EFFECT OF HYGROTHERMAL TREATMENT ON THE TENSILE PROPERTIES OF HOT-PRESSED JUTE FIBRE COMPOSITES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUREMENT FOR THE DEGREE OF

Bachelor of Technology
In
Metallurgical and Materials Engineering

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CERTIFICATE

This is to certify that the thesis entitled, **"Effect of Hygrothermal treatment on the tensile properties of hot-pressed jute fibre composites"** submitted by **Mr Braj Sundar Das and Mr Gaurav Kumar Goyal** in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in **Metallurgical and Materials Engineering** at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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ACKNOWLEDGEMENT

ABSTRACT

The growing environment awareness demands the use of nature fibres as reinforcement materials in commercial. The natural fibres however are hydrophilic in nature and their composites undergo environmental degradation during service. Hygrothermal conditioning on as received jute fabric at different temperatures of hot-press have been studied in the present investigation. These composites are usually subjected to various loading conditions. Therefore, an attempt has been made to study the tensile properties of the composites. Fractography studies were carried out to study the fracture surface under SEM. It is noticed that the major mode of failure is due to fibre pullout and matrix cracking. The result from the hygrothermal studies shows the decrease in strength values of the composites on prolong exposure to humid environment.

CONTENTS

Chapter 1

INTRODUCTION

1. INTRODUCTION

1.1 Introduction

The most important types of natural fibres used in composite materials are flax, hemp, jute, kenaf, and sisal due to their properties and availability. Jute is an important bast fibre with a number of advantages. Jute has high specific properties, low density, less abrasive behaviour to the processing equipment, good dimensional stability and harmlessness. Jute textile is a low cost eco-friendly product and is abundantly available, easy to transport and has superior drapability and moisture retention capacity. It is widely being used as a natural choice for plant mulching and rural road pavement construction. The biodegradable and low priced jute products merge with the soil after using providing nourishment to the soil. Being made of cellulose, on combustion, jute does not generate toxic gases.

 Due to jute's low density combined with relatively stiff and strong behaviour, the specific properties of jute fibre can compare to those of glass and some other fibres.

Table 1.1: Properties of jute fibre in comparison with other fibres

Fibre	Density (g/cm³)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation At break (%)	Specific Tensile Strength (MPa/ g.cm⁻³)	Specific Young's Modulus (GPa/g.cm⁻³)
Jute	1.3-1.45	393-773	13-26.5	1.16-1.5	286-562	9-19
Flax	1.5	345-1100	27.6	2.7-3.2	230-773	18
Ramie	1.5	400-938	61.4-128	1.2-3.8	267-625	41-85
Sisal	1.45	468-640	9.4-22.0	3-7	323-441	6-15
Coir	1.15	131-175	4-6	15-40	114-152	3-5
E-glass	2.5	2000-3500	70	2.5	800-1400	28
S-glass	2.5	4570	86	2.8	1828	34

The natural fibres can be used to reinforce both thermosetting and thermoplastic matrices.

Thermosetting resins, such as epoxy, polyester, polyurethane, phenolic, etc. are commonly used today in natural fibre composites, in which composites requiring higher performance applications. They provide sufficient mechanical properties, in particular stiffness and strength, at acceptably low price levels. Compared to

compounds based on thermoplastic polymers, thermoset compounds have a superior thermal stability and lower water absorption. However, in the case of the demand for improved recycling and in combination with new long fibre reinforced thermoplastic (LFT) processing, thermoplastic polymers have been expected to substitute the thermoset polymers.

Considering the ecological aspects of material selection, replacing synthetic fibres by natural ones is only a first step. Restricting the emission of green house effect causing gases such as CO_2 into the atmosphere and an increasing awareness of the finiteness of fossil energy resources are leading to developing new materials that are entirely based on renewable resources.

The natural fibre composites can be very cost effective material for following applications:

- Building and construction industry: panels for partition and false ceiling, partition boards, wall, floor, window and door frames, roof tiles, mobile or pre-fabricated buildings which can be used in times of natural calamities such as floods, cyclones, earthquakes, etc.

- Storage devices: post-boxes, grain storage silos, bio-gas containers, etc.

- Furniture: chair, table, shower, bath units, etc.

- Electric devices: electrical appliances, pipes, etc.

- Everyday applications: lampshades, suitcases, helmets, etc.

- Transportation: automobile and railway coach interior, boat, etc.

- Toys

The reasons for the application of natural fibres in the automotive industry include [1, 2]:

- Low density: which may lead to a weight reduction of 10 to 30%.

- Acceptable mechanical properties, good acoustic properties.

- Favourable processing properties, for instance low wear on tools, etc.

- Options for new production technologies and materials.

- Favourable accident performance, high stability, less splintering.

- Favourable ecobalance for part production.

- Favourable ecobalance during vehicle operation due to weight savings.

- Occupational health benefits compared to glass fibres during production.

- No off-gassing of toxic compounds (in contrast to phenol resin bonded wood and recycled cotton fibre parts).

- Reduced fogging behaviour.

- Price advantages both for the fibres and the applied technologies.

Besides the advantages mentioned above, the natural fibre composites possess also some disadvantages. The main disadvantage is the poor compatibility between a hydrophobic polymer matrix and the hydrophilic fibres. This leads to the formation of weak interfaces, which result in poor mechanical properties of the composites. Other important disadvantages of the natural fibre composites are the high sensitivity of natural fibres towards water and the relatively poor thermal stability. Water absorption on composites is an issue to be considered since the water absorbed by the fibres in the composite could lead to swelling and dimensional instability and to a loss of mechanical properties due to
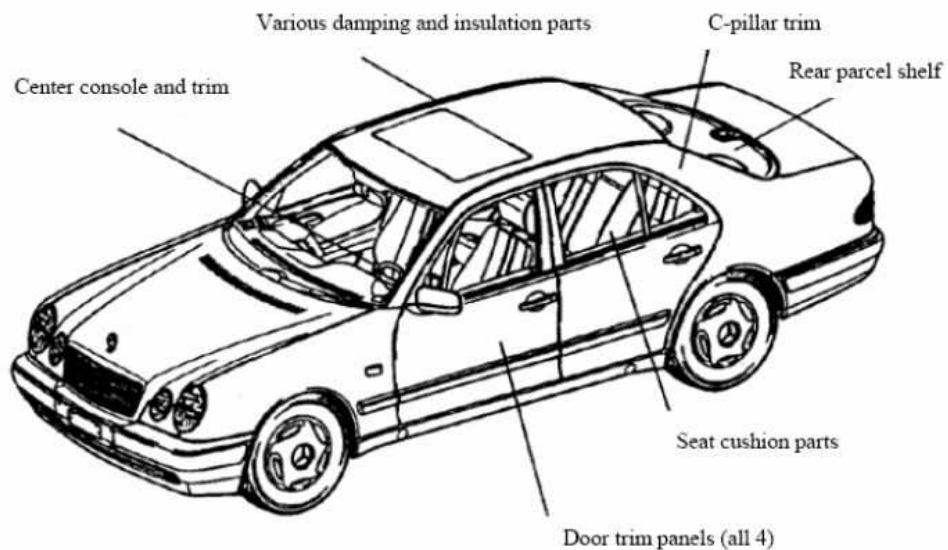

Fig.1.1. Plant fibre applications in the current Mercedes-Benz R-class [2]

the degradation of the fibres and the interface between the fibre and matrix [3, 4, 5, 6, 7, 8].

