

Thermal Characterization of Bamboo and Flax (BFBF) Mat Reinforced Epoxy Hybrid Composite

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Abstract - Hybrid composite materials are the great potential for engineering material in many applications. Hybrid polymer composite material offers the designer to obtain the required properties in a controlled considerable extent by the choice of fibers and matrix. The properties are tailored in the material by selecting different kinds of fiber incorporated in the same resin matrix. In the present investigation, the mechanical properties of Bamboo and flax mat reinforced epoxy hybrid composite were studied. The hand lay-up technique was adopted for the fabrication of hybrid composite materials. The thermal properties such as DSC, TGA of the hybrid composites were determined. The thermal properties were improved as the mat reinforcement content increased in the matrix material.

Key word- DSC, TGA Hand lay up technique.

I. INTRODUCTION

Composites are multi-functional materials consisting of two or more chemically distinct constituents, on a macro-scale, having a distinct interface separating them. More than one discontinuous phases are embedded in a continuous phase to form a hybrid composite. The discontinuous phase is usually harder and stronger than the continuous phase and it is called the hybrid reinforcement and the continuous phase is termed the matrix. The matrix material can be classified into metallic, polymeric and ceramic. Recently, the polymer matrix composites have been widely used for many applications like automotive parts, airplanes interior parts, household appliances and construction materials [1]. The reinforcing phase can either be fibrous or non-fibrous (particulates) in nature and if the fibers are derived from plants or some other living species, they are called natural fibers. The environmental issues have resulted in considerable interest in the development of new composite materials with addition of more than one reinforcement that are biodegradable resources, such as natural fibers as low-cost and environment-friendly alternative for synthetic fibers [2]. The hybrid fibers in the composites can withstand higher load compared to single-fiber reinforcements in different direction based on the reinforcement, and the surrounding matrix keeps them in the desired location and orientation, acting as a higher load transfer medium between them [3]. The increased environmental consciousness is promoting the use of eco-friendly fibers, extracted from plants, vegetables, minerals and animals instead of synthetic fibers [4]. Automobile sector has various applications of NFRP composites to manufacture different parts such as door panels, headliners, package trays, dashboards and interior parts of vehicle [5]. In recent years, the use of flax fibers as reinforcement in composites has gained popularity due to an increasing requirement for developing sustainable materials. Flax fibers are cost effective and offer specific mechanical properties comparable to those of synthetic fillers. Composites made of flax fibers with thermoplastic, thermoset, and biodegradable matrices have exhibited good mechanical properties [6]. The mechanical properties of composite materials depend on many factors, which include fiber length, shape, size, composition, orientation and distribution, as well as volume fraction, mechanical properties of the polymer matrix, manufacturing techniques and adhesion or connection between the fibers and the matrix [7].

Thermal analysis is an important tool in the characterisation of polymeric materials. During the fabrication of new products from polymer composites, a knowledge of the thermal stability of their components is essential. The threshold temperature for break down determines the upper limit of temperature in fabrication. Optimisation of the processing temperature and time with an understanding of the matrix, the reinforcing element and the interface can lead to a best balance of composite properties. Differential scanning calorimetry (DSC) helps us to obtain quantitative information about the melting and phase transitions by measuring the heat flow rate associated with a thermal event as a function of time and temperature. [8] The thermal stability of individual polymers can be enhanced to a greater extent by blending it with other polymers or by

