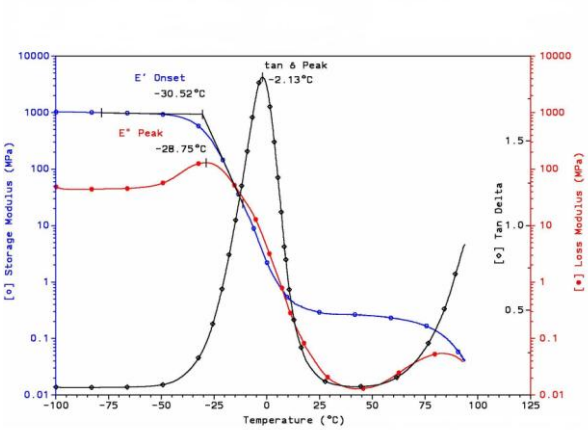

Fig. 3 DMA results (E', E" and tan delta) for polymer dampening material

III. RESULTS AND DISCUSSIONS

Dynamic mechanical analysis has been a well established method in thermal analysis. The DMA analysis consists of the observation of the time dependent deformation behaviour X(t) of a sample under periodic, mostly sinusoidal deformation force with very small amplitudes F(t). It is possible to calculate the young's modulus (or) storage modulus (E') and loss modulus (E") as well as mechanical loss factor (or) tan δ (damping) is dependence on temperature and deformation frequency. Results are typically provided as a graphical plot of E', E", and Tan δ versus temperature. DMA identifies transition regions in composites, such as the glass transition, and may be used for quality control or product development.

The viscoelastic properties of polymer matrix composites were measured using TA instruments diamond dynamic mechanical analyzer. Rectangular specimens of 50 x10 x 3 mm^3 were used for the analysis. The analysis was done in three-point bending mode at a frequency of 1 Hz. The samples heated from room temperature to 150°C at a heating rate of 5°C/min. Dynamic mechanical properties of fiber-reinforced composites depend on various factors such as fiber loading, fiber orientation and the nature of fiber-matrix interface region. Dynamic tests, over a wide range of temperature and frequency are especially sensitive to all kinds of transitions and relaxation processes of matrix resin.

A. Glass transition temperature (Tg)

Temperature region over which the material changes from a rigid, glassy solid to a more flexible, elastomeric solid. The glass transition region is denoted by T_g, the glass transition temperature.

During measurement of the moduli (E', E") and damping behavior (tan δ) of a polymer at a chosen oscillatory frequency over a sufficiently wide range of temperature, the effect of the polymer's glass transition can be clearly observed. The glass transition (sometimes called the α transition) is a reversible change of the polymer between rubbery and glassy states, the temperature at which this occurs, called the glass transition temperature Tg, can be measured accurately.

The glass transition is detected as a sudden and considerable (several decades) change in the elastic modulus and an attendant peak in the tan δ curve. This underscores the importance of the glass transition as a material property, for it shows clearly the substantial change in rigidity that the material experiences in a short span of temperatures. Accordingly, the glass transition temperature is a key factor in deciding the usefulness of a polymer.

Tg is defined as the temperature at which

- Peak on Tan δ curve.
- Peak on Loss Modulus curve (E").
- Half height of Storage Modulus curve (E').
- Onset of Storage Modulus curve(E')

Hence for a polymer system, the above value should be the value at a specific frequency i.e. the above mentioned temperatures in the E", tan δ and E' curves should coincide with one another. But it was observed by that these points won't coincide for composites, this is due to the complexity of dynamic mechanical behaviour of composites, arising for the restricted movement of molecules in matrix. This behaviour mainly depends on the dynamic mechanical properties of the matrix.

The variation of storage modulus, loss modulus and damping factor as a function of temperature of hemp fiber and hemp & glass fiber composites as shown in Figures 4 to 9.