Chapter 2

LITERATURE REVIEW

2. LITERATURE REVIEW

2.1. Natural fibres and their chemical compositions

Climatic conditions, age and the digestion process influences not only the structure of fibres but also the chemical composition. Component mean values of plant-fibres are shown in Table 2.1. With the exception of cotton, the components of natural fibres are cellulose, hemi-cellulose, lignin, pectin, waxes and water soluble substances, with cellulose, hemi-cellulose and lignin as the basic components with regard to the physical properties of the fibres.

Table 2.1: Composition of different cellulose based natural fibres

	Cotton	Jute	Flax	Ramie	Sisal
Cellulose	82.7	64.4	64.1	68.6	65.8
Hemi-cellulose	5.7	12.0	16.7	13.1	12.0
Pektin	5.7	0.2	1.8	1.9	0.8
Lignin	-	11.8	2.0	0.6	9.9
Water soluble	1.0	1.1	3.9	5.5	1.2
Wax	0.6	0.5	1.5	0.3	0.3
Water	10.0	10.0	10.0	10.0	10.0

2.2. Mechanical properties of natural fibres

Natural fibres are in general suitable to reinforce plastics (thermosets as well as thermoplastics) due to their relative high strength and stiffness and low density (Table 3). The characteristic values for flax and soft-wood-kraft-fibres reach levels close to the values for glass-fibres, types E ("E" because of their early use in electronic applications and today the most typically used glassfibres reinforcing plastics in non-aggressive media). Nevertheless and also obvious in(Table 3), the range of the characteristic values, as one of the drawbacks for all natural products, is remarkably higher than those of glass-fibres, which can be explained by differences in fibre structure due to the overall environmental conditions during growth. Natural fibres can be processed in different ways to yield reinforcing elements having different

mechanical properties. The elastic modulus of bulk natural fibres such as wood is about 10 GPa. Cellulose fibre with

moduli up to 40 GPa can be separated from wood, for instance, by chemical pulping processes.Such fibres can be further subdivided by hydrolysis followed by mechanical disintegration into microfibrils with an elastic modulus of 70 GPa. Theoretical calculations of the elastic moduli of cellulose chains have given values of up to 250 GPa, however, there is no technology available to separate these from microfibrils.

Table 2.2: Mechanical properties of natural fibres as compared to conventional reinforcing fibres

Fibre	Density(g/cm3)	Elongation (%)	Tensile strength(MPa)	Young's modulus (GPa)
Cotton	1.5-1.6	7.0-8.0	287-597	5.5-12.6
Jute	1.3	1.5-1.8	393-773	26.5
Flax	1.5	2.7-3.2	345-1035	27.6
Hemp	-	1.6	690	-
Ramie	-	3.6-3.8	400-938	61.4-128
Sisal	1.5	2.02.5	511-635	9.4-22.0
Coir	1.2	30.0	175	4.0-6.0
Viscose(cord)	-	11.4	593	11.0
Soft wood kraft	1.5	-	1000	40.0
E-glass	2.5	2.5	2000-3500	70.0
S-glass	2.5	2.8	4570	86.0
Aramide(normal)	1.4	3.3-3.7	3000-3150	63.0-67.0
Carbon(standard)	1.4	1.4-1.8	4000	230.0-240.0

As in the case with glass-fibres, the tensile strength of natural fibres also depends on the test length of the specimens which is of main importance regarding reinforcing efficiency. The actual tensile strength of a single fibre is typically given for a test length of zero as in the case for glassfibres. The tensile strength of flax-fibres is significantly more dependent on the length of the fibre than for the case of glass-fibres. Compared to this, the tensile strength of pineapple-fibres is less dependent on the length, while the scatter of the measured values for both is located mainly in the range of the standard deviation. This dependence, strength vs. test length, could be

seen as degree or the homogenity or amounts of defects of a fibre or a material in general. Hydrophilic nature is a major problem for all cellulose-fibres if used as reinforcement in plastics.

The moisture content of the fibres, dependent on content of non-crystalline parts and void

content of the fibre, amounts up to 10 wt. % under standard conditions. The hydrophilic nature of natural fibres influences the overall mechanical properties as well as other physical properties of the fibre itself.

2.2. Jute fibres

2.2.1. Properties of jute fibres

The need for using jute fibres in place of the traditional glass fibre partly or fully as reinforcing agents in composites stems from its:

• Lower specific gravity (1.29) and higher specific modulus (40 GPa) of jute compared

with those of glass (2.5 & 30 GPa respectively),

• Jute fibre is 100% bio-degradable.

• It has high tensile strength, low extensibility, and ensures better breathability of fabrics,

• It has good insulating and antistatic properties, as well as having low thermal conductivity.

2.2.2. Advantages of jute fibres

Apart from much lower cost and renewable nature of jute, much lower energy requirement for the production of jute (only 2% of that for glass) makes it attractive as a reinforcing fibre in composites. Jute fibre is recyclable and thus environment friendly starting from the seed to expired fibre, as the expired fibres can be recycled more than once. Jute fibres are easily available and these are environment friendly.

2.2.3. Uses of jute fibres

Jute has entered various diversified sectors, where natural fibres are gradually becoming better substitution. Among these industries are paper, celluloid products (films), nonwoven textiles (for car interiors and other uses), composites (pseudo-

wood), and geotextiles. The jute composites may be used in everyday applications such as lampshades, suitcases, paperweights, helmets, shower and bath units. They are also used for covers of electrical appliances, pipes, post-boxes, roof tiles, grain storage silos, panels for partition & false ceilings, bio-gas containers, and in the construction of low cost,

mobile or pre-fabricated buildings which can be used in times of natural calamities such as floods, cyclones, earthquakes, etc.

2.2.4. Limitation of jute fibre composites

Unfortunately, all mechanical and other physical properties of natural fibres are influenced by their growing conditions, fibre processing technique and, as for other fibre types, by the fineness of the fibre and sample test-length. Although, as with most of the other plant-based natural fibres, cellulose forms the main structural component of jute, the non-cellulosic components e.g., lignin and hemicellulose, also play an important part in determining the characteristic properties of the fibres.

There is, however, a major drawback associated with the application of jute fibres for reinforcement of resin matrices. Due to presence of hydroxy and other polar groups in various constituents of jute fibre, the moisture uptake is high (approx. 12.5% at 65% relative humidity & 20° C) by dry fibre and 14.6% by wet fibre. All this leads to (i) poor wettability with resin and (ii) weak interfacial bonding between jute fibre and the relatively more hydrophobic matrices. Environmental performance of such composites is generally poor due to delamination under humid conditions. With increase in relative humidity upto 70%, the tenacity and Young's modulus of jute increases but beyond 70%, a decrease is observed.

2.5. Polyester

Cure at room or elevated temperature. It has good chemical resistance.

Limitations:- emission of styrene, shrinkage on curing (7-10%)

The term polyester covers a very large chemical family of which "unsaturated resins" covering orthophthalic, isophthalic, vinyl esters and blends form the largest single group of fibre reinforced thermosets. They are usually manufactured by reacting together dihydric alcohols (glycols) and dibasic organic acid, either or both of which contain a double-bonded pair of C atoms. By elimination of water between the acids and glycols, ester linkages are formed, producing a long chain molecule comprising

alternate acid and glycol units. The polymer chain is dissolved in a reactive organic solvent, usually styrene monomer or methyl methacrylate, vinyl toluene or alpha-methyl styrene. With heat and a chemically activated free radical initiation, the polyester and the reactive diluents crosslink to form 3-D non melting network. The reaction is in a kettle at 170-200^{0}C with components in equal molecular ratio and water eliminated.

