Study of Moisture Absorption Behaviour of Agave Americana Fibre Reinforced Composite Material

A Thesis Submitted in Partial Fulfillment of Requirements for the Degree of

MASTER OF ENGINEERING

In Production Engineering

BY

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CERTIFICATE

I hereby declare that the dissertation entitled "Study of Moisture Absorption Behaviour of Agave Americana Fiber Reinforced Composite Material" is an authentic record of my work carried out as requirements for the award of the degree of Master of Engineering in Production Engineering at Thapar Institute of Engineering and Technology, Patiala under the supervision of Dr. Tarun Kumar Bera (Associate Professor, Mechanical Engineering Department) and Dr. Deepak Jain (Assistant Professor, Mechanical Engineering Department). No part of the matter embodied in this report has been submitted to any other university or institute for the award of any degree.

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ABSTRACT

Due to the increase in concern for the environment, the material scientists are shifting towards the natural fibres. Composites based on Agave Americana fibre reinforced epoxy resin are prepared using hand lay-up method. Agave Americana fibres are chemically treated with 2%(w/v) NaOH, alkali hornification and water hornification to reduce their affinity for water. Four cycles were carried for the water and alkali hornification. The SEM studies were carried out to analyze the cross section of the untreated fibre, NaOH treated fibre, alkali hornified fibre and water hornified fibres, the decrease in the holes and smoothing of texture is observed after the treatment was observed. The specimens prepared by ASTM D570-98 were characterized in terms of water absorption properties under controlled humidity and temperature environments. The effects of the treatment were investigated by means of a digital weighing machine and further validated with FEM analysis in the Abaqus software. The test partly attributed to disappearance of hydrophilic nature of the fibre. The alkali hornification gave the best results. The mass gain of the NaOH treated and the alkali hornified specimen are close in some conditions. The maximum mass gain is 8.02% at 75° C (Water Bath) in the untreated fibres whereas after the alkali hornification, NaOH treatment and water hornification the mass gain is 4.72%, 5.08% and 6%, respectively.

Key words Natural fibre; Agave Americana fibre; Moisture absorption; Chemical modifications; Mass gain analysis, FEA, Experimentation

NOMENCLATURE

a	Length [mm]
b	Width [mm]
c	Height [mm]
D	Diffusivity [m^2/s]
f_{ssc}	Edge correction factor
m	Mass [g]
S	Slope
t	Temperature [°C]
v	Volume [ml]
w	Weight [g]

SUBSCRIPTS

amb	Ambient temperature [°C]
eff	Effective diffusivity
i	Dry process
max	Maximum mass gain
w	Wet process

ABBREVATIONS

ARCS Agave Americana reinforced composite sheet
CO_2 Carbon dioxide
FEM Finite element modelling
FRCM Fibre reinforced composite material
R.H Relative humidity
SEM Scanning electron microscope

LIST OF FIGURES

(a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

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CHAPTER 1

INTRODUCTION

1.1 GENERAL INTRODUCTION

Due to the increase in the global warming material scientist are shifting towards the use of bi-composites containing a high amount of cellulose which captures CO_2 and reduces the amount of heat in the nature. Natural fibres are even replacing the glass fibres and other synthetic fibres because of its specific properties like less density, low cost and biodegradability. There are different ways to reinforce the natural fibres, the natural fibres also have a huge variety and this is discussed in this section.

1.2 FIBRE REINFORCED COMPOSITE MATERIAL

When the natural fibres are embedded in the matrix this lead to the emergence of new material known as fibre reinforced composite material. The length of the fibres plays an important role to define material properties. The short length fibres have good tensile properties. When subjected to axial loading the fibres elongates and bends easily which leads to a savage of the material. Figure 1.1 represents the structure of the FRCM

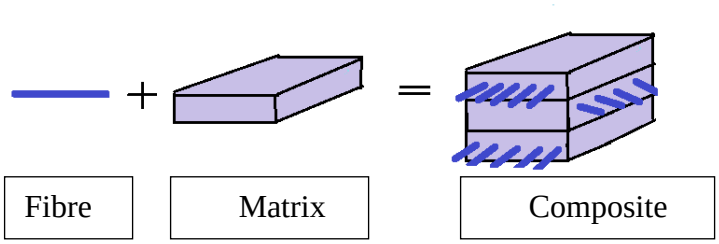


Figure 1.1 Structure of fibre reinforced composite material

- **Fibre**

A fibre is an elongated thread like structure which can be either man-made i.e. artificial or natural. They can be spun into ropes or can be matted into sheets. The reinforcement should be thermally stable with the matrix. Its affinity, density should be compatible with the matrix as it is a major factor in the fabrication.

- **Matrix**

A matrix acts as a supporting element for the composite material. It carries the load, transfers stress and maintains the stability of the composite material. It is a succouring element to the fibres as it protects the surface of the material and acts as a shield to the fibres by standing up in the adverse conditions like high temperature and wet environment.

1.3 CLASSIFICATIONS OF THE COMPOSITE MATERIAL

The composite materials can be classified in two ways—the first one can be according to the matrix material and the second classification is based upon the material selection. The main discussion will be done on the material selection under this section.

1.3.1 Types of Fibre Reinforced Composite Material

While fabricating the composite material the fibre size, orientation and its coatings or thickness plays a vital role. There are three ways by which we can disparate the organization of fibre for different conditions which are described in Figure 1.2.

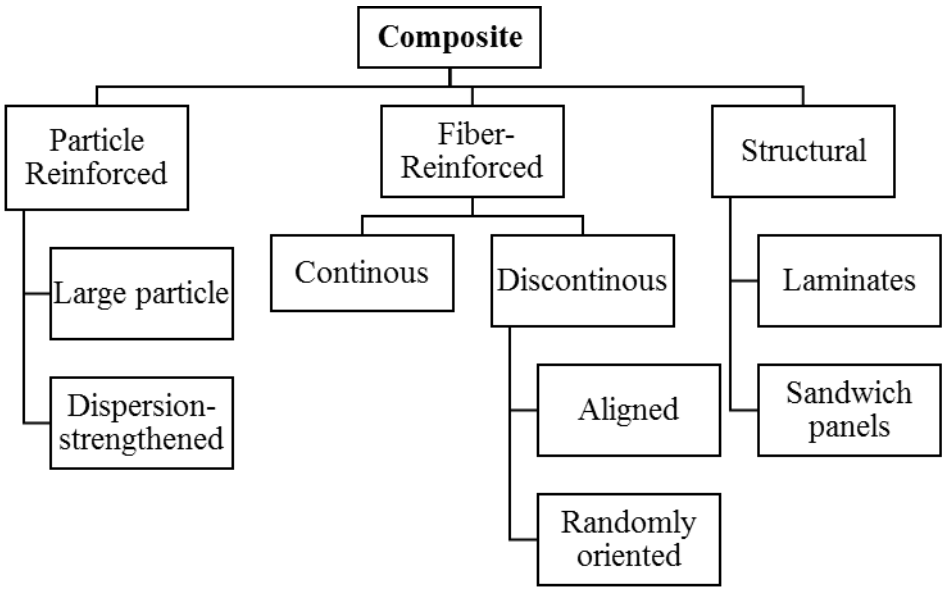


Figure 1.2 Classification of the composite material

1.3.1.1 Particle Reinforced Composite Material

When the fibres (particle) are reinforced in the matrix (Figure 1.3) whether in the dispersed form or in the form of flakes that are parallel to each other, that is known as a particulate composite. The particulates enhance many properties like thermal conductivity and moisture durability.

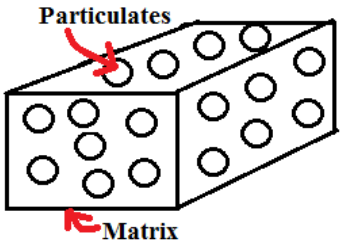


Figure 1.3 Particle reinforced composite material

It is further of two types:

- **Large particle**

The flakes are arranged parallel to each other in this kind of arrangement. Long fibres prevents the movement of the particles and many defects like edge dislocation and screw dislocation can be avoided.

- **Dispersion-strengthened**

The particulates or fibres are dispersed in the whole matrix. This phase increases many properties like tensile strength and provides elasticity.

1.3.1.2 Continuous and Discontinuous Fibre-Reinforced Composite Material

The embedment of the fibres can be done into two ways one can be in the form of continuous fibres and secondly, they can be discontinuous i.e. short fibres.

Figure 1.4 Fibre-reinforced composite material: (a) Continuous and (b) Discontinuous fibre

- **Continuous fibre-reinforced composite material**

The long fibres are reinforced into the matrix. The orientation of the fibres can be unidirectional or bidirectional (woven).

- **Discontinuous fibre-reinforced composite material**

The short fibres are reinforced into the matrix in the dispersed form. The length of the fibres should be greater than hundred times of its diameter. The short fibres can be arranged in aligned or random way according to the requirement of the material.

1.3.1.3 Structural Composite Material

The fibres, when reinforced into a matrix forms a single layer but when more layers are arranged with different manner a unique structure is being constructed that is known as structural composite material. Ply, large structure made by rapid prototyping can be seen as its few examples. It can be of two types:

- **Laminates**

The stack of two or more laminae (or layers) forms laminate (Figure 1.5 (a)). The term multilayer (angle-ply) can be used for this kind of composite.

- **Sandwich panels**

The core is used as a sandwiched material between the face sheets. The sandwich panels are having low density and light weight. (Figure 1.5 (b))

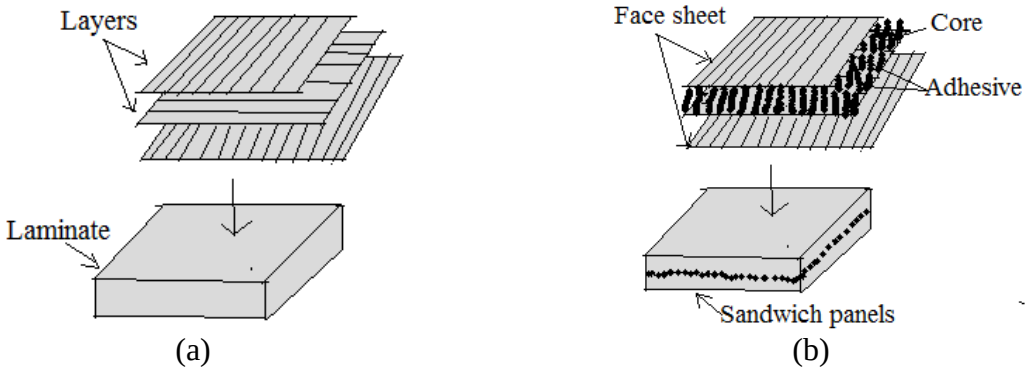


Figure 1.5 Structural composite material: (a) Laminates and (b) Sandwich panels

1.4 NATURAL FIBRES

Due to increase in the environmental concerns scientists are shifting towards natural fibres as they have been found as a promising reinforcement in the polymer matrix composite. The fibres after their extraction are embedded within the matrix and a new composite material or hybrid is obtained with better properties. The limitations of using natural fibres are that they are hydrophilic in nature. So, they require chemical treatments in order to become hydrophobic and adhesive with the matrix.