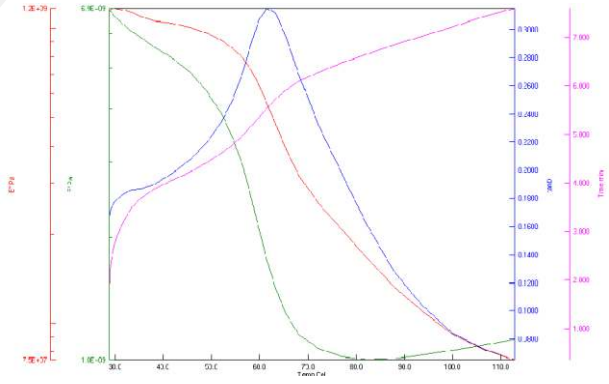

Fig.4 Shows DMA results (E', E" and tan delta) for polymer dampening material (0.5 gr. Hemp Fiber)

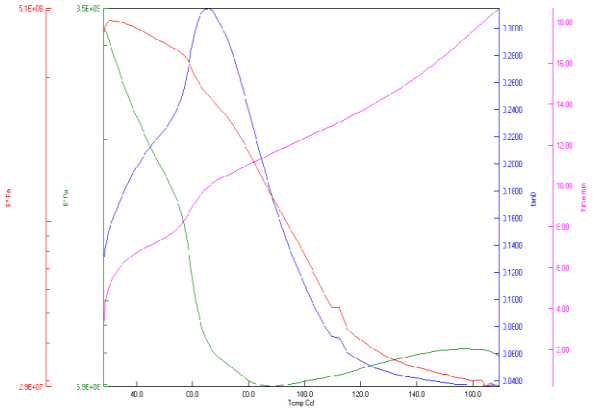

Fig.5 Shows DMA results (E', E" and tan delta) for polymer dampening material (0.7 gr. Hemp Fiber)

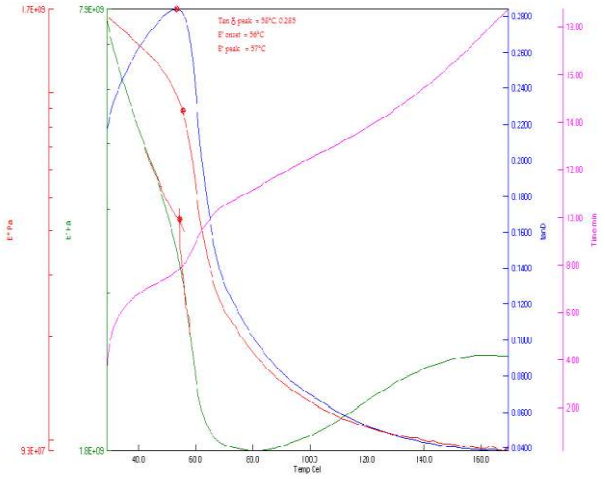

Fig.6 Shows DMA results (E', E" and tan delta) for polymer dampening material (1.0 gr. Hemp Fiber)

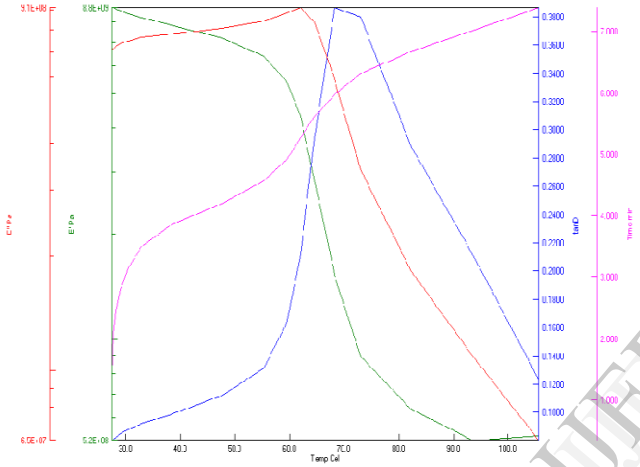

Fig.7 Shows DMA results (E', E" and tan delta) for polymer dampening material (0.5 gr. Hemp + Glass Fiber