Polyester resins are cured by organic peroxides which initiate a free radical copolymerization reaction. This can occur at room temperature, under heat (60-90^{0}C) or by UV or visible light radiation. The catalyst system comprises organic peroxides (initiators) which are activated by accelerators or promoters. The resin begins to cure as soon as the initiator is added, the speed of reactivity depending on the temperature, resin and catalyst reactivity.
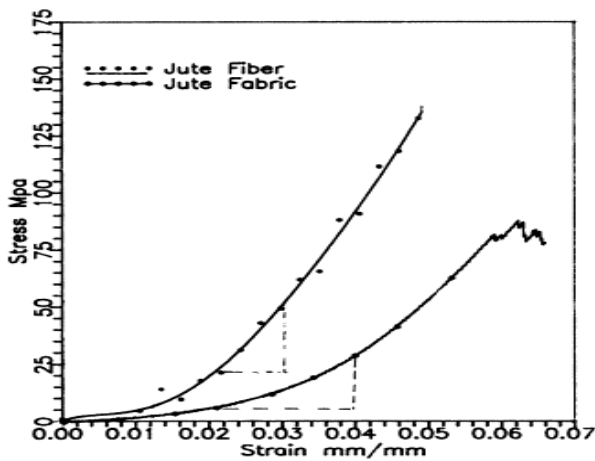
2.6. Tensile Properties

Fig. 1. Stress—strain response of jute fibre and fabric.
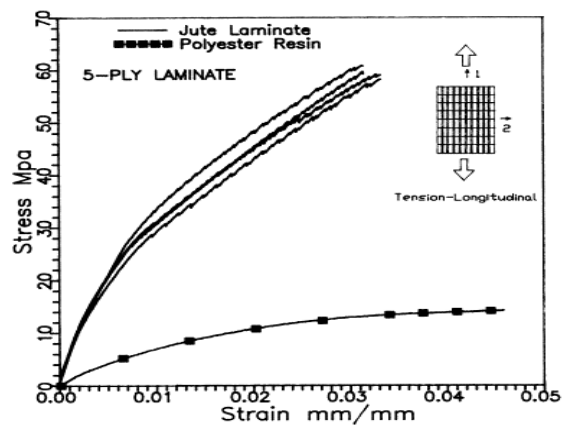
Fig. 2. Tensile stress—strain response of jute-reinforced polyester composites and resin.

Fig. 2.1: Tensile Properties of jute fibre, fabric and composites

Fig. 1 shows the stress–strain diagram for jute fibre and jute fabric. Jute fibre exhibits stiffer characteristics as compared to jute fabric. This is due more to initial stretching of the fabric than the nature of the fibre. For both fibre and fabric, the curves are initially horizontal due to the stretching effect caused by removing slack from the system. Strands in the fabric break at different times as each fibre can stretch independently and break individually when reaching their breaking stress. The failure mode is by progressive breaking of the fibres. It has been observed that strands in the

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fabric begin to fail from the centre of the fabric and propagate width-wise on either side. The ultimate tensile strength and the tangent modulus of elasticity after initial stretching of the 'as received' jute fibre is found to be 120 MPa and 3.75 GPa; corresponding values for jute fabric are 85 MPa and 0.8 GPa. The variation in the values of strengths may be due to: (i) the assumption that the cross-sectional area of each yarn is circular, (ii) the difference in the strength of individual fibres in the fabric arising out of process defects.

The tensile strength of a composite material is mainly dependent on the strength and modulus of fibres, the strength and chemical stability of the matrix and the effectiveness

of the bonding strength between matrix and fibres in transferring stress across the interface [10]. Fig. 2 shows the stress–strain diagram for four jute laminates and a polyester resin. The initial linear portion of the jute laminate curves shows the elastic behaviour of the composite. The deviation from linearity is an indication of the beginning

of initial matrix cracking, and the first major change in slope in the curve is the sign of a major crack in the matrix or the beginning of fibre failure. The first fibre failure occurs at a stress level of 26 MPa (approx.) and the corresponding resin stress is 5.5 MPa for the same amount of strain. The rest of the drops in the curves are indications of progressive failure of fibres as the applied load increases, and the end of the curve represents the ultimate stress which is due to fibre fracture and may be fibre pull-out. However, the failure mode exhibits breakage and little pull-out of fibres. The average values of ultimate tensile strength, initial tangent modulus and Poisson's ratio for these composites are 60 MPa, 7 GPa and 0.257, respectively, and corresponding values for polyester resin are 12.1 MPa, 1.4 GPa and 0.38. The values obtained by the rule of mixture are 63 MPa and 1.12 GPa. The difference in the initial tangent modulus obtained from experiment and rule of mixture is due to initial stretching and the nature of the fibres.
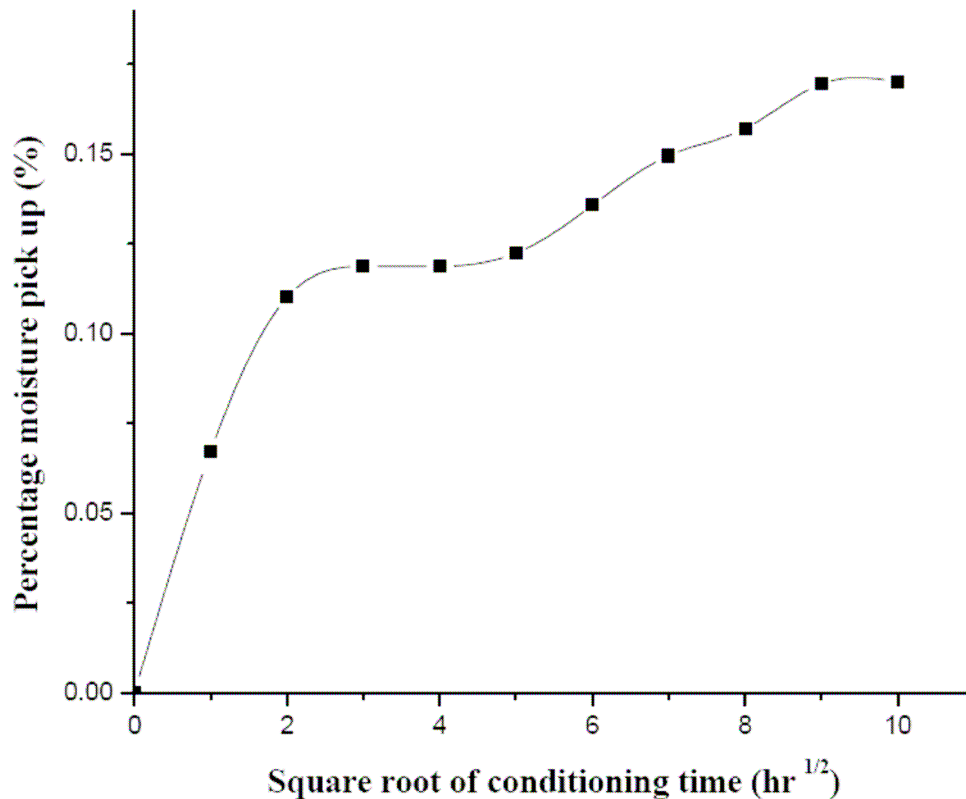
2.7. Hygrothermal Effects:-

Fig. 2.2: Percentage moisture pickup vs square root of conditioning time

The amount of moisture absorption by FRP composites in humid conditions is a function of time and temperature [7]. Fig. 1 is representative of the moisture absorption kinetics in FRP composites. The first region shows a marked linearity indicating Fickian absorption, where the moisture is absorbed according to Fick's second law of diffusion. Following this a saturation level is achieved, which is indicated by the horizontal region of the curve. The amount of moisture absorbed depends on the partial pressure of the moisture in the surrounding and the moisture in the composite. Once a temporary equilibrium is reached, a saturation region is attained. Prolonged exposure to hygrothermal environment results in the third stage of the curve clearly indicating non-Fickian moisture absorption kinetics. Fickian and non-Fickian moisture absorption kinetics have been explained by Li-Rong Bao and coworkers [8]. The diffusion properties of the interface may be different from those of the bulk matrix probably due to the formation of a boundary layer. When the fraction

of matrix in the interface region is significant, moisture transport may be considerably affected.