reinforcing with fibres. The synergism so obtained is usually attributed to the interfacial adhesion of the components. Various researchers have previously studied the thermal behaviour of rubber blends and composites in detail [9]. CorreAa et al. examined the influence of short fibres on the thermal resistance of the matrix, its T, and kinetic parameters of the degradation reaction of thermoplastic polyurethane. They also found that the thermal resistance of aramid fibre-reinforced composites was greater than that of carbon fibre-reinforced composites.[10] TG has been used by Faud et al. to determine the filler content of wood-based composites [11]. Suhara reported the thermal degradation of short polyester fibre-polyurethane elastomer composites. They observed that incorporation of short fibres enhanced the thermal stability of the elastomer. [12] George characterised the thermal behaviour of pineapple fibre reinforced polyethylene composites. Ahmed et al.[13] reported the thermal studies on sulphur, peroxide, and radiation cured NBR and SBR gum vulcanisates and also with fillers such as carbon black and silica. It was found that the radiation cured NBR and SBR vulcanisates possessed better thermal stability.[13] Seema investigated the thermal degradation of short nylon 6 fibre reinforced SBR composites.[14]

II. EXPERIMENTAL

Natural Fibre and Matrix

Flax and Bamboo mat were supplied by Compact Buying Services, Faridabad, Haryana. Bi-directional mats of these fibers have been used for fabrication of bio- composites. Epoxy resin and hardener was supplied by Excellence Resins, Meerut, UP

Bamboo Fiber:

Bamboos include some of the fastest-growing plants in the world due to a unique rhizome-dependent system. Certain species of bamboo can grow 91 cm (36 in) within a 24-hour period, at a rate of almost 4 cm (1.6 in) an hour (a growth around 1 mm every 90 seconds, or 1 inch every 40 minutes). Giant bamboos are the largest members of the grass family. Bamboos are of notable economic and cultural significance in South Asia, Southeast Asia and East Asia, being used for building materials, as a food source, and as a versatile raw product. Bamboo has a higher specific compressive strength than wood, brick or concrete, and a specific tensile strength that rivals steel. Density of bamboo fibre is 1.1gm/cm³[12].

Flax Fiber:

Flax (*Linum usitatissimum*), also known as common flax or linseed, is a member of the genus *Linum* in the family Linaceae. It is a food and fiber crop cultivated in cooler regions of the world. Textiles made from flax are known in the Western countries as linen, and traditionally used for bed sheets, underclothes, and table linen. Its oil is known as linseed oil. In addition to referring to the plant itself, the word "flax" may refer to the unspun fibers of the flax plant. The plant species is known only as a cultivated plant, and appears to have been domesticated just once from the wild species *Linum bienne*, called pale flax. Density of flax fibre is 1.5gm/cm³[13].



Fig.1 - Bamboo Mat(0⁰/ 90⁰orientation)



Fig.2 - Flax Mat(0⁰/ 90⁰orientation)

Table 1- Chemical And Mechanical Properties of Natural Fiber [14]

Fiber name	Cellulose [wt%]	Lignin [wt%]	Hemi-cellulose [wt%]	Fiber name	Density [g cm ⁻³]	Diameter [μm]	Tensile strength [MPa]
Abaca	56-63	7-9	20-25	Abaca	1.5	-	400
Bamboo	26-43	1-31	30	Bamboo	1.1	240-330	500
Banana	83	5	-	Banana	1.35	50-250	600
Coir	37	42	-	Coconut	1.15	100-450	500
Cotton	82.7-91	-	3	Coir	1.2	-	175
Curauá	73.6	7.5	9.9	Cotton	1.6	-	287-597
Flax	64.1-71.9	2-2.2	64.1-71.9	Curauá	1.4	170	158-729
Hemp	70.2-74.4	3.7-5.7	17.9-22.4	Flax	1.5	-	800-1 500
				Hemp	1.48	-	550-900

Epoxy Resin

Araldite LY 556 is manufactured by Huntsman Advanced Materials having the following outstanding properties has been used as the matrix material. Epoxy Resin was purchased from Excellence Resins, Meerut, UP. Epoxy shows long open time ,high shear, peel strength, easy to apply, electrically insulating, good resistance to static and dynamic loads.