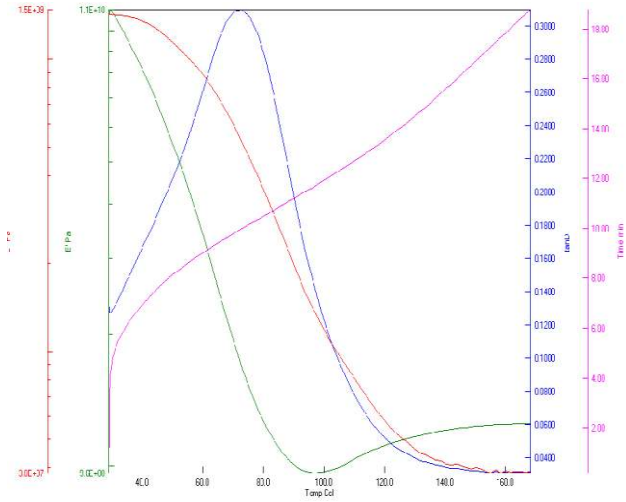

Fig.8 Shows DMA results (E', E" and tan delta) for polymer dampening material (0.7 gr. Hemp + Glass Fiber)

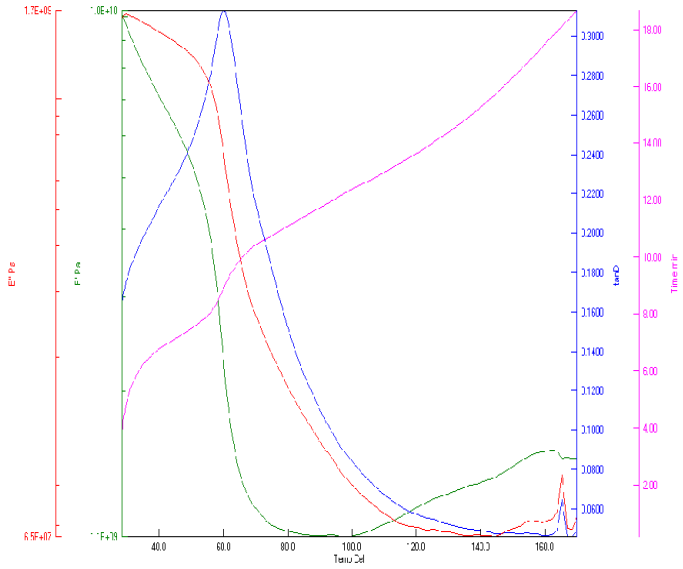

Fig.9 Shows DMA results (E', E" and tan delta) for polymer dampening material (1.0 gr. Hemp + Glass Fiber)

B. Effect of temperature on dynamic storage modulus (E')

Dynamic storage modulus (E') is the most important property to assess the load bearing capacity of a composite material. The variation of storage modulus as a function of temperature of hemp fiber and hemp & glass fiber composites is shown in Figures 4 to 9.

There is a prominent increase in the modulus of the matrix with the incorporation of hemp & glass fiber over the entire region compared to hemp fiber composites from figures 7 to 9. This may be due to the increase in the stiffness of the matrix with the reinforcement effect imparted by the fibers that allowed a grater degree of stress transfer at the interface. As the temperature increases, E' decreases and then there is a sharp decline in the E' value at the glass transition region. This behaviour can be attributed to the increase in the molecular mobility of the polymer chains above Tg. The drop in the modulus in the glass transition region is much less for hemp & glass fiber reinforced composites than for the hemp fiber composites. That is, the difference between moduli of the glassy state and rubbery state is smaller in composites than in the hemp fiber composites, which clearly indicates the reinforcing effect of glass fibers. This is due to the considerable increase in the modulus both in the rubbery region as well as in the glassy region. From the figures, it is clear that there is a gradual increase in the E' values upon fiber loading.