Surface absorption and diffusion through the matrix is the primary mechanism for moisture pick up in most of the well fabricated composite materials during the initial period of exposure [10]. The composite materials contain cracks and micro voids in the matrix. These are formed during the polymerization of the matrix. The free polymer chains get entangled with each other and create these micro voids in the matrix [11]. These micro voids are the major sites for the moisture pick ups in the composite materials, especially during the initial period. Gradually when the moisture diffuses inside the matrix, it starts to interact chemically with the polymer.

This is marked by a chemical phenomenon called matrix hydrolysis [7]. When this mechanism is spread through out the composite in a wider range, then the over all chemistry of the polymer is significantly affected. Thus the adhesion between the fiber and the matrix is compromised stupendously, causing the de-bonding at the fiber matrix interface [12]. Finally, the composite fails completely, when its constituents separates out from each other. In other words, the composite fails when the interface between the fiber and the matrix collapses completely. Thus the failure of the composite deals with surface chemistry of the fiber-matrix interface due to physical, chemical and physico-chemical changes of the matrix during its service conditions.

The exposure of composite at higher temperature enhances the matrix cracking due to mismatch in thermal strain. There is also a possibility of polymerization in them. The crosslinking during the polymerization process results in an increased number of micro voids in the composites. Consequently, there exists more sites for moisture pick ups. Hence samples subjected to 64 hours of treatment show less resistant to failure than the 4 hours conditioned samples.

Exposure to elevated temperature can result in degradation of mechanical properties, cracking, chalking and flaking of polymers [8]. The first form of damage in laminated composite is usually matrix microcraks. These microcraks are transverse to the loading direction and are thus called transverse cracks. Matrix microcracks cause degradation in properties in composite laminates and also act as precursors to other forms of damage leading to laminate failure. Delamination is a critical failure mode in composite structure. The interfacial separation caused by the delamination may lead to premature buckling of laminates, excessive intrusion of moisture and stiffness

degradation. While in some cases a delamination may provide stress relief and actually enhance the performance of a composite component.
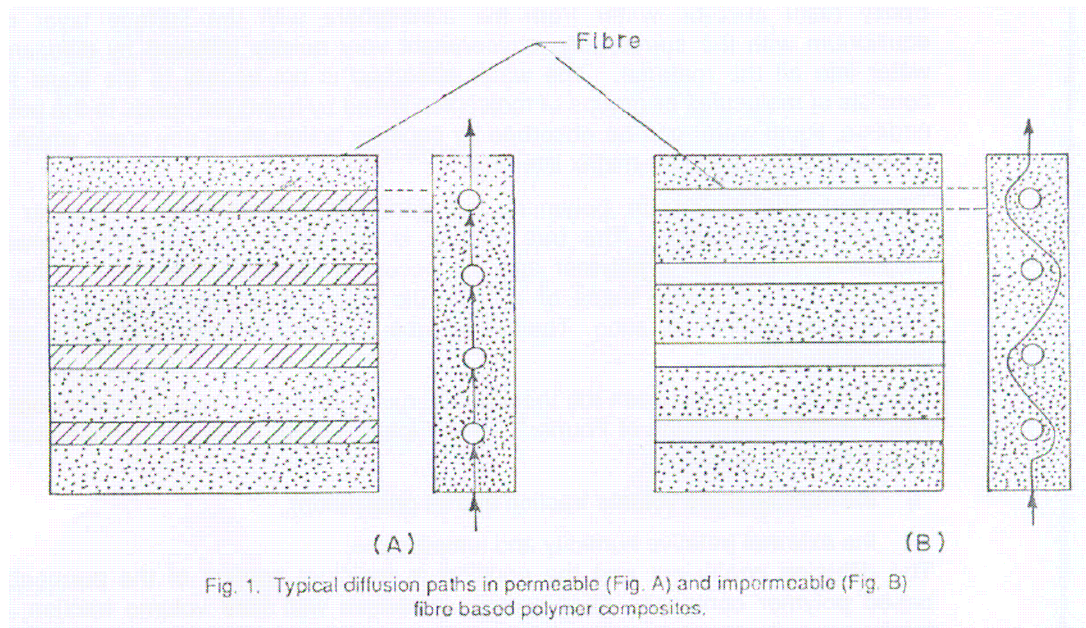

Fig. 1. Typical diffusion paths in permeable (Fig. A) and impermeable (Fig. B) fibre based polymer composites.

Fig. 2.3: Diffusion paths for water adsorption in composites

Chapter **3**

EXPERIMENTAL WORK

3. EXPERIMENTAL WORK

■ 3.1. Preparation of composite

- ☐ By hand laying technique
- ☐ Resin=60% of fabric weight
- ☐ Hardener=2.6% of resin weight
- ☐ Accelerator=2% of resin weight
- ☐ The mixture of polyester resin with the given weight % of hardener and accelerator was applied in between as received jute fabric pieces to get alternate layers of resin matrix and jute fabric reinforcement.
- ☐ A hot–plate setup was constructed and the laminates were hot pressed at three different temperatures 60, 80 and 100^0C under 2 ton load for 8 minutes with the help of a hydraulic press to enhance the rate of curing and bring out uniform distribution of resin mix.
- ☐ The green composite is then left out to atmosphere for 48 h under a load of 8kg for avoiding relaxation.
- ☐ The specimens were then cut from the laminate for tensile test with the help of hacksaw according to the specific standard.

■ 3.2. Hygrothermal Treatment

The specimens were hygrothermally conditioned in a humidity cabinet where the conditions were maintained at a temperature of 60^0C and 95% relative humidity (RH). The humidity cabinet had an inbuilt thermometer for temperature and hygrometer for relative humidity measurements. The temperature variation was maintained between 0-0.5^0C whereas the RH variation was allowed in the 0-1% range. The composite laminates were placed on perforated trays. The hygrothermal conditioning was carried out for different lengths of time ranging from four to sixty-four hours.

■ 3.3. Tensile Testing

Tensile studies were carries out to determine the as tensile yield strength, tensile strength at break (ultimate tensile strength), tensile modulus (Young's modulus), elongation at yield and break and energy to yield and break point.
Tensile tests were performed in an Instron 1195 machine in accordance with a specific standard.
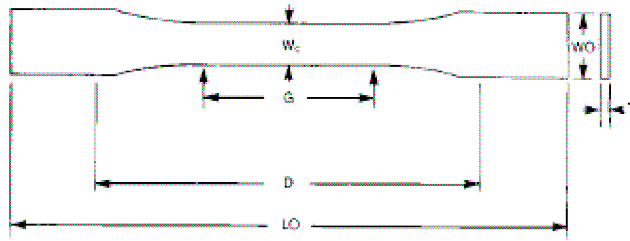
Fig.3.1: Dimensions of a tensile specimen

w_c- Width of narrow section = 11mm,

w_o- Width overall = 25mm,

T- Thickness = 3 – 4mm,

G- Gauge length = 40mm,

Lo- Length overall = 120mm,

D- Distance between grips = 70mm.

Chapter 4

RESULTS
AND
DISCUSSION

4. RESULTS & DISUSSION

The tensile test results for samples hot-pressed at 60, 80 and 100^0C and subjected to hygrothermal treatment for 4, 16 and 64 hours is given as follows. A set of samples, hot-pressed at different temperatures, was not given any hygrothermal treatment. Their tensile values were obtained and the results compared.