Hardener

Hardener is a curing agent for epoxy resin. Epoxy resins require a hardener to initiate curing. It is also called the catalyst, the substance that hardens the adhesive when mixed with resin. It is the specific selection and combination of the epoxy and hardener components that determine the final characteristics and suitability of the epoxy coating for a given environment. Optimum levels of a hardener are used to formulate epoxy coatings. The ratio differs from product to product. The use of an improper hardener may result in an undercatalyzed or overcatalyzed product. In the present work hardener (HY951) is used. This has a viscosity of 10-20MPa at25°c.

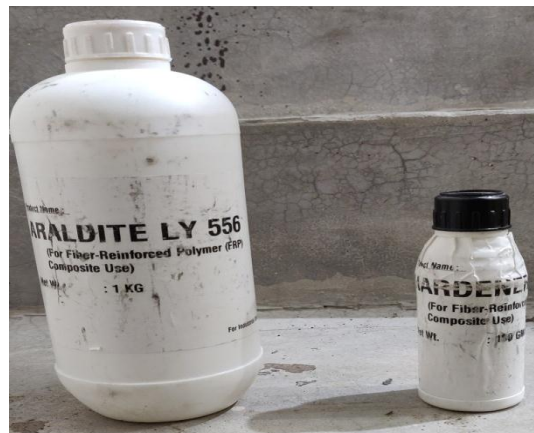


Fig.4-Epoxy and Hardener

PROCESSING

Hybrid composites were fabricated with the help of detachable closed mold of mild steel using hand lay-up technique. The silica gel was applied to the inner surface of mold plates to avoid sticking of polymer with the steel plates during curing.



Fig.5-Mould



Fig.6-Roller



Fig. 7- Wooden Frame



Fig 8- Composite Processing

The matrix was prepared by proper mixing of epoxy resin of Araldite LY556 grade and hardener HY951 in proportion of 10:1 as per manufacturer (Huntsman). Epoxy resin and hardener were properly mixed to reduce the air bubbles present in the liquid of resin and hardener. A wooden frame of dimension (250*260*5) was prepared. Flax and bamboo mat were washed with 10 % Sodium Hydroxide (NaOH) solution for 30 min and was cleaned with normal water until normal pH was attained. Now these fibers were dried by keeping them in sunlight for 8-10 hours. The liquid matrix was then uniformly spread on the inner surface of the mold and the resin was rolled by the steel roller to achieve the equal thickness of resin layer over the surface of the mold. Fiber mats were cut in equal size as of Frame cavity and placed over the layer of resin. Roller was again rolled over to remove any air bubble trapped within the layer. This process was repeated again and again till the pre-decided specifications. For each type of developed composite, the weight fraction of fiber was 28%. Load is applied with the help of C-Clamp. Composite is left for curing for 48 hours at room temp with humidity 55%. Final Composite Sheet is shown in Fig. 9&10 a.



Fig.9- Final Sheet (Top View)

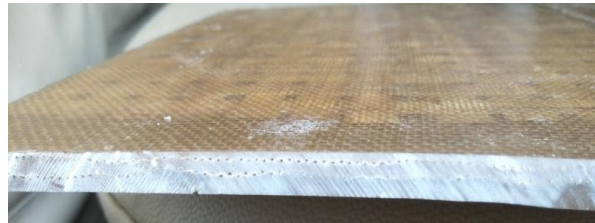


Fig.10(a)- Final Sheet (Side View)

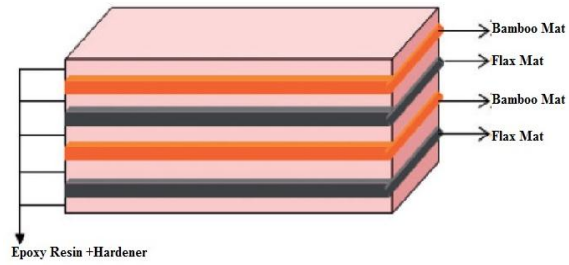


Fig 10 (b) systemic view of hybrid composite

III. TESTING AND RESULT

THERMO GRAVIMETRIC ANALYSIS (TGA)



Fig 11- Thermo gravimetric analyser

Thermo gravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss).

TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles (such as moisture). Common applications of TGA are materials characterization through analysis of characteristic decomposition patterns, studies of degradation mechanisms and reaction kinetics, (3) determination of organic content in a sample, and determination of inorganic (e.g. ash) content in a sample, which may be useful for corroborating predicted material structures or simply used as a chemical analysis. It is an especially useful technique for the study of polymeric materials, including thermoplastics, thermosets, elastomers, composites, plastic films, Fibres, coatings and paints

Thermo gravimetric analysis (TGA) relies on a high degree of precision in three measurements: mass change, temperature, and temperature change. Therefore, the basic instrumental requirements for TGA are a precision balance with a pan loaded with the sample, and a programmable furnace. The furnace can be programmed either for a constant heating rate, or for heating to acquire a constant mass loss with time.

Though a constant heating rate is more common, a constant mass loss rate can illuminate specific reaction kinetics. For example, the kinetic parameters of the carbonization of polyvinyl butyral were found using a constant mass loss rate of 0.2 wt. % min. Regardless of the furnace programming, the sample is placed in a small, electrically heated furnace equipped with a thermocouple to monitor accurate measurements of the temperature by comparing its voltage output with that of the voltage-versus-temperature table stored in the computer's memory. A reference sample may be placed on another balance in a separate chamber. The atmosphere in the sample chamber may be purged with an inert gas to prevent oxidation or other undesired reactions. A different process using a quartz crystal microbalance has been devised for measuring smaller samples on the order of a microgram (versus milligram with conventional TGA).

TGA Test Result

Thermo gravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss).

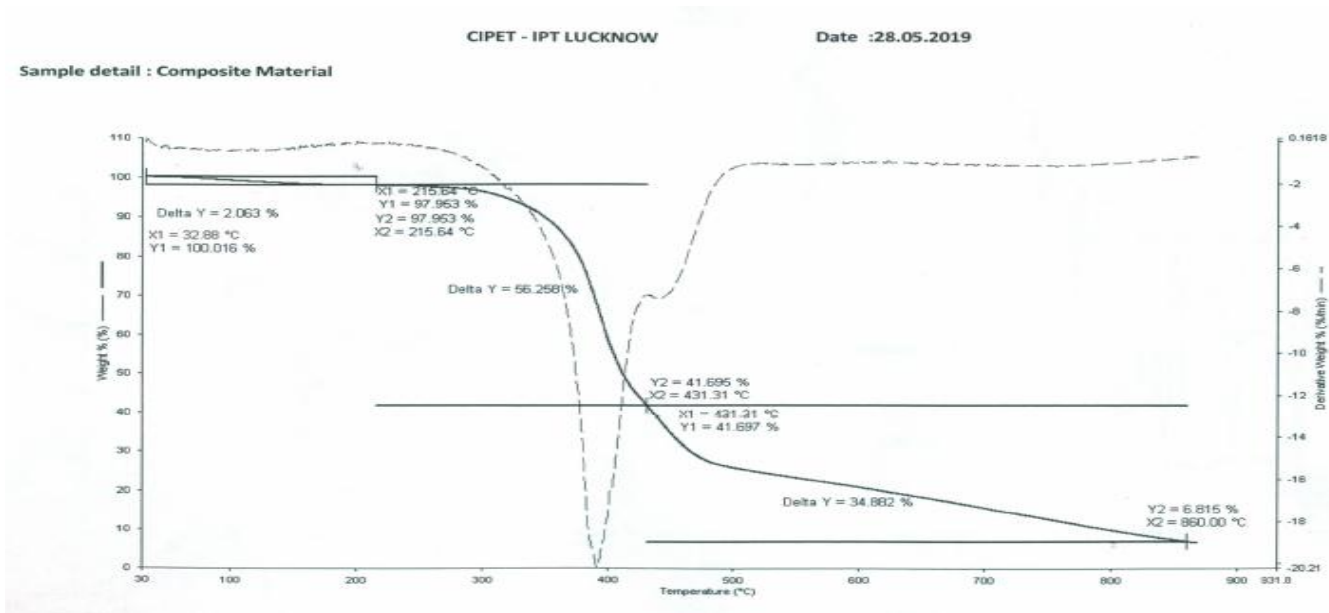


Fig 12- Graph Of TGA Test

Result from graph

Initial degradation temperature - 215.64°C

Final degradation temperature -860°C.