C. Effect of temperature on loss modulus(E'')

Loss modulus (E") is a measure of the energy dissipated as heat per cycle under deformation (or) it is the viscous response of the material. Figures 7 to 9 show the trends of variation of loss modulus for the different composite systems with variation of temperature. From the figures 4 to 9, it is clear that the incorporation of fiber causes broadening of the loss modulus peak. The peak broadening can be attributed to the inhibition of relaxation process with in composites. This may be due to the increase in the number of chain segments

as well as more free volume upon fiber condition. It can be seen that addition of glass fiber, there is a shift in the glass transition Tg towards the higher temperatures on increasing the fiber content as shown in figures 7 to 9. This is primarily attributed to the segmental immobilization of the matrix chain at the fiber surface. The loss modulus value in the transition region is much high for hemp & glass fiber composites when compared to the hemp fiber composites. Also, the loss moduli of the composites were increased on increasing the fiber content. The higher modulus at this temperature is due to the increase in internal friction that enhances the dissipation of energy. Additionally, the presence of high modulus glass fibers that reduced the flexibility of the material by introducing constraints on the segmental mobility of the polymeric molecules at the relaxation temperatures.

D. Effect of temperature on damping factor(Tanδ)

The ratio of the loss modulus to the storage modulus is measured as the mechanical loss factor (tan δ). The damping properties of the material give the balance between the elastic phase and viscous phase in a polymeric structure. In composites, damping is influenced by the incorporation of fibers. It is observed that as temperature increases, damping goes through a maximum in transition region and then decreases in the rubbery region. Below T_g, damping is low because, in that region, chain segments are in the frozen state. Hence, the deformations are primarily elastic and the molecular slips resulting in the viscous flow are low. Also, in the rubbery region, the molecular segments are quite free to move and hence the damping is low and thus there is no resistance to flow.

The position and height of tanδ peak are indicative of the structure and properties of composite material. The variation of the mechanical damping parameter with variation of temperature for different composite systems is shown in figures 4 to 9. Composites have very less damping in the transition region compared to hemp fiber composites because fibers carry a greater extent of stress and allow only a small part of it to strain the interface. Therefore energy dissipation will occur in the polymer matrix at the interface and strong interface is characterized by less energy dissipation. Among the composites, lowering of tan δ peak height upon fiber loading was observed. This may be due to the restriction of movement of polymer molecules by the incorporation of rigid fibers. The lowering of peak height also indicates good interfacial adhesion. Addition of glass fiber has shifted the tan delta peak to the right, as well as increasing it. This shows the effectiveness of glass fiber as a reinforcing agent. The shifting of Tg to higher temperature can be associated with the decreased mobility of the chain with the addition of fibers.

IV. CONCLUSIONS

In the present work, the Diamond dynamic mechanical analyzer of TA instruments is used which is excellently suitable for the characterization of the Viscoelastic properties of hemp & glass fiber reinforced composites. The glass transition can be determined considerably more easily using Dynamic Mechanical Analysis. In addition, the quality of the composites material, such as variation of fiber-matrix adhesion, property profile of the coupling agent, damping behavior, elasticity with respect to temperature is studied.

Based on the work the following conclusions were drawn:

- Dynamic mechanical properties of hemp and hemp & glass fiber reinforced general purpose resin (GPR) composites are greatly dependent on the volume fraction of the fiber.
- The dynamic modulus shows a decrease with incorporation of fiber below the glass transition temperature and has a positive effect on the modulus at temperatures above Tg.
- The maximum improvement in properties is observed for composites with 1 gram fiber loading, which is chosen as the critical fiber loading. At this maximum fiber loading i.e., 1gram, the loss modulus peak gets broadened emphasizing the improved fiber/matrix adhesion.
- Moreover, an additional peak occurs at high fiber loading in the tan δ curves, due to the interlayer effect.
- Addition of fiber lowers the tan δ peak height, which again points to the improved fiber/matrix adhesion. The glass transition temperature is shifted positively on the addition of fiber.

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