Longitudinal Tensile Stress of laminate pressed at room temperature was found to be 39.68 MPa

Temperature of hot press = 60^0C

Sl. No.	Duration of Hygrothermal Treatment (hrs)	UTS (MPa)	Displacemnt at break (mm)	Energy to yield point (J)	Dry Weight (in g)	Weight after treatment (in g)	%age increase
1	0	79.04	1.208	1.918			
2	0	82.58	1.080	2.028			
3	0	48.96	0.998	0.988			
4	4	46.47	1.190	.1469	8.72	8.92	2.29
5	4	85.31	1.739	.7766	10.83	11.01	1.66
6	4	79.31	1.309	.5678	9.17	9.45	3.05
7	4	78.60	1.201	.6510	8.38	8.66	3.34
8	4	80.79	1.483	.7263	10.18	10.46	2.75
9	16	43.38	1.291	.1454	8.37	8.77	4.78
10	16	81.46	1.382	.3926	10.00	10.34	3.40
11	16	75.68	1.501	.3509	9.56	9.91	3.66
12	16	67.15	1.208	.3482	9.92	10.30	3.83
13	16	78.33	1.684	.4978	8.26	8.70	5.33
14	64	76.13	1.135	.9796	9.30	9.68	4.09
15	64	71.06	1.391	.3554	9.36	9.72	3.85
16	64	66.02	1.236	.3433	8.93	9.35	4.70
17	64	71.10	1.456	.4433	10.85	11.36	4.70

Table 4.1: Experimental results obtained for samples hot-pressed at 60^0C and subjected to hygrothermal treatment for 0,4,16 and 64 hours

In the above table, data obtained from sample no. 3, 4 and 9 were not considered for analysis as these values showed deviation from the rest of the data.

Temperature of hot press = 80^0C

Sl. No.	Duration of Hygrothermal Treatment (hrs)	UTS (MPa)	Displacement at break (mm)	Energy to yield point (J)	Dry Weight (in g)	Weight after treatment (in g)	%age increase
1	0	83.24	1.142	1.951			
2	0	84.69	1.087	2.045			
3	4	76.82	1.421	.5421	10.22	10.42	1.96
4	4	87.88	1.529	.5378	9.65	9.86	2.18
5	4	90.51	1.595	1.074	10.41	10.66	2.40
6	4	87.06	1.437	.9414	10.29	10.53	2.33
7	16	84.5	1.245	.5893	9.92	10.23	3.13
8	16	77.5	1.505	.5896	9.35	9.67	3.42
9	16	95.55	1.364	.6921	9.17	9.52	3.82
10	16	81.59	1.382	.5962	9.76	10.15	4.20
11	16	78.99	1.410	.8159	10.02	10.45	4.29
12	64	63.84	1.144	.2840	9.39	9.74	3.73
13	64	100.20	1.474	.4356	9.39	9.75	3.83
14	64	58.71	1.545	.3926	10.25	10.67	4.09
15	64	72.08	2.035	.8298	10.43	10.93	4.71
16	64	51.25	0.989	.3672	9.64	10.07	4.46

Table 4.2: Experimental results obtained for samples hot-pressed at 80^0C and subjected to hygrothermal treatment for 0,4,16 and 64 hours

Temperature of hot press = 100^0C

Sl. No.	Duration of Hygrothermal Treatment, t (in hrs)	UTS (MPa)	Displacemnt at break (mm)	Energy to yield point (J)	Dry Weight (in g)	Weight after treatment (in g)	%age increase
1	0	75.91	1.044	.8510			
2	0	79.69	1.117	.9599			
3	0	62.68	0.879	.6166			
4	0	65.69	0.732	.9234			
5	4	63.79	1.117	.5866	8.40	8.63	2.74
6	4	61.57	1.199	.4484	9.88	10.11	2.33
7	4	75.63	1.071	.6597	8.49	8.72	2.71
8	4	66.68	1.275	.5260	9.04	9.30	2.88
9	4	67.45	1.300	.6645	9.89	10.14	2.53
10	16	64.34	1.401	.2984	9.47	9.90	4.54
11	16	66.01	1.080	.3877	8.73	9.17	5.04
12	16	51.96	1.199	.3326	9.54	9.96	4.40
13	16	57.71	1.318	.2187	8.46	8.89	5.08
14	16	73.08	1.437	.6254	8.93	9.31	4.26
15	16	65.27	1.391	.4108	10.08	10.52	4.37
16	64	51.89	1.172	.3305	11.04	11.45	3.71
17	64	46.55	1.227	.2460	10.97	11.39	3.83
18	64	70.57	1.748	.6780	9.10	9.54	4.84
19	64	77.56	1.556	.4702	9.55	10.03	5.03

Table 4.3: Experimental results obtained for samples hot-pressed at 100^0C and subjected to hygrothermal treatment for 0,4,16 and 64 hours

4.1. Deductions from Stress-Strain curve:

(i)   (4 hrs):- The slope of the plot is high. That means the samples are more elastic in nature. The load is bore by the fibre rather than the matrix. From the drop in 80^0C, it is clear that the fibre bearing the load suffers from pullout and then fractures. 80^0C shows high strength and ductility, 100^0C shows progressive failure.

(ii)   (16 hrs):- Most of the plots show brittle behavior. The termination of the plot at a point signifies that the bonding between the matrix and fibre is

weak and the fracture is due to the breakage of fibres embedded in the matrix. This may be either due to matrix fracture or due to fibre pullout. It may also be due to fibre breakage when the bonding between the fibre-matrix interfaces is strong.

The series of ups and down in the curve may indicate successive fibre failure. A large load is required to break a single fibre, then the crack propagates through the matrix, when a drop in stress is seen. The remaining fibres then share the load and another fibre fracture occurs. This may be the case when the bonding between the fibre and matrix is strong enough to transfer the load, serving its purpose effectively.

(iii)   (64 hours): The curve is seen to rise steadily until it fractures. The initial linear portion of the plot shows the elastic nature of the composite. The nature of the graph shows the failure is brittle in nature and there is a sharp breaking point.

The drop in stress at the point of fracture may indicate fibre pullout as for the same level of strain the value of stress is decreasing. Another reason may be that it is due to the fracture of the matrix. The matrix may have become stiffer due to loss of toughness and thus unable to transfer the load from one fibre to another. The crack formed at the interface would have propagated through the matrix due to the loss of crack-blunting property of the matrix.

Samples hot pressed at 80^0C show progressive fracture of fibres.