Amount of char left at 860°C -6.815%

DIFFERENTIAL SCANNING CALORIMETRY

Differential Scanning Calorimetry or DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. The technique was developed by E.S. Watson and M.J. O'Neill in 1962, and introduced commercially at the 1963 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The first adiabatic differential scanning calorimeter that could be used in biochemistry was developed by P.L. Privalov and D.R. Monaselidze in 1964. The term DSC was coined to describe this instrument which measures energy directly and allows precise measurements of heat capacity.



Fig 13-Differential Scanning Calorimetry

Detection of phase transitions

The basic principle underlying this technique is that when the sample undergoes a physical transformation such as phase transitions, more or less heat will need to flow to it than the reference to maintain both at the same temperature. Whether less or more heat must flow to the sample depends on whether the process is exothermic or endothermic. For example, as a solid sample melts to a liquid it will require more heat flowing to the sample to increase its temperature at the same rate as the reference. This is due to the absorption of heat by the sample as it undergoes the endothermic phase transition from solid to liquid. Likewise, as the sample undergoes exothermic processes (such as crystallization) less heat is required to raise the sample temperature. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters are able to measure the amount of heat absorbed or released during such transitions. DSC may also be used to observe more subtle physical changes, such as glass transitions. It is widely used in industrial settings as a quality control instrument due to its applicability in evaluating sample purity and for studying polymer curing.

Glass Transition Temperature (T_g) The Glass Transition Temperature (T_g) is one of the most important properties of any epoxy and is the temperature region where the polymer transitions from a hard, glassy material to a soft, rubbery material. As epoxies are thermosetting materials and chemically cross-link during the curing process, the final cured epoxy material does

not melt or reflow when heated (unlike thermoplastic materials), but undergoes a slight softening (phase change) at elevated temperatures. The ultimate Tg is determined by a number of factors: the chemical structure of the epoxy resin, the type of hardener and the degree of cure. Tg is usually measured using Differential Scanning Calorimetric (DSC): ASTM E1356, "Standard Test Method for Assignment of the Glass Transition Temperature by Differential Scanning Calorimetry". The format of the Tg scan is similar to that of a kinetic scan except that it is performed with a cured sample. Temperature is plotted on the X axis and the heat flow response on the Y axis (see above figure) As discussed previously, Tg is actually a temperature range, rather than a specific temperature. The convention, however, is to report a single temperature defined as the midpoint of the temperature range, bounded by the tangents to the two flat regions of the heat flow curve. For the epoxy material in the above figure, the reported Tg would be 75°C. Tg spans a temperature range, rather than occurring at a specific temperature, due to the cross-linked polymer chains having multiple degrees of freedom and modes of polymer chain movement in response to any applied thermal energy. The Tg value can also vary depending on its degree of cure.

DSC for neat Epoxy and BFBF hybrid composite.