1.4.1 Types of Natural Fibres

There are variety of fibres that can be extracted from the plants, animals and the minerals. The different types of natural fibres are described in Figure 1.6:

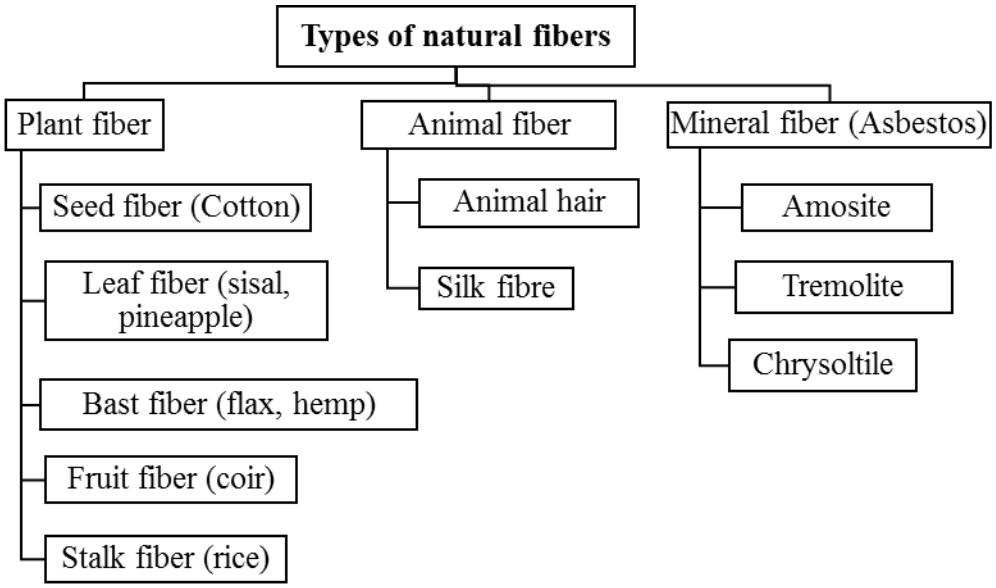


Figure 1.6 Types of natural fibre

- **Animal fibre**

The main constituent of the fibre extracted from the animals is proteins. Silk extracted from the larvae of silkworms, wool from the fur and feathers, alpaca fibre and motoir fibre are obtained from the Angora goat, angora wool which is very famous is obtained from the rabbits are the biggest examples of the animal fibres.

These fibres are having different properties some are very soft, fine whereas some are very coarse and dense.

Silk is woven from the cocoons which are obtained from the larvae of the silkworms is expensive fibre. The length of the silk depends upon how it is prepared. There are varieties of silk: spider silk which is known as the strongest natural fibre whereas dragline silk is even stronger than steel and kelvar. Dragline silk which is elastic in nature, is used by the military in the U.S for the border safety.

Wool is basically obtained from the fur of the animals. Caprine family that is the family of the sheep is thankful for providing the wool. Wool have different categories like Alpaca wool is famous for its light weight it is even warmer than the sheep's wool, Angora wool acquired from the rabbits is very soft. The diameter of this wool is 12-16 micro meters and hence known as a renowned quality fibre. Bison that is the native of America, its hair are very soft. The coarse hair fibres have 59 micro meter diameter are used to make the under coat and gloves. Horse hair is used to make brushes and bows of various musical instruments. Hence, there are many animals like Cashmere, Mohair, Sheep, Camels, Yak etc who are a source of fibre to us.

- **Mineral Fibres**

Mineral fibres are commonly known as Asbestos, these fibres are an amalgamation of six natural silicate minerals. The fibres obtained from the Asbestos have millions of fibrils which are released from abrasion. The Asbestos is found in three colours mainly i.e. blue, brown and green. The white colour is seldom observed. Inhalation of Asbestos is very harmful it may lead to serious diseases like lung cancer, dizziness etc. They are very expensive and used in making of ceilings, buildings, roofs, sheets, panels etc. There are basically three main types of Asbestos these are: Amosite, Tremolite and Chrysolite.

Amosite is a brown coloured mineral who belongs to a cummingtonite-grunerite solid solution and belongs to South Africa. It is generally brown in colour but grey white colour is also observed. The sheets made from the amosite fibre are fire retardant. They are used as insulation material in the roof ceilings, tiles etc.

Tremolite belongs to amphibole group and rich in dolomite and quartz. The pure magnesium tremolite is white in colour while some tremolite fibres are green in colour. Inhaling of tremolite abrasives are very harmful and toxic and leads to lung cancer.

Chrysolite the white fibres are emerged from the serpentinite rocks. These fibres are very flexible, soft and can be easily spun or woven. Chrysolite fibres sheet are used in a cement roofing, warehouse garage, panels etc.

- **Plant Fibre**

The field crops provide us the fibres which are rich in cellulose. Cellulose is responsible in providing strength to the fibre. The main worry of getting the fibres is due to their seasonal availability. There are thousands of natural fibres available to us.

Bast fibre (stem skin fibres) have a huge variety of fibres. Flax which helps in the production of linen, jute: cheapest fibre, Esparto: obtained from the grass, Indian hemp: useful for native Americans, Hemp: soft and strong fibre, Hoopvine: used to make baskets and barrels, kenaf fibre, Beans, Ramie fibre and papyrus fibre are the few examples of Bast fibre.

Seed fibre and **Fruit fibre** also have a variety of fibres within them i.e. Coir which is the residual of coconut husk, Cotton, Kapok, Milkweed, Luffa fibre used to make loofa sponge etc are the few examples of seed fibre.

Leaf fibres are the greatest source of natural fibres. They are extracted from the plants by decortication process. During this process, the non-fibrous tissues are left behind and only the fibrous ropes are extracted. Due to the toughness and rigidness of these fibres they are used in the production of ropes. Here, are the few examples of leaf fibres: Abaca fibre from the banana produce "manila" rope. Sisal, Agave Americana, Henequen, Yucca and Bowstring Hemp from the agave family are the few examples of leaf fibres.

Banana Fibre (Figure 1.9(a)): Alkali treatment cleans the fibre surface, impurities, modifies surface structure and moreover enlarges its surface area. It is known as a textile fibre having low density and high disposability. These fibres are highly recyclable with 13% of moisture weight gain. It can be spun easily and have low elasticity.

Palmyra Fibre (Figure 1.9(b)): It is also known as Borassus Flabellifer or petiole fibre. The palmyra is 30 m in height with fan shaped leaves which are 2-3 m long. Its globe shaped fruits are 15-25 cm wide and contains 1-3 large seed. It is cultivated in Southeast Asia. The palmyra is having more than 800 uses in that region, few examples are mats, baskets, fans, hats, umbrella etc.

Pineapple Fibre (Figure 1.9(c)): The white and glossy fibres are produced in tonnes. These fibres are hydrophilic in nature due to its high cellulose content. At high temperature, the

hydrophilic nature of pineapple fibre do not let it bond with hydrophobic matrix. The fibres can be easily extracted by retting or mechanical methods.

Sisal Fibre (Figure 1.9(d)): Agave Sisalana (native to Southern Mexica) is the botanical name of the sisal fibre. Sisal fibre shed around 200-250 sword shaped leaves which are 1.5-2 metre in length. Each leaf further contributes to give 1000 fibres. The fibres are extracted by the decortication process. The best working temperature for the sisal fibre is 25°C. Under high moisture content its quality deteriorates. The applications are: it can be used in making of ropes, carpets, fabric industry, footwear, hats etc.

Agave Americana Fibre (Figure 1.9(e)): The Agave Americana plant or commonly known as century plant is native to Mexico, Texas but is cultivated along the railway tracks, roads in the world wide. There natural growth aids a benefit in the arid and semi-arid regions. They possess a high amount of cellulose i.e. 73%-78% (**Crop Production Guide, 2012**) which is nearly about four times than other fibres so they really help in decreasing the global warming by capturing CO_2 from the surroundings. The Agave Americana plant have age of 8-30 years and shed around 40-50 leaves per year with a fibre content of 2.5%-4.5%.

(a)

(b)

(c)

(d)

(e)

Figure 1.7 Different types of leaf fibre (a) Banana fibre (Alavudeen, 2016), (b)Palmyra fibre (Srinivasababu et al., 2014), (c) Pineapple fibre (Santos et al., 2013), (d) Sisal fibre (Ramalingam et al., 2017) and (e) Agave Americana Fibre

Table 1.1 Properties of agave Americana leaf fibres (Hulle et al., 2015)

Property	**Value**
Length	2.8 m
Diameter	100-150 µm
Tenacity	16-41 cn/tex
Strain	2%-4 %
Work fracture	7.7–25.4 (mN/tex)
Color	Off white to yellowish
Luster	Semi dull
Texture	Strong and Durable
Moisture	7%-9% by weight
Tensile strength	Non-Uniform
Density	1.36 gm/cm^3
Fibre percentage	2.5%-4.5%
Age	8-30 years
Cellulose	68%-80%
Hemicellulose	15%
Lignin	5%-17%
Wax	0.26%

Agave Americana fibres which are extracted from the leaves of the plant are not uniform in nature. They vary from thick in thin in diameter and hence the tensile strength varies (**Steyn and H.J.H., 2006**). The fibres are biodegradable and is utilized completely i.e. “Zero Wastage”

of the plant is observed. The fibre is mechanically extracted and is hydrophilic is nature so it is necessarily important that it goes under chemical treatments in order to exhibit its hydrophobic nature, or the reduction of water up-take in its nature. It is stable under low acids and low bases and under the presence of high bases and acids it becomes very unstable. The high bases and acids leads to the extraction of lignin which leads to decrease of the tensile strength.

The Agave Americana Fibre used for the research work is bought from the Coimbatore, South India. The fibres are very coarse, hard in texture due to the presence of more than 5% lignin content and have low elongation. The properties of the Agave Americana fibre are expressed above in the tabular form.