Table 4.4: Average values of Longitudinal Tensile Stress corresponding to the first fibre failure

Temp. of press Time	60^0C	80^0C	100^0C
0 hrs	80.81 MPa	83.97 MPa	70.99 MPa
4 hrs	81.00 MPa	85.57 MPa	67.02 MPa
16 hrs	75.66 MPa	83.63 MPa	63.06 MPa
64 hrs	71.08 MPa	69.22 MPax	61.64 MPa

Table 4.5:

Average values of Displacement corresponding to first fibre failure

Temp. of press Time	60^{0}C	80^{0}C	100^{0}C
0 hrs	1.144 mm	1.115 mm	0.943 mm
4 hrs	1.433 mm	1.495 mm	1.192 mm
16 hrs	1.444 mm	1.381 mm	1.304 mm
64 hrs	1.305 mm	1.437 mm	1.199 mm

Table 4.6:

Average value of Energy at yield point

Temp. of press Time	60^{0}C	80^{0}C	100^{0}C
0 hrs	1.973 J	1.998 J	0.838 J
4 hrs	0.680 J	0.774 J	0.577 J
16 hrs	0.397 J	0.657 J	0.379 J
64 hrs	0.530 J	0.462 J	0.432 J

Fig. 4.1: Average Longitudinal Tensile Stress of samples hot-pressed at 60, 80 and 100^{0}C
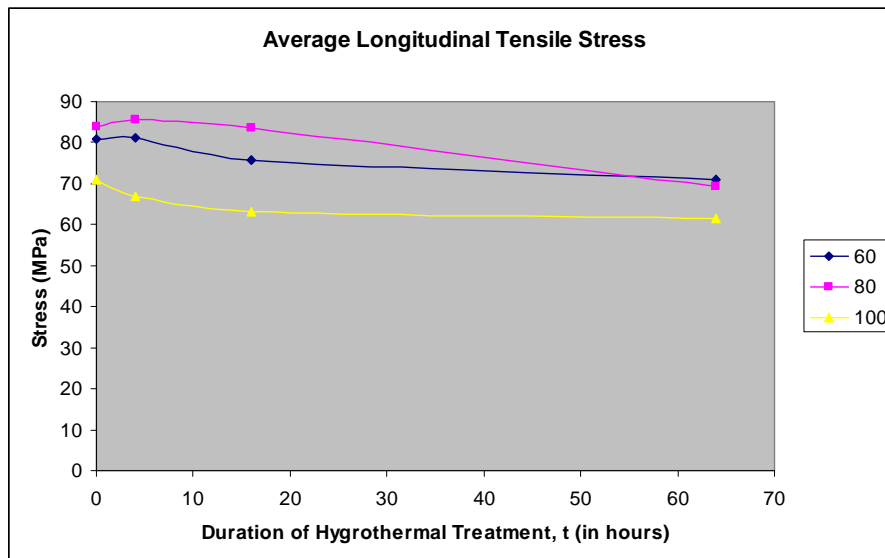
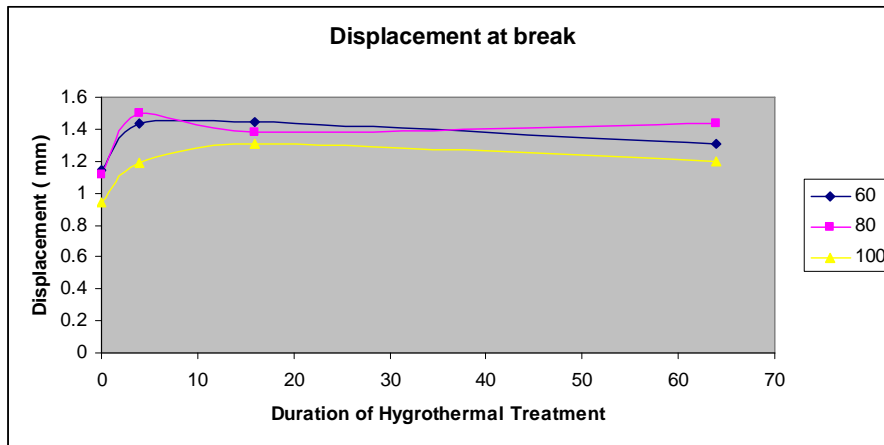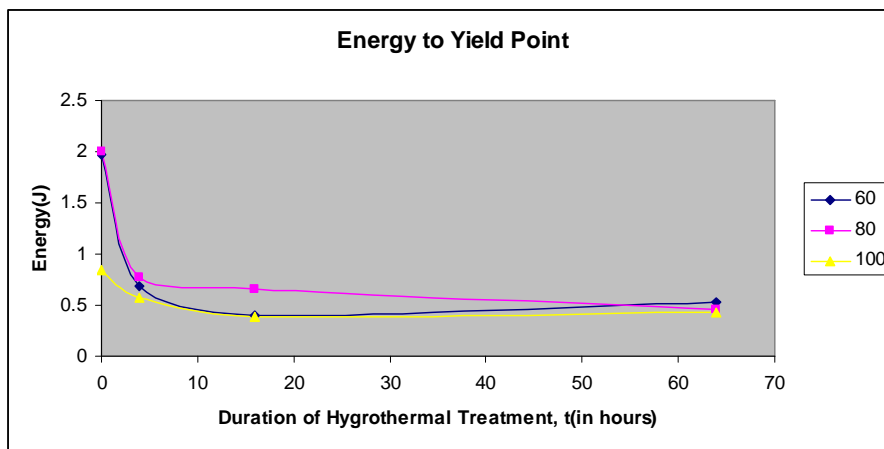Fig. 4.2: Displacement at break of samples hot-pressed at 60, 80 and 100^{0}C

Displacement at break

Fig. 4.3: Energy to yield point of samples hot-pressed at 60, 80 and 100^{0}C

Energy to Yield Point

The average longitudinal stress value is seen to decrease with the duration of hygrothernal treatment. There is a slight increase in the value of stress when the samples hot-pressed at 60 and 80^{0}C are subjected to 4 hours of hygrothermal conditioning which is in contrast to the decrease for samples hot-pressed at 100^{0}C. With further exposure for long durations the ultimate tensile strength is found to a decrease in all the three cases. The increase in values for 60 and 80^{0}C hot pressed sample may be because of cross-linking of the UP resin facilitated by an exposure to constant temperature at 60^{0}C. This may have lead to an increase in the bonding between the fibre and matrix at the interface. It can also be seen from the energy-time plot that there is a marked loss in energy to yield point for the first 4 hours of hygrothermal treatment. In the case of hot-pressing at 100^{0}C, the samples may have

undergone complete curing and all the sites for cross-linking must have participated in the reaction leaving behind no empty sites for additional polymerization. The exposure to 60^0C would have no effect then. The matrix may have become stiffer due to loss of toughness.

The other explanation for the increase in the strength values for short hygrothermal exposure may be due to relieve of curing stresses in the matrix. Adsorption of moisture by the fibre and matrix components in the matrix would cause swelling stresses to develop, which are of opposite nature to that of curing stresses. The moisture absorbed as water molecules may indulge in hydrogen bonding with the fibre as well as the matrix. This may enhance the strength by further polymerization.

Increase in exposure time leads to low strength values. The adsorbed moisture interacts chemically with the matrix. The first water molecules are adsorbed directly into the hydrophilic groups at the fibre and after that the other water molecules are attracted either other hydrophilic groups or they may form further layers on top of the water molecules already adsorbed. This leads to poor adhesion with the resin matrix, ultimately causing debonding. The fracture depends on the strength of the fibre-matrix interface. Also it depends on the toughness and strength of the matrix. The reduced values of displacement at break and energy at yield point may be due to plasticization of matrix because of moisture uptake. This helps in the propagation of fracture and accounts for the low strength values.

The fracture as observed in the composite samples can be classified into the following types:-

(i)   By fibre pullout only

(ii)   By fibre pullout and tearing of the fibres due to adhesion mechanisms induced by reactions at the fibre-matrix interface.

(iii)   By tearing of the fibres only when there is no pullout as the fibres are completely impregnated by the matrix

(iv)   By shear yielding of matrix and tearing from the fibre.