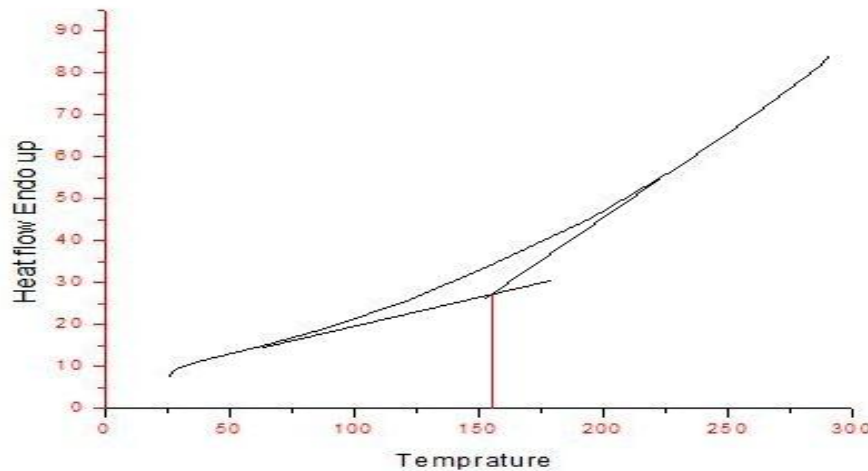


Fig 14- DSC for Epoxy

The Value of Tg for neat Epoxy (Glass transition Temperature) : 155°C

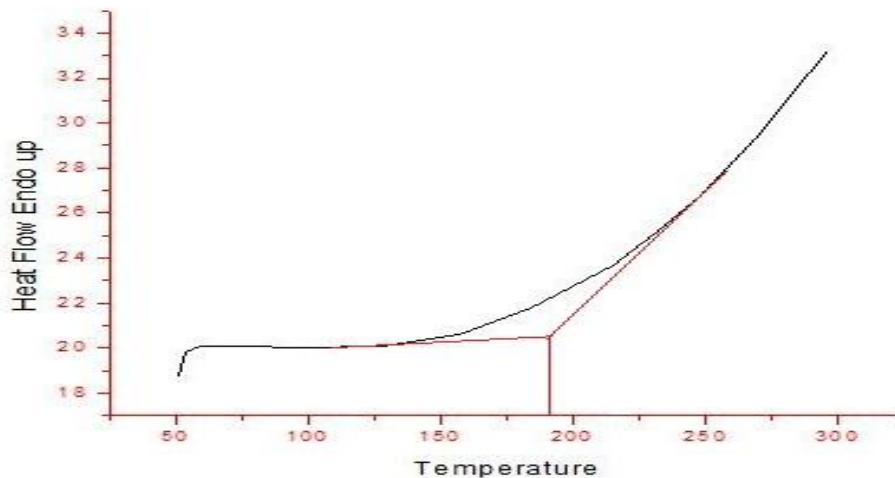


Fig 15- DSC for BFBF Hybrid Composite material

The Value of Tg BFBB hybrid Composite (Glass transition Temperature) : 190°C

Summary of DSC Test Result

S.No.	Material	Tg (Glass transition Temperature)
1	Epoxy	155°C
2	BFBB Hybrid composite	190°C

DSC test result:

Dsc test is performed by PERKIN-ELMER thermal analysis method . The glass transition temperature Tg was found to be 195°C. In this analysis the graph is drawn between Heat Flow Endo Up (on y-axis) and Temperature (on x-axis). And the value of Tg is obtained by tangent method. Initially specimen was held for 1 min. at 50°C and heated from 50°C to 300°C at 20°C /min.

IV. CONCLUSIONS

1. The significant thermal behaviour of the compound(hybrid) was determined from the initial degradation temperature which is taken as the temperature at which degradations started and the residue weight percentage denoted as char. From the thermograms it is apparent that sample undergoes a t-step degradation process .The first stage, thermal degradation process occurred in the temperature range of 215.64°C.-390°C. .The second stage degradation in the temperature range of 431°C.-860°C. and may be due to the compound decomposition of mat and matrix. Amount of char left at 860°C -6.815%
2. DSC test is performed on the hybrid composite and Tg temperature is found to be 195°C. This value of Tg Temperature is obtained by PERKIN-ELMER thermal analysis method. Hold for 1 min. at 50°C and heat from 50°C to 300°C at 20°C /min. The glass transition temperature (Tg) of HYBRID composite increases as compared to plain epoxy from 155°C to xxx due to reinforcement of Bamboo and Flax mat .

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