Comparison of Natural Fibres

- On the basis of chemical composition

Table 1.2 Comparison of Natural fibres on the basis of chemical composition

Type of fibre	**Cellulose**	**Lignin**	**Hemicellulose (%)**	**Pectin (%)**	**Ash (%)**	**Moisture Content (%)**	**Waxes**
Fibre flax	71	2.2	18.6-20.6	2.4	-	8-12	1.7
Jute	45-72	12-26	13.6-21	0.3	0.5-2	12.5-13.7	0.5
Hemp	58-78	3.7-13	14-22.4	0.9	0.8	6.2-12	0.8
Ramie	68.6-92	0.6-0.8	5-16.7	10	-	7.5-17	0.3
Jute	41-50	21-25	18-22	-	0.8	12.5-13.7	0.5
Abaca	56-65	7-10	15-17	-	3	5-10	-
Sisal	47-80	7-11	10-24	10	0.6-1	10-22	2
Banana	61-66	56-14	0.15-0.25	-	-	13	-
Pineapple	70.56-82.31	5.4-12.4	23.22-28.6	-	-	13	-
Agave Americana	68-80	5-17	15	-	-	7.69-8	-

- On the basis of mechanical properties

Table 1.3 Comparison of Natural fibres on the basis of mechanical properties.

Type of fibre	Density (g/cm^3)	Elongation (%)	Tensile strength (MPa)	Young's Modulus (GPa)	Moisture Content (%)
Cotton	1.5-1.6	3.0-10	287-597	5.5-12.6	8-25
Jute	1.5-1.46	1.5-1.8	394-801	10	12
Flax	1.4-1.6	1.3-3.3	345-1500	28-80	7
Hemp	1.50	1.7	550-900	71	8
Ramie	1.6	2.0-3.9	220-938	44-126	12-17
Sisal	1.33-1.6	2.1-15	400-700	9-37	11
coir	1.3	15-31	175-220	4-6.1	10
Banana	0.6-1.3	1.22-3.56	51.6-55.2	3-3.8	13

The motivation of the thesis is to improve the hydrophilic nature of the fibre by chemical treatments so due to the less moisture absorptivity of Agave Americana fibre i.e. 7.69%-8% it is being chosen for further experimentation.

1.5 APPLICATIONS OF NATURAL FIBRES

Over the decades, the applications of natural fibres are increased now, they are being used in automotive industry, medical industry, buildings, packaging, sports and textile industry. Its few applications are:

- **Building**: The panels, door frames, ceiling, door shutters are made from the natural fibres. The jute, sisal and coir are used for dough moulding. In India, the insulation boards are made from the bagasse. In Thailand, hardboards are made from coconut coir.
- **Packaging**: The plates are made from the banana fibre. The bio degradability is really very appreciative. But, the low mechanical property is the main limitation of using natural fibre in the packaging.
- **Medical**: The palm tree fibre is used in Indonesia for medical purposes it is first treated with polyurethane and polyvinyl alcohol.
- **Sports**: Bicycles and tennis rackets are made from the flax fibre.
- **Automotive**: The interior and exterior of the automotive which includes seat back, door and side pads, spare tire lining, dash-boards are made from natural fibres. The brake pads are made from the palm Kernel fibre. The famous brands like Audi, BMW, Fiat, Ford, Volvo and Volkswagen etc are using natural fibres these days.

1.6 LIMITATIONS OF NATURAL FIBRES

Natural fibres are widely used due to their biodegradability and low cost. Despite of its benefits its main limitation is its hydrophilic nature which is responsible to moisture up take when it is exposed to high temperatures, moisture, water etc.

This may lead to volumetric expansion and degradation and long term structural damage. If the fibre reinforced composite matrix is exposed to the harsh surroundings for long term then it leads to deterioration. The matrix and fibre bonding also get delaminate and leads to matrix cracking.

Diffusivity i.e. the rate of moisture uptake is responsible for the volume expansion in the composite material. The mass gain due to moisture can be checked by gain weight gain of the specimen till the saturation of specimen do not arrive. This process is a cumbersome and time consuming as it takes several months.

1.7 COMMERCIAL AVAILABLE DATA FOR MATRIX RESIN AND HARDENER

Huntsman Epoxy Resin i.e. LY556 and Hardener HY951 are used as a matrix material. In winters, at room temperature the matrix resin is transparent in colour and can be used for a period of half hour after then the matrix resin becomes solid. The glass beakers should be used to mix the epoxy resin and hardener as while mixing there is an exothermic reaction and the beaker gets heated up but the matrix resin does not turn up into solid whereas, in plastics within a span of ten minutes the matrix resin becomes solid. Even under high temperatures like 30°C the matrix resin heats up so fast and turns into solid. Here, are the few available data for the epoxy resin and hardener:

Table 1.4 Specifications of Epoxy Resin i.e. LY556

S/N	Parameter	Specified value
1	Grade	LY556
2	Colour	Pale yellow, clear liquid
3	Specific gravity at RT	1.10-1.20
4	Viscosity at 25°C, cPs	8000-12000
5	Epoxy content, Eq/kg	5.0-5.9
6	Volatile content, by weight	0.75% (maximum)

Table 1.5 Specifications of Hardener HY951

S/N	Parameter	Specified value
1	Viscosity at 25°C, mPa s	10-20
2	Specific gravity at 20°C, g/cm^3	0.95-1.05
3	Flash point, °C	110
4	Vapour pressure at 20°C, Pa	0.3

The mixing ratio of the resin and hardener is 10:1 (by ml). The setting time takes 5 hours at 25°C whereas it takes 7 days to get the matrix fully cured.

1.8 PROBLEM FORMULATION

Natural fibres are used as an alternative material over the synthetic fibres due to its low cost and bio degradability. The limitation of using natural fibres is that these are hydrophilic nature. In the adverse conditions like moisture, water and temperature, the fibre leads to mass gain and degradation of the composite material occurs. So, efforts are required in regard to disappear the hydrophilic nature of the natural fibres.

1.9 SCOPE AND CONTRIBUTION OF THE WORK

The agave Americana fibres are prepared by the hand lay-up method. The work contributed to reduce the affinity of natural fibres towards moisture by chemically treating them. Agave Americana fibre is chosen as a reinforcement material and in order to improve its hydrophilic nature, three types of treatments will be done i.e. NaOH treatment, alkali hornification and water hornification. After the fabrication, the specimen will be weighed for a long period of time till it does not get saturate. Comparisons are being drawn between the untreated and chemically modified fibres at seven different conditions i.e. 0°C in the cryostat bath, 25°C, 50°C & 75°C in the water bath and 25°C, 50°C & 75°C with 70% R.H in the environment chamber.

1.10 ORGANIZATION OF THESIS

Chapter 1 introduces the fibre reinforced composite material. The different types of natural fibres are discussed under this section and the comparison on the basis of the moisture absorption is done in regard to the select the best possible fibre for the experimentation.

Chapter 2 presents the detailed literature review. It clearly explains about the literatures regarding the natural fibres and the treatments which can improve the hydrophilic nature of the fibre.

Chapter 3 includes the chemical treatment of the chosen fibre in chapter 1 i.e. agave Americana fibre and the cross section of the fibre is analyzed by SEM image.

Chapter 4 is dedicated to the preparation of the specimen from the untreated and chemically modified agave Americana fibre by hand lay-up method for the experimental analysis.

Chapter 5 includes the results and discussions. The experimental results are obtained from the seven submerged conditions and are further validated with the finite element analysis.

Chapter 6 presents the conclusions and the scope of future work.

Summary of the work done is also presented using flowchart.

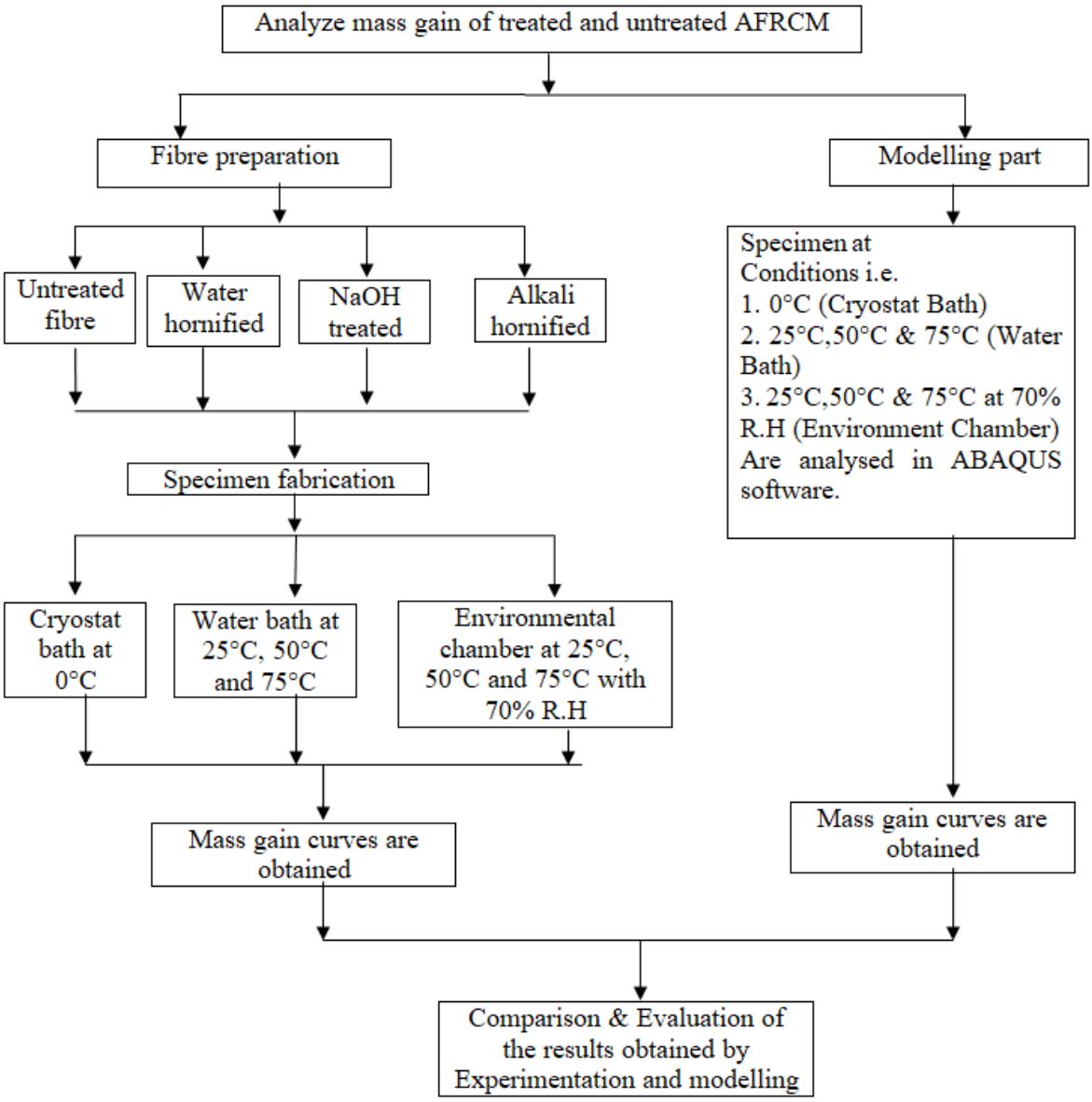


Figure 1.8 Summary of the work done

CHAPTER 2

LITERATURE REVIEW

Literature study on innumerable research papers was carried out in order to find out the best possible way by which we can understand the agave Americana fibre's hydrophilic nature.

2.1 LITERATURE REVIEW ON NATURAL FIBRES

Reinforcements of fibres play a vital role in deciding the strength of the composite material. There is a huge variety of natural fibre. The study has been done on the different types of banana fibres i.e. knitted banana fibre, raw banana fibre and the banana yarn. It is being found that the banana yarn possesses the highest strength among all the varieties of the fibre. If coupling agent i.e. MAPP is added as a treatment to the fibre, it even acts like a strong adhesion between the fibre and the matrix (Amir et al., 2011). The banana fibre's volume is varied, the strength and the water absorption of the short length of the banana fibre is evaluated. The fibres are mechanically tested after chemically modifying its surface and structure with the 10% alkali solution. The results are mesmerizing the strength and the young's modulus of the fibre increased to a large extent. The SEM analysis provided a large analytical view in which surface smoothness can be observed. The treated fibres are beneficial contribution in this work (Merlini et al., 2011). The Increase in number of layers of banana leaf fibres enhances the strength and the strength along the orientation of the fibre is maximum. The banana leaf fibres are laminated and the strength analysis is done. The work is further continued as, in regard to increase the tensile strength the lamination also plays very important role. The banana leaves itself contribute for the formation of the laminated fibres. The banana leaf tapes are prepared using adhesives on both the sides. The four layered laminated composites possess the highest flexural strength. The orientation leads to the degradation in some cases, a parallel oriented organization adds more strength to the composite sheet (Nongman et al., 2016).

Hybridization also enhances the strength of the fibres. The palmyra fibres are extracted from the process of deterioration. The alkali treated palmyra possesses high strength as compared to the untreated fibres. The palmyra fibres are hybridized with the jute fibre. The uni-directional orientation is followed layer by layer. The compression moulding technique helped

in the preparation of the specimen. The SEM gave us the analysis of the improvement of the surface after the alkali treatments. The mechanical properties i.e. the static and dynamic are improved with the hybridization of the fibres. The results can be comparable to the glass fibres (Shanmugam and Thiruchitrambalam, 2011).

Pineapple family has a huge variety. The six different varieties of the pineapple are studied in regards to find out the best and promising reinforcement. The elastic modulus, young's modulus and tensile test are analysed on each variety. With in the ranges the tensile strength varied between 210 to 695 MPa and the young's modulus between 15 to 53 GPa. So a huge variation is observed. The crystallity index also changes with the change of the cross section of the fibre (Neto et al., 2013). The work is further continued by Neto by selecting the Ananas variety of the pineapple. The twelves fibres from different varieties are analysed, the strength varies and the mechanical properties also. Hence the proper selection of the fibre plays a vital role for the preparation of the composite material. After selecting the proper variety of the fibre the next step is to modify its structure chemically to enhance its surface and properties. The alkaline and saline treatments are carried on the pineapple fibre for the surface enhancements. The treatments lead to the removal of the impurities. The silane treatment gives appreciating results by improving the tensile strength of the fibre. The images from SEM show us the surface modifications. The addition of the acrylate butadiene made the fibre very stiff. Hence, the chemical treatments proved to be an essential part (Asim et al., 2016).

Screw pine fibers degrade with time. In regard to increase the strength of the fibres to bear the adverse conditions the 5% NaOH treatment is done. The small length fibres of 5 and 10 mm are extracted and the NaOH treatment is carried. The clean i.e. the untreated fibres and the modified fibres are soaked in water for different intervals of time i.e. 30, 60, 90, 120 and 150 mins. The degradation of the untreated fibres increased with the time whereas the chemically modified fibres showed a slowd in the degradation. The 7.5 % NaOH treated fibres further enhanced the properties to a large extent. The moisture absorption is decreased (Hairul Abral, 2012)

The volume fraction and the time of immersion is a matter of concern to study the saturation and the degradation of the fibres. The untreated and the treated sisal fibres are compared. The NaOH treatment is carried out on the fibres. The 2 m, 3 m, 4 m, 5m and 6 m length fibres are extracted. The short length fibres have high tensile strength due to their

capability of moving. The fibre volume also varied from 10%, 15%, 20%, 25% and 30%. Due to the increase in the thickness of the fibre the tensile strength increases, correspondingly the water up take also increases. Hence the combination must be selected carefully (Prasad et al., 2017)

2.2 LITERATURE REVIEW ON CHEMICAL TREATMENTS OF THE FIBRES

The effect of Maleic anhydride polypropylene copolymer is investigated on the flax fibre. MAPP enhanced the adhesion between the fibre and the matrix, the water uptake was decreased and more the fibre content more will be the water uptake. MAPP treatment also improved the mechanical properties (Arbelaiz et al., 2005). The pretreatment of non-woven flax fibre was done with 5% NaOH. After that the pretreated fibres were further treated with 5% butane tetra carboxylic acid (BTCA) and 1% amino propyl tri ethoxy silane (APTES). The fibres became very rough after the treatment. The specimens prepared from NaOH-BTCA were further immersed in 1% BTCA and the specimens prepared with NaOH-APTES were immersed in the solution of (80:20) ethanol and APTES. These solutions acted as insulation for the specimen. The 100% humidity conditions were investigated. Although the results were not satisfying, still NaOH-BTCA showed better results (Zhu et al., 2013).

In regard to improve the water affinity and the fibre bonding with the matrix agave Americana fibres are chemically treated with maleic anhydride, acetic anhydride, acrylic acid and styrene. The resultant is acetic anhydride, acrylic acid and styrene showed the similar results whereas maleic anhydride showed better results among all. The size of fibre is also decreased which helped in fibre matrix bonding (Bessadok et al., 2008). Alkali treated Agave fibres were subjected to moisture absorption test in the cold and hot water conditions. The treated fibres were having good adhesions and have showed less water absorption as compared to the untreated fibres. The tensile strength and compression strength were also improved with the alkali treatment. (Mylsamy & Rajendran, 2011). The work further continues with Fibre length as consideration in this study. The short Agave fibres were taken of length (3 mm, 7 mm and 10 mm). The alkali treated short fibres and the untreated ones were further compared. The alkali treated fibres have slightly better tensile strength, compression strength, flexural strength, impact and less water absorption. The short fibres improved the fibre wetting and impregnation. (Mylsamy & Rajendran, 2011) The surface the Agave fibres were modified by graft copolymerization of methyl methacrylate and ceric ammonium was used as an initiator. The fibre weight was varied from 10% to 30% in the polystyrene rich matrix. The 20% fibre rich and

treated agave fibre specimen showed the best results. It had good tensile strength, higher thermal stability and better mechanical properties (Singha & Rana, 2012).

Wood Flour/Wheat Husk fibres were prepared using melt blending process. These fibres were treated with NaOH and Benzoyl chloride. Due to high cellulose content the water absorption rate is high in these fibres but after the treatment, the mechanical properties like tensile strength, flexural and impact strength were improved and the water absorption rate was decreased (Upadhyaya et al., 2012).

Alkali treatment was carried on the Abaca fibre in regard to reduce the water up take. The 20% NaOH treated abaca fibre had less moisture absorption as compared to 5%, 10% and 15%. This showed that higher NaOH lead to more removal of lignin. (Ramadevi et al., 2012) This paper focused to reduce the moisture absorption and water uptake on the Hemp fibre which are reinforced in the polyethylene composite. The effects of fibre orientation along with the different coupling agents are also varied. Before the embedment, the fibres were treated with maleic anhydride, grafted copolymerized into polyethylene. The result was appreciating the moisture absorption is decreased whereas it leads to decrease in the flexural strength so that was one of the limitation of this treatment. The engraving method of batch mixing also helped in decreasing the moisture content as compare to the twin screw extruding (Fang et al., 2013).

In this work the ultrasonic treatment was done on the Banana fibre. Firstly, the fibres were treated with the NaOH and then went under ultrasonic treatment. In the initial stage, Fickian rule was followed and higher water absorption was achieved and no more water absorption was further observed. With this treatment the fibre matrix bonding was increased and further the strength of the fibres increased (Ghosh et al., 2014).

The thermal degradation of untreated and silane treated sisal fibre has been studied. With the FTIR spectroscopy it has been seen that a layer of film is formed which consists of siloxane and polysiloxane. This layer helps in improving the thermal stability (Zhou et al., 2014). In this paper, the durability of Sisal fibre can be improved by two methods thermal treatment and Na_2CO_3. The hornification process also accelerates aging. With the thermal treatment the durability and compressive strength is increased by 35.5% and 31.1% respectively moreover, the structure of cellulose's crystallization is enhanced. Whereas with the Na_2CO_3 treatment the durability and compressive strength is increased by 46.2% and 45.4% respectively and there

formed a layer on the fibres which resists corrosion (Wei & Meyer, 2014). In regard to reduce water absorptivity Sisal, Jute and Curaua fibre were tested with hornification process. The number of cycles chosen was 5 and 10 cycles. Jute and Curaua showed best results under 5 cycles whereas Sisal was still having good properties under 10 cycles. The result observed after the hornification process is the tensile strength was increased, the water uptake was reduced, the volume and the diameter of the fibre is reduced which further lead to greater dimensional stability. Moreover, the new bonds were formed in the microfibrils which helped in good fibre matrix interface (Ferreira et al., 2016). In this investigation the Sisal fibre and the Bamboo fibre were hybridized. The fibres were treated with 10% alkali solution and then further reinforced in the unsaturated polyester resin. The hybridization leads to increase in tensile properties by 30%, flexural strength by 27.4%, impact strength by 36.9% and the water absorption was further reduced. The 100 wt% sisal fibres had the least water absorptivity whereas the hybridization having 25 wt% sisal / 75 wt% bamboo fibres have the maxims water up take. (Venkatesh et al., 2016)

Silane treatment was carried out using Vinyl trimethoxy silane on the Ijuk fibre. The 10 wt%, 20 wt%, 30 wt% fibres were reinforced after the treatment. The resultant was the mechanical properties got enhanced whereas the water absorption was reduced. The 10 wt% and treated fibre showed less water absorption as compared to untreated 30 wt% fibres. (Zahari et al., 2015)

Agave fibres (10, 20, 30% wt) are treated with Polyhydroxy butyrate and its copolymer hydroxy valerate. The treatment enhanced many properties like tensile modulus by 80% and 50%, flexural modulus by 36% and 41%, impact strength with 44% and 66% with PHB and hydroxy valerate respectively. The use of 30% fibre by weight gave the best results (Tello et al., 2017). In this investigation, Agave marginata and Agave sisalana were compared on the basis of age and other mechanical properties. Agave marginata reinforced in epoxy and PLA matrices were proved to be three times beneficial than Agave sisalana. The extraction of fibres from the simple pressing proved to be the most beneficial method. Moreover, high aged fibres were stiff as compared to 1-2 year aged. The mercerization treatment improved the tensile properties and the water up is decreased (Zuccarello & Zingales, 2017).

Bleached and unbleached Eucalyptus and Pine fibres were hornified. The number of cycles chosen was four. After the hornification process i.e. the wetting and drying cycles the water

retention value was calculated. The resultant was the decrease in water retention value after each cycle, the loss of water absorption and swelling reduced. The fibre's coarseness, fineness increased and the width reduced. With the hornification process no deterioration was observed and the cellulose of the fibre was preserved. Further, due to the fineness of the fibre it leads to greater dimensional stability (Ballesteros et al., 2017).

Hornification process (1 cycle) was done on the Jute fibre. The medium of hornification was alkaline with the presence of calcium hydroxide. The water uptake is reduced, mechanical strength is increase by 70%, tensile strength by 176% and young's modulus by 4%. There formed a greater dimensional stability, positive changes in the structure which lead to great fibre matrix bond. The lignin and hemicellulose were removed even with the 1 cycle and the crystallity is increased (Santos et al., 2017). Cordia-Dichotoma fibre were extracted by water retting method. The fibres were treated with 5% NaOH. After preparing the specimen from the hand-lay-up method with the variation in fibre weight from 0% to 25%, moisture absorption test was conducted which resulted in decrease of moisture percentage as compare to the untreated ones (Reddy et al., 2018).

2.3 OBSERVATIONS DRAWN FROM THE LITERATURE

The main drawback of using natural fibre is that it can't withstand under adverse conditions like humidity, moisture, water, high temperature *etc*. The literature is mainly concentrated on the chemical treatments that were used in regard to convert the hydrophilic nature of the fibre to the hydrophobic nature. Table 2.1 below summarizes all the treatments on different fibres which helped in the reduction of the water up-take or moisture gain.

Table 2.1 Different treatments of the natural fibres

Sl. No.	Fibre name	Treatments
1	Flax fibre	Maleic anhydride polypropylene copolymer
2	Agave Americana fibre	Maleic Anhydride, Acetic Anhydride, Acrylic acid and Styrene
3	Agave fibres	Alkali treated
4	Short Agave fibres	Alkali treated
5	Agave fibres	Graft copolymerization of methyl methacrylate and ceric ammonium was used as an initiator
6	Wood Flour/Wheat Husk	NaOH and Benzoyl chloride
7	Abaca fibre	(5% ,10% ,15% & 20%) NaOH
8	Hemp fibre	maleic anhydride, grafted copolymerized into

		polyethylene
9	Flax fibre	5% NaOH, 5% butane tetra carboxylic acid (BTCA) 1% amino propyl tri ethoxy silane (APTES)
10	Banana fibre	NaOH and Ultrasonic treatment
11	Sisal fibre	Silane treatment
12	Sisal fibre	thermal treatment, Na_2CO_3 and hornification process
13	Ijuk fibre	Silane treatment using Vinyl trimethoxy silane
14	Sisal, Jute and Curaua fibre	Hornification process (5 & 10 cycles)
15	Sisal fibre and the Bamboo fibre	10% alkali solution
16	Agave fibres	Polyhydroxy butyrate and its copolymer hydroxy valerate
17	Agave marginata and Agave sisalana	Mercerization treatment
18	Eucalyptus and Pine fibres	Hornification process (4 cycles)
19	Jute fibre	Alkali Hornification process (1 cycle)
20	Cordia-Dichotoma fibre	5% NaOH

From the above literature, it is clear that NaOH treatment, water hornification and alkali hornification process are not carried on the Agave Americana fibres. So, we have chosen these treatments in regard to check whether it is creating a difference to its hydrophilic nature.

2.4 OBJECTIVES OF THE THESIS WORK

The main limitation of the natural fibre is to withstand in the adverse environment. Agave Americana fibre is chemically modified with the three types of treatments and is tested under different conditions.

- The agave Americana fibres are treated with 2% (w/v) NaOH solution, alkali hornification and water hornification. The effect of the NaOH treatment and the four cycles of the alkali and water hornification on the cross section of the fibres are analyzed by SEM

- The specimens are prepared by ASTM D 570-98 and the mass gain analysis is done experimentally under seven controlled submerged environments.

- The comparison and validation of the experimental results are done with the modelling by finite element method.

CHAPTER 3

CHEMICAL TREATMENTS OF FIBRES

The fibre modification is known to have a pronounced effect on the material properties of natural fibres. The objective of present work is to observe the effect of different fibre treatments on the moisture uptake behaviour of Agave Americana. The fibre modification is carried out by the following methods:

3.1 FIBRE PREPARATION BY NaOH TREATMENT

The fibres (50 gm by weight) was soaked in the 2% (w/v) NaOH solution (Oudiani *et al.,* 2011) for the duration of 1 hour. After 1 hour, the fibres were washed with plenty of distilled water until the NaOH is being removed and then it is left aside to dry at the room temperature.

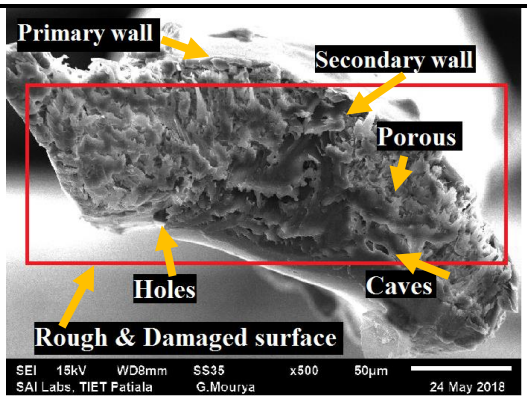
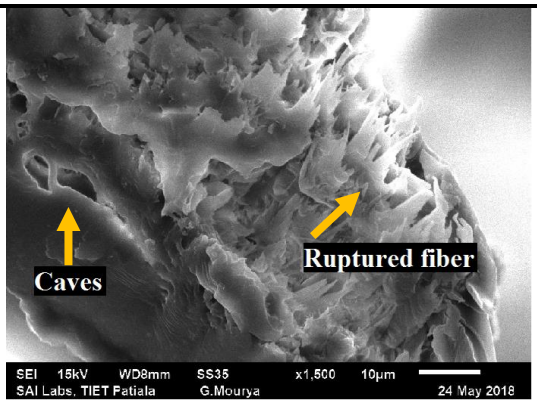
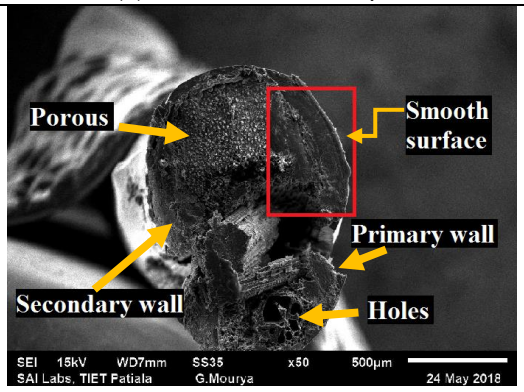
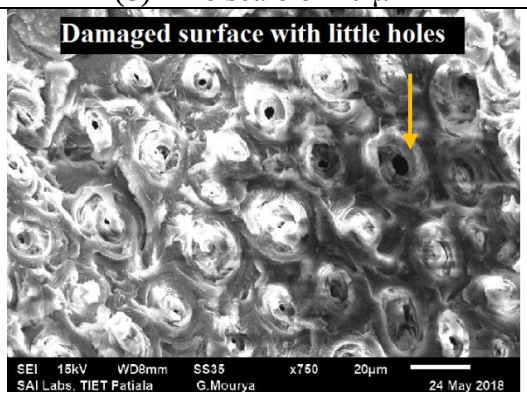
The untreated fibre before chemical treatments of Agave Americana fibre is shown in Figure 3.1(a). With the alkali treatment, the hemi-cellulose and lignin get ceased and this treatment lessens the volume of the fibre and the colour of the fibre turns to yellowish (Figure 3.1(b)) (Mylsamy & Rajendran, 2011). The removal of impurities and reduction in the diameter of the fibre enhance the fibre-matrix adhesion.

(a) (b)

Figure 3.1 Chemical treatments of Agave Americana fibre (a) Untreated fibre and (b) 2% NaOH treated fibre

The untreated fibres have (Table 3.1(a & b)) rough and damaged surface with more ruptured fibres, holes and caves whereas after treating agave Americana fibre with NaOH, the ruptured fibres are reduced (Table 3.1(c & d)) and the holes are turned into porous.

Table 3.1 SEM images of the fibre's cross-section: (a & b) Untreated fibre and (c & d) NaOH treated fibre

SEM / Fibre	SEM Micrograph	
Un-treated	 (a) Line scale of 50 µm	 (b) Line scale of 10 µm
2% (w/v) NaOH treated	 (c) Line scale of 500 µm	 (d) Line scale of 20 µm

3.2 HORNIFICATION

The hornification is known to increase the cross link within fibre microstructure. This in turn results in a better fibre matrix bond and improvement of the hydro-phobicity in the composites. In the horification process, alternate wet and drying cycles were applied to study its influence on the water uptake behaviour.

Water retention value (WRV) is the standardized method in order to calculate the amount of water retained in the fibre. WRV is calculated from wet mass obtained after the wetting process. Dry mass obtained after drying process is subtracted from the wet mass and the resultant is divided by the dry mass. The water retention value is expressed as (Ballesteros *et al.*, 2017).

$$\text{Water Retention Value} = \frac{(m_w - m_i)}{m_i} \tag{3.1}$$

Where m_w is mass during wet process and m_i is mass obtained after drying process.

3.2.1 Alkali Hornification

In the alkali hornification, the fibre bundles weighing 200 gm were measured and were soaked with 8% (w/v) (Oudiani *et.al*, 2011) alkali solution in the water bath. The temperature range and duration selected during the experiment was:

- For wetting cycle: 23°C temperature and 2 hours of duration
- For drying cycle: 110°C temperature and 5 hours of duration

The similar conditions have been referred in the literature (Kato & Cameron, 1999). The temperature was gradually decreased in order to avoid thermal shocks. Therefore, after 4 hours of drying in the remaining last 1 hour, the temperature was reduced gradually. The wetting and drying process (Figure 3.2) together form 1 cycle. In this way, 4 cycles were carried out in order to reduce the diameter of the fibre so that the dimensional stability is improved. The numbers of cycles were decided after the experimental work (Ballesteros *et al.*, 2017).

(a) (b)

Figure 3.2 Alkali hornification procedure: (a) Wetting process in the water bath at 23°C and (b) Drying process in the furnace at 110°C

Figure 3.3 shows the fibre textures and the end of successive wet and drying cycles. The fibres conditioned after the 4th cycle was further used for the preparation of specimen. During hornification process, there is an accelerated ageing of the fibre. The swelling of cellulose results in increased dimensional stability in each cycle. The irreversible hydrogen bond is formed with the repetition of the cycles which results in reduced water retake in the inter-fibrillar space (Ballesteros *et al.*, 2017).

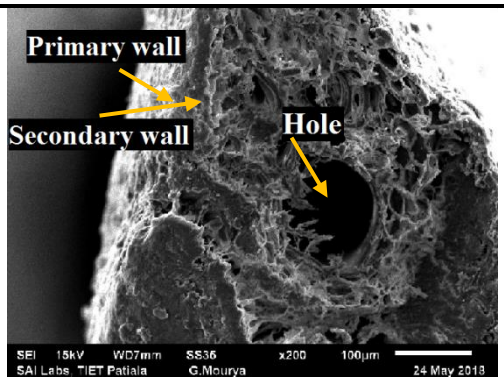
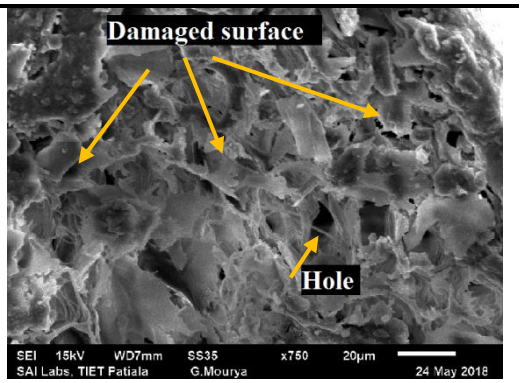
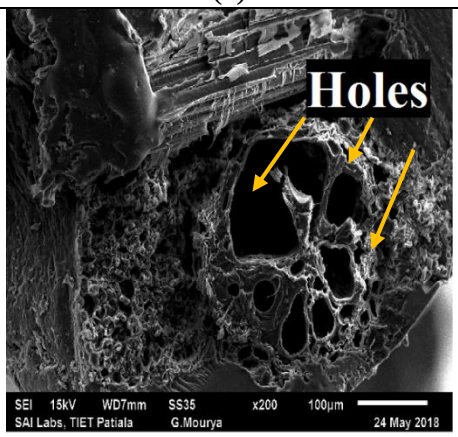
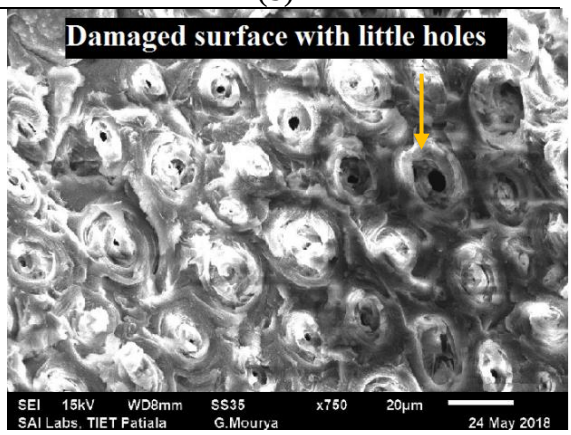
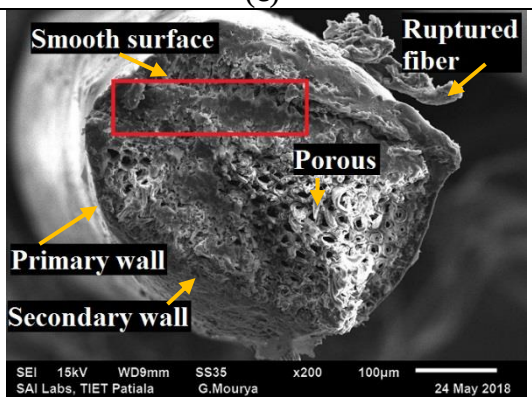
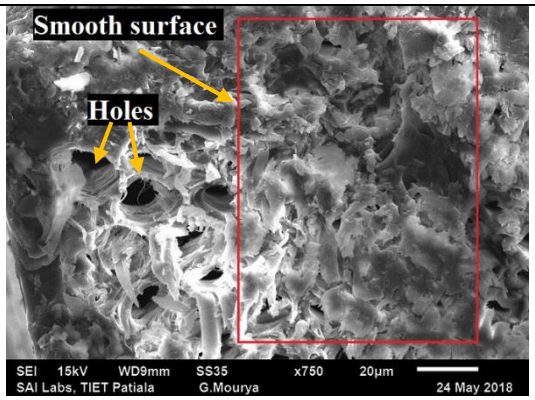
(a) (b) (c) (d)

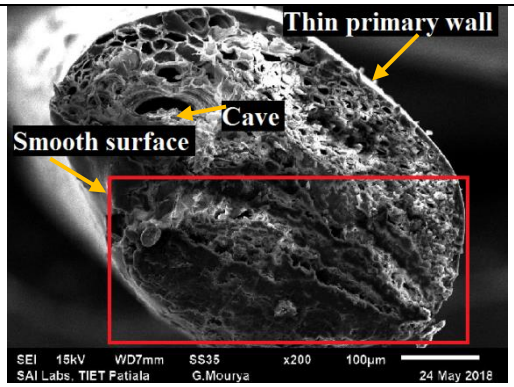
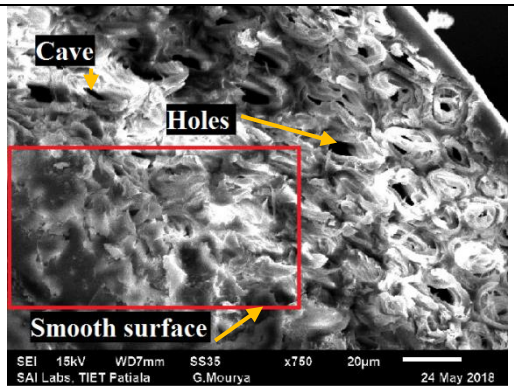
Figure 3.3 The texture of the fibres after (a) 1st cycle, (b) 2nd cycle, (c) 3rd cycle and (d) 4th cycle

The shrinkage of the fibre was observed visually with each cycle which leads to the good fibre-matrix adhesion. The increased overall shrinkage results in reduced water uptake. Alkali solution also reduces the cellulose and lignin contents. This was observed through change of color from flax color to bumblebee color.

Table 3.2 represents the SEM images of the cross-section of the alkali hornified fibres. In the 1st cycle (a & b), holes and damaged surface can be seen, but as the number of cycles are increased, the holes are divided themselves into smaller holes, porous and caves after each cycle whereas the damaged surface is turned towards smooth surface. In the 4th cycle (g & h), few holes can be observed with thin primary wall. Hence, alkali hornification helps in reducing the holes in the fibre which leads to less water intake.

Table 3.2 SEM images of the fibre's cross-section after Alkali hornification (a & b) 1st cycle, (c & d) 2nd cycle, (e & f) 3rd cycle and (g & h) 4th cycle

SEM / A.H.	SEM Micrograph (Line scale of 100 µm)	SEM Micrograph (Line scale of 20 µm)
1st cycle	 (a)	 (b)
2nd cycle	 (c)	 (d)
3rd cycle	 (e)	 (f)

4th cycle	 (g)	 (h)

3.2.2 Water Hornification

In the water hornification, the fibres are soaked in the distilled water and then allowed to dry in the furnace. The similar temperature range and duration were selected as were in the alkali hornification.

Although visually it is observed that the water treatment resulted in reduction of the fibre diameter, the magnitude of this reduction was lesser as compared to the fibre diameter as noticed during the alkali hornification. Single cycle of water hornification is shown in Figure 3.4. The images of the fibre after each cycle are shown in Figure 3.5. The swelling of cellulose and removal of lignin along with the temperature conditions lead to a change in the color from golden to brown as evidenced visually.

(a) (b)

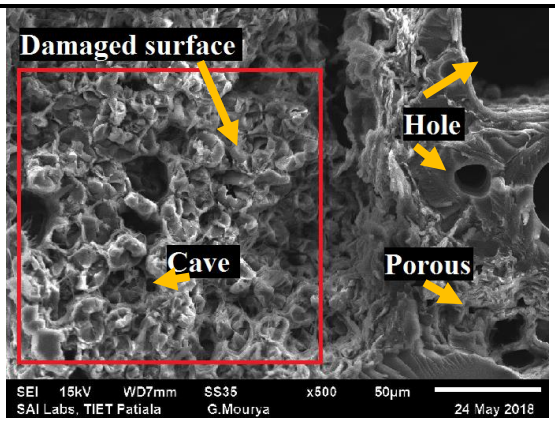
Figure 3.4 Single cycle of water hornification: (a) Wetting process in the water bath at 23°C and (b) Drying process in the furnace (inside view) at 110°C

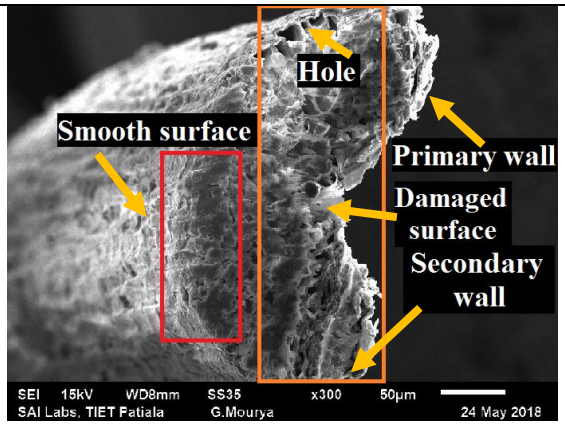
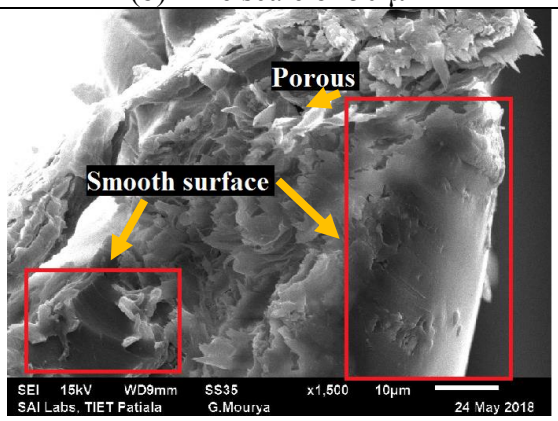
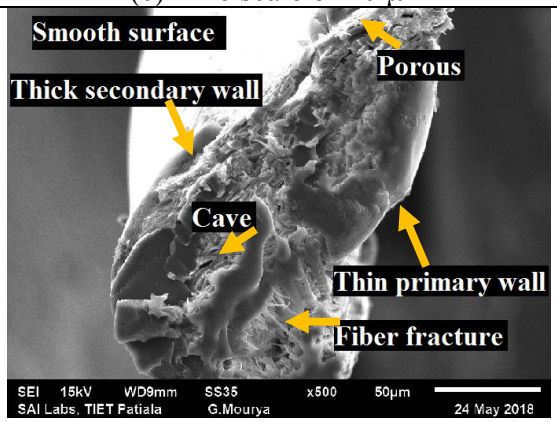
(a) (b) (c) (d)

Figure 3.5 Fibre textures after (a) 1st cycle, (b) 2nd cycle, (c) 3rd cycle and (d) 4th cycle

Table 3.3 represents the cross section of the fibres for each cycle during water hornification. After the 1st cycle (a), a rough texture and porous can be observed with a plenty of holes. After 2nd cycle (b), small holes are noticeable, after 3rd cycles, a texture smoothening is noticeable with porosity. Finally, after the 4th cycle, across the smoothening of cross section results in fewer porous and caves as compared to the other cycles. The textural improvement through reduction in porosity is also noticeable.

Table 3.3 SEM images of the fibre's cross-section after water hornification (a) 1st cycle, (b) 2nd cycle, (c) 3rd cycle and (d) 4th cycle

SEM / W.H	SEM Micrograph
1st cycle	 (a) Line scale of 50 µm

2nd cycle	 (b) Line scale of 50 µm
3rd cycle	 (c) Line scale of 10 µm
4th cycle	 (d) Line scale of 50 µm

CHAPTER 4

SPECIMEN FABRICATION FOR EXPERIMENTATION

The Agave Americana fibre composite sheets were fabricated using hand lay-up method. After calculating the required number of specimen for different conditions, the specimen was machined according to ASTM D570-98.

4.1 HAND LAY-UP METHOD

Four different types of composites with (a) untreated fibre (b) alkali hornified fibre (c) NaOH treated fibre and (d) water hornified fibre were prepared by the hand lay-up method. The hand lay-up method is depicted below pictorially step by step.

- **Preparation of the Epoxy Resin**

The matrix was prepared mixing epoxy resin LY56 and hardener HY 951 at a ratio of 10:1 according to the industrial specification (Chapter 1) as shown in Figure 4.1. The material was procured from Huntsman India Ltd., Bangalore.

(a)

(b)

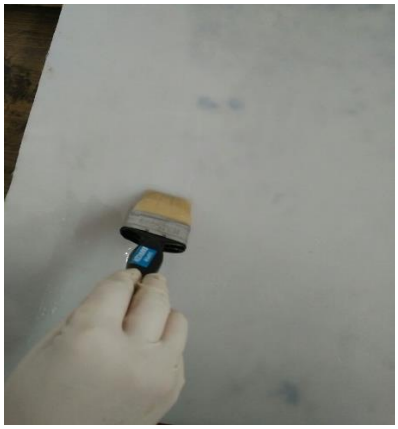
Figure 4.1 Preparation of the resin (a) Epoxy and hardener and (b) Mixing of epoxy and hardener (10:1) with the glass rod

- **Placement of Fibre**

The resin was spread on the teflon sheet with the help of a brush (Figure 4.2 (a)) because teflon sheet has fluorine atoms on it, these atoms do not let other material stick to its surface. So, as soon as the material get cured it was easy to peel the sheet. The Agave Americana fibres were placed one by one (Figure 4.2 (b & c)) evenly on the layer of the resin. In order to ensure a structurally uniform distribution of fibres across all the specimen, a fixed ratio of fibre weight to the matrix volume was determined through several trails. While maintaining the thickness according to ASTM, it was ensured in these trials that fibres get evenly dispersed within matrix.

After the placement of the fibres, roller was used (Figure 4.2 (d)) to ensure the evenness of the fibre distribution on the surface, the second layer of the epoxy resin was applied afterwards (Figure 4.2 (e)). Hence, each specimen contains a single layer of unidirectional fibres.

The uncured sheet was covered with another teflon sheet from the top and a uniform mass was placed on the top for the compression and curing purpose (Figure 4.2 (f)). The uncured sheet was left under the room temperature for a span of 7 days as prescribed under manufacturer's catalogue.



(a)

(b)

(c)

(d)

(e)

(f)

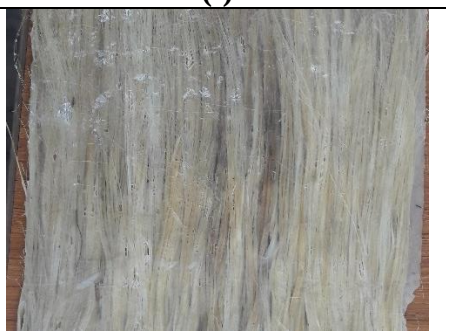
Figure 4.2 Fabrication by hand lay-up method: (a) Resin applied on the teflon sheet, (b) & (c) Step by step reinforcement of the fibre, (d) Roller is applied on the reinforced fibre, (e) Resin was applied after reinforced fibre and (f) Uncured fabricated sheet

4.2 FABRICATION OF AFRC SHEET

The prepared fibres (Chapter 3) were used for the fabrication of the sheets using hand lay-up method (Section 4.1). The cured and uncured sheets are represented in Table 4.1. The cured sheet (Table 4.1 (b, d, f & h)) is stiffer and takes a structure of glass type material. As the epoxy and hardener are mixed, it goes under exothermic reaction which leads to further increase in the temperature and the golden colour is slightly converted to a yellowish tone due to the further removal of the remained lignin content.

Table 4.1: Fabricated Agave Americana fibre reinforced sheets (uncured & cured)

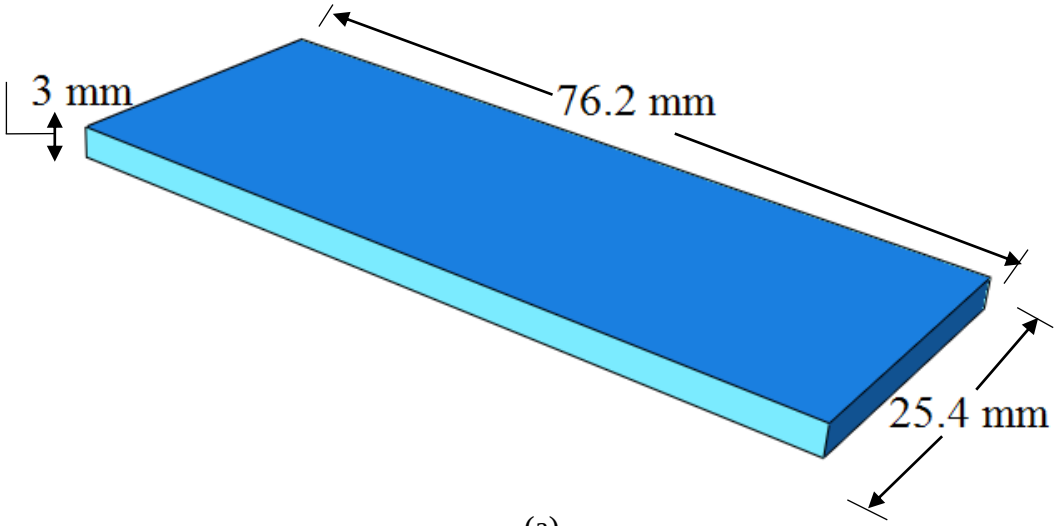
Treatment	Uncured sheet	Cured sheet
Untreated fibre sheet	 **(a)**	 **(b)**
2% (w/v) NaOH treated fibre sheet	 **(c)**	 **(d)**

Alkali hornified fibre sheet		
	(e)	**(f)**
Water hornified fibre sheet		
	(g)	**(h)**

4.3 SPECIMEN PREPARATION

Once the sheets are cured, specimens are cut with the help of the hand grinder. The specimens are cut according to the dimensions prescribed under ASTM D 580-98.

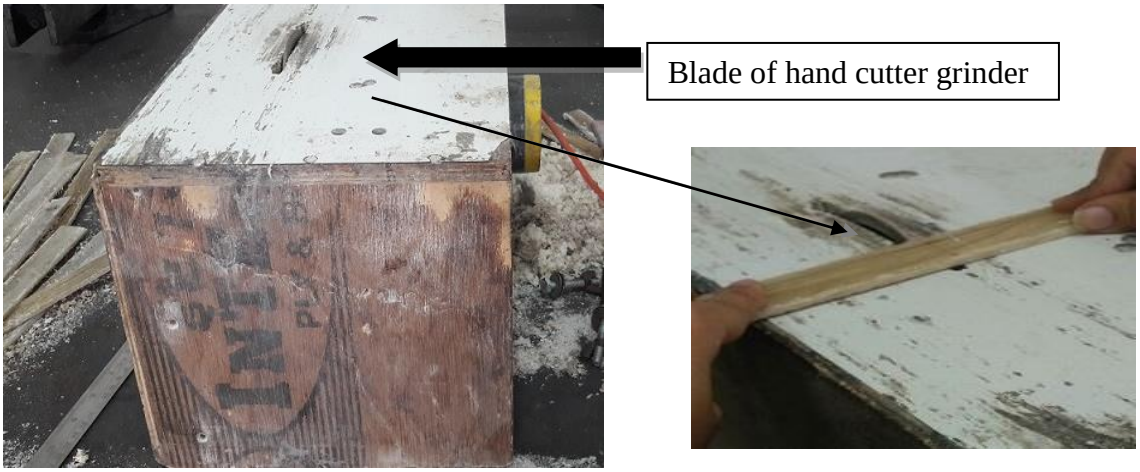
According to this standard, there should be three test specimens for each condition. The recommended specimen size (Figure 4.3) is as Length = 76.2 mm, Width = 24.5 mm and Thickness = 3–5 mm. The specimen should be balanced using analytical balance which should be capable of reading of 0.0001 g.



(a)

Figure 4.3 Dimensioning of test specimen

According to ASTM D570-98, in order to prevent the loss of mass gain while taking the specimen from one place to the other, it should be properly carried into the water filled container having same temperature, whereas the specimen to be tested under humid conditions should be taken in the sealed container. The specimens from the cured sheets are prepared with the help of hand grinder as represented in Figure 4.4. The untreated and treated sheets are shown in Figure 4.5.



(a)

Figure 4.4 Sheet cutting: (a) Preparation of specimen with the help hand grinder (Courtesy: TIET, Patiala)

(a)

(b)

(c)

(d)

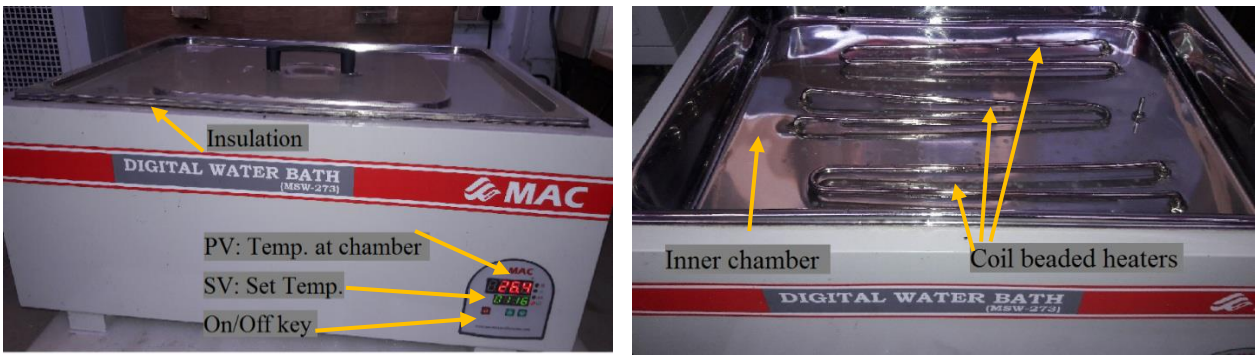
Figure 4.5 Specimen (a) Untreated AFRCS, (b) NaOH treated AFRCS, (c) Alkali hornified AFRCS and (d) Water hornified AFRCS

4.4 EXPERIMENTAL SET-UP

The specimens were tested under different environmental conditions. The water bath, cryostat bath and environment chamber helped in maintaining those conditions, whereas, weighing balance was used to measure the mass gain. Furnace was used in the process of alkali hornification and water hornification.

4.4.1 Digital Water Bath

The digital water bath (Figure 4.6) is fabricated for carrying out test in growing field of industrial research. The outer wall is made up of electro galvanized powder coated steel and the inner chamber is made up of thick stainless steel with mirror polished and is insulated with high grade glass wool fitted all around.



(a) (b)

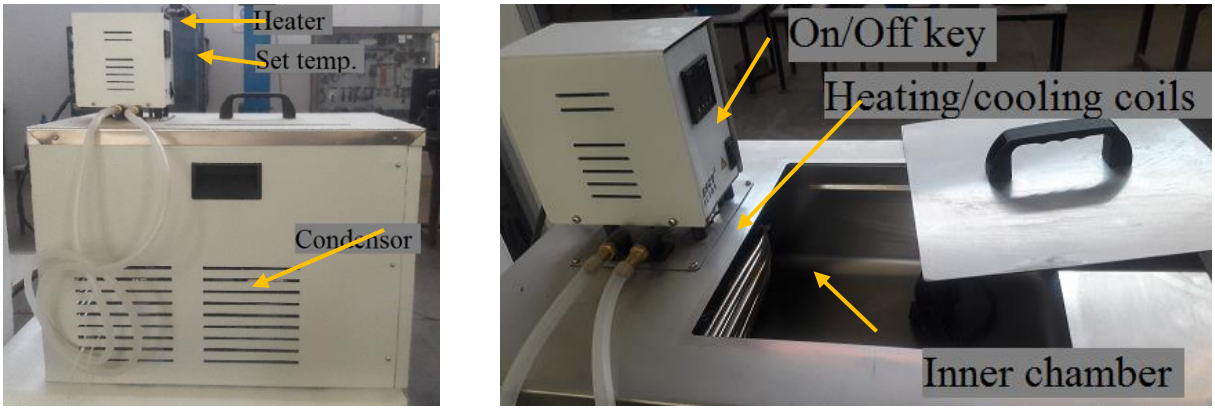
Figure 4.6 (a) Digital water bath "MAC" (MSW-273 SL) and (b) Inner chamber (Courtesy: TIET, Patiala)

There are coil beaded heaters inside the chamber with a temperature controller. The ambient temperature range is 5–99.9°C with an accuracy of ±0.5°C.

4.4.2 Cryostat Bath

The Cryostat bath (Figure 4.7) is required for carrying out test in the condition below and above 0°C. The temperature range is -10°C to 50°C. In regard to set working conditions below 0°C, ethylene glycol was mixed with distilled water in the ratio of 1:1.

For achieving high temperature, the condenser automatically gets off and starts operating when the required temperature is achieved. The heating coils serve both the purposes of heating and cooling. So, the system works on high accuracy.



(a) (b)

Figure 4.7 (a) Digital cryostat bath and (b) Inner chamber
(Courtesy: TIET, Patiala)

4.4.3 Environment Chamber

A forced air circulated environment chamber (Figure 4.8) was used for the specimen to a humid climate. The working temperature range of this equipment was from ambient temperature to 80°C with temperature constancy of ±1.0°C. The humidity range is between 0% R.H to 100% R.H. For the present experiments, four different temperatures *viz.* 25°C, 50°C & 75°C are chosen under a relative humidity of 70%.

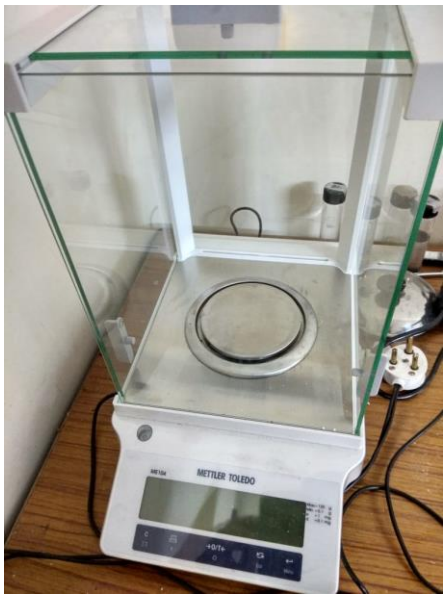
Figure 4.8 Environment chamber ACM-78091S (Courtesy: TIET, Patiala)

4.4.4 Weighing Balance

A high precision weight balance (Make: Mettler Toledo) (Figure 4.9 (a)) was used to weigh the specimen. The precision balance could measure a weight ranging from 0.1 gm to 120 gm respectively with least count of 0.2 mg.

4.4.5 Furnace

The furnace (Figure 4.9 (b)) was used to heat the fibres during alkali and water hornification. The maximum temperature is 110°C.



(a)

(b)

Figure 4.9 (a) Weighing balance METTLER TOLEDO and (b) Furnace (Courtesy: TIET, Patiala)

4.4.6 Scanning Electron Microscopy (SEM)

Scanning electron microscopy i.e. SEM (Figure 4.10 (c)) was used to scan the cross section of the fibre with a beam of high energy electrons. When the electrons interact with the atoms present in the specimen, various signals are produced and this contains information about the specimen's surface, cross section, composition and various properties. It can produce the specimen's surface images with high-resolution. The specimen (Figure 4.10 (a)) were first prepared dipping half in the epoxy resin and then, were further coated (Figure 4.10 (b)) in the machine so that electrons can fall on that coating with high energy and signals can be transferred easily. After this, the specimens were scanned in the SEM machine (Figure 4.10 (c))

(a)

(b)

(c)

Figure 4.10 (a) Specimen used in SEM, (b) Instrument used for coating and (c) SEM machine (Courtesy: TIET, Patiala)

4.5 Test Matrices

In order to calculate the number of test specimen, a test matrix was prepared considering the types of specimen according to fibre treatment method and temperature conditions. For each test condition, in order to have a better approximation and reduce the effect of manufacturing inaccuracies, 3 specimens are considered for each condition and treatment. A total of 84 specimens were required for the experimentation. Table 4.2 shows a detailed test matrix.

Table 4.2 Test matrices

Bath type	Bath name	Bath temp (°C) with sample no.						Holding Time (days)	Specimen required
Cryostat bath		0°C							
	Untreated (UN1)	3						39	3
	NaOH treated (2%) (T1)	3							3
	Water hornified (T2)	3							3
	Alkali hornified (T3)	3							3
									Specimen count(a) = 12
Water bath		25°C	50°C	75°C	25 °C	50 °C	75 °C		
	Untreated (UN2)	3	3	3	33	22	16		9
	NaOH treated (2%) (T4)	3	3	3					9
	Water hornified (T5)	3	3	3					9
	Alkali hornified (T6)	3	3	3					9
									Specimen count(b) = 36
Environ--ment chamber		25°C	50°C 70%	75°C R.H	25°C 70%	50°C R.H	0°C		
	Untreated (UN3)	3	3	3	34	25	19		9
	NaOH treated (2%) (T7)	3	3	3					9
	Water hornified (T8)	3	3	3					9
	Alkali hornified (T9)	3	3	3					9
									Specimen count(c) =36
									Total specimen (a +b +c) = 84