4.2. Hygrothermal Behaviour:-

Table 4.7: %age weight gain after hygrothermal treatment

Temp. of press Time	60^0C	80^0C	100^0C
4 hrs	2.70 g	2.22 g	2.64 g
16 hrs	4.06 g	3.77 g	4.39 g
64 hrs	4.34 g	4.16 g	4.35 g

Fig. 4.4: Hygrothermal Diffusion of hot-pressed samples for different periods of exposure
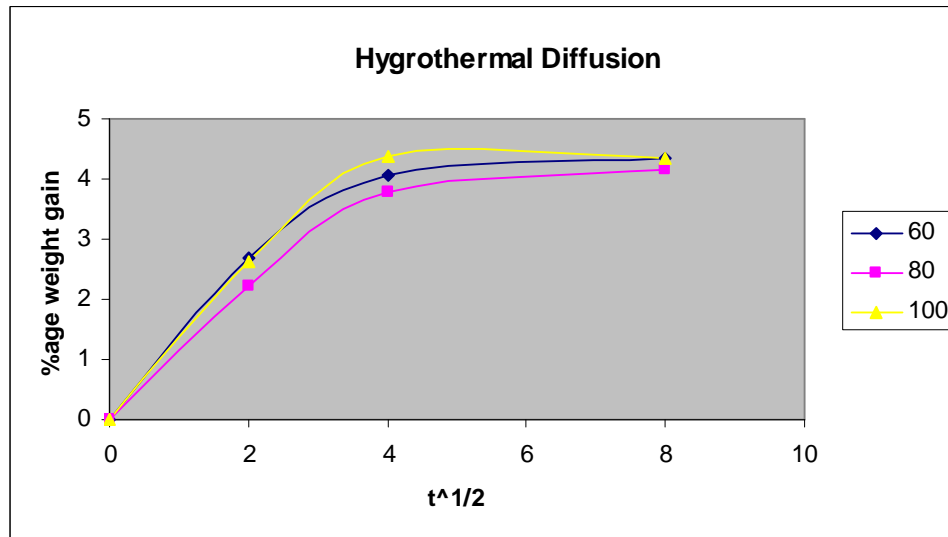
The diffusion process of water in jute FRP composites followed a Fickian model. This is to be expected, as the hydrophilic moieties on the fibre surface act as passageways for the water entry. Unexpectedly, the water uptake does not deteriorate the mechanical composite properties when the time of exposure was for 4 hours. In contrast, there is even a slight improvement in the properties for the aged sample. This is explained as a result of the better interfacial contact due to the swelling of the jute fibre, which outweighs the loss of adhesion due to the breakup of hydrogen bonds in presence of water.

The moisture was first absorbed at the voids or pores present in the matrix when the rate of moisture in take was higher. Thus there is a fall in the rate in the absorbance,

as the voids available for moisture pick up reduces down. The rate continues to fall until there are no more defects to absorb moisture. By then, the moisture thus absorbed starts to interact with the matrix both chemically and physico-chemically. This leads to the spreading of moisture through the matrix, making the path ways for newer sites for moisture pick ups. Hence, during this, the rate gets enhances again till the newer sites are completely occupied with the moisture. This cycle of absorption, dissolution and diffusion continues till most of the matrix is soaked up by the moisture and the moisture interact with the fiber-matrix interface. Absorbed moisture can damage the interface over time by interrupting the hydrogen bonding within the matrix and fiber, thereby weakening the interface. Furthermore, stresses created by swelling can be very high and may eventually damage the interface. The absorption of moisture causes plasticization of the resin to occur with a concurrent swelling and lowering the glass transition temperature of the resin. This adversely affects the fiber-matrix adhesion properties, resulting debonding at fiber/matrix interfaces, micro cracking in the matrix, fiber fragmentations, continuous cracks and several other phenomena that actually degrades the mechanical property of the composites.

It can be seen from the graph that the lowest %age weight gain is for the samples hot-pressed at 80^0C. It is also seen from the earlier plots that samples hot-pressed at 80^0C show maximum breaking strength values, while it is low for samples ho-pressed at 60 and 100^0C. Thus high moisture uptake results in lowering the resistance to fracture.

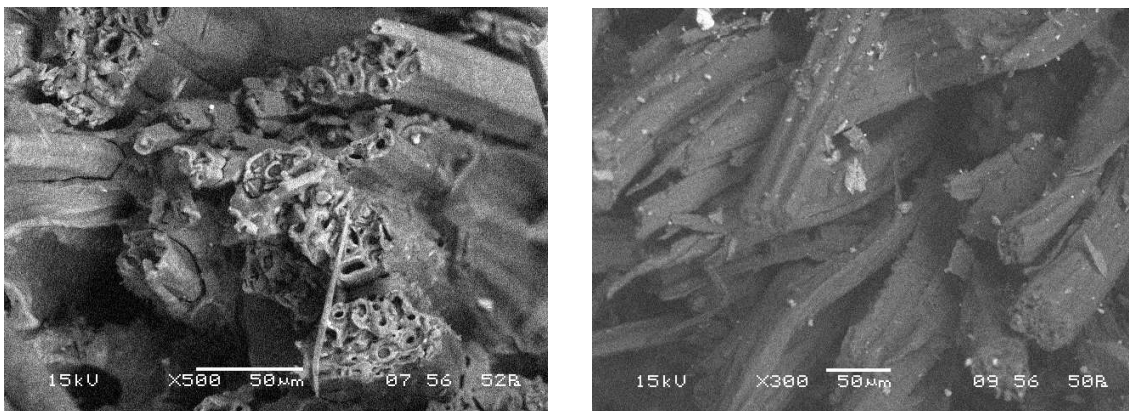## 4.3. Fractography by SEM Analysis :-

Fig 4.5: Fractography of untreated and Hygrothermally treated composite.

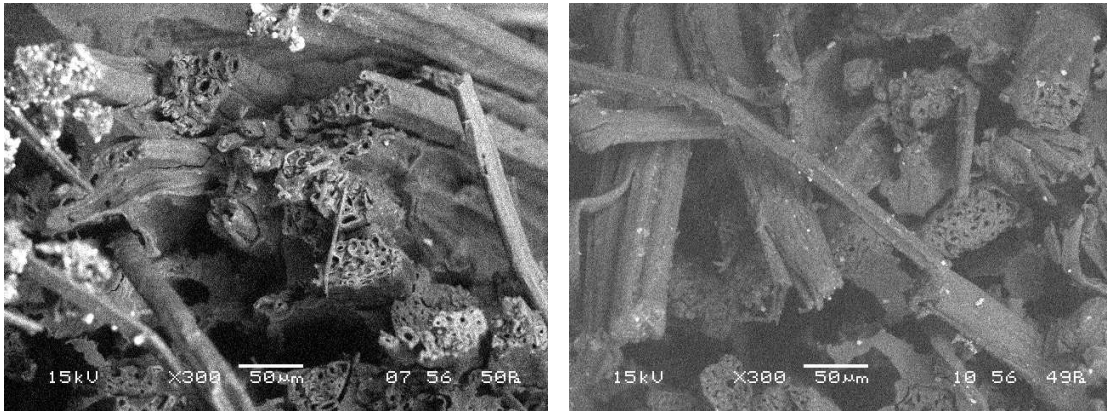The figure shows shows swelling due to absorption of moisture on the surface of the jute fibre.

60^{0}C

Fig. 4.6: Above- Untreated and 16 hours hygrohermally treated samples

At 60^{0}C, it can be seen that, the major cause of fibre failure is due to fibre pull-out. The matrix has developed voids in them due to removal of fibre. The degree of fibre pullout is found to increase with duration of hygrothermal treatment. Larger number of voids is found on increasing the exposure time. Moisture adsorbed on the fibre surface is clearly visible.
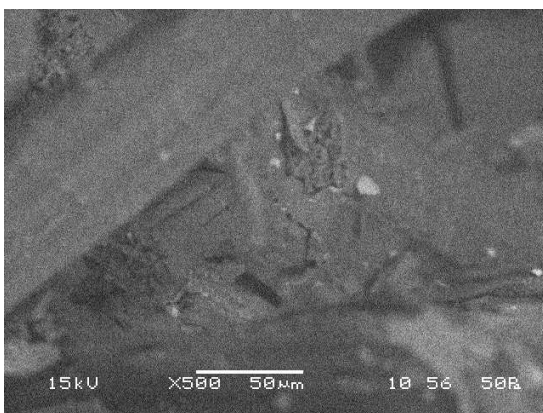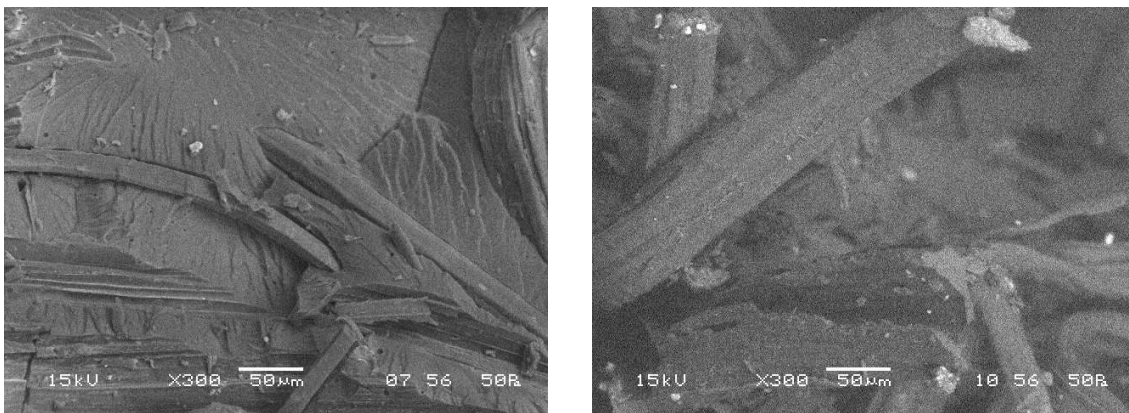

Fig. 4.7: The top left figure shows the matrix of untreated sample. The top right figure shows the matrix of 16 hours hygrothermally treated sample at 300X. The adjacent figure shows the same matrix at 500 X.

The matrix of the untreated sample shows no damage. In contrast, the hygrothermally treated samples show damage in their matrix. A crack is formed at the fibre-matrix, which leads to debonding at the interface, and finally fibre pullout occurs.
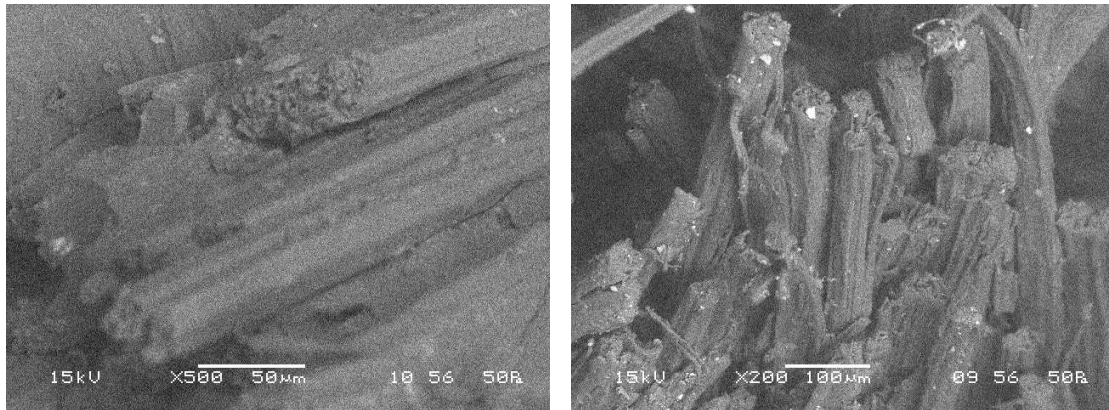
$100^{0}C$

Fig. 4.8: Fibre region of untreated and 16 hours hygrothermally treated sample

The untreated samples do not show any sign of fibre pull-out. The failure of the composite may be attributed to the failure of matrix. There are some signs of fibre breakage in the above figure in the case of hygrothermally conditioned samples. Mositure adsorbed onto the matrix and fibre can be seen.
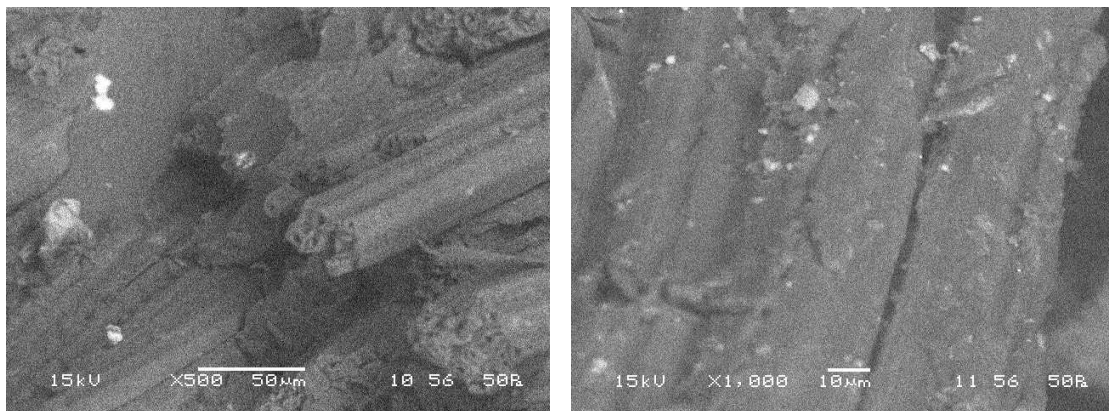

Fig. 4.9: Matrix cracking of untreated and 16 hours hygrothermally treated sample
The failure in case of $100^{0}C$ hot pressed samples is due to matrix cracking. The loss of toughness due to complete curing of the unsaturated polyester resin doesn't provide enough time for the transfer of load from matrix to fibre. The matrix behaves as a

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rigid material and the crack in the matrix doesn't get opportunity to blunt the tip resulting in loss of strength. The degradation is more when the sample is hygrothermally conditioned for 16 hours. The loss of material and the crack in the matrix is clearly visible in the above figures.

The reason for fibre pullout in most of the sample in contrast to fibre breakage can be ascertained to the fact that the bonding between the fibre and the matrix at the interface is weak. Hydroxyl groups(-OH) in the main backbone chain of a resin provide sites for hydrogen bonding to the surface of the natural fibres, which contain many hydroxyl groups in their chemical structure. Thus, polyester resins, having no hydroxyl group in its backbone chain generally has the weakest bonding, hence the lowest adhesive properties compared to other resins.

Chapter 5

CONCLUSIONS

5. CONCLUSION

- Hot pressing of the laminates resulted an increase in the tensile strength. This may be due to enhanced cross linkage during hot pressing.

- Optimum properties are obtained in case of hot press temperature of 80^0C. 100^0C hot press indicated a loss of strength, strain to fracture and energy at yield. This maybe due to larger cross linkage at 100^0C hot press condition resulting loss of ductility of the matrix. Fractographs show matrix-induced fracture in case of 100^0C hot pressed composites. 60^0C hot pressed composites exhibited fibre pullout.

- As expected a loss of strength, ductility and energy at yield are observed on hygrothermal treatment.

- Specimens hot pressed at 80^0C exhibited minimum loss in properties on hygrothermal treatment. this further proves the superiority of 80^0C over 60^0C and 100^0C hot press conditions.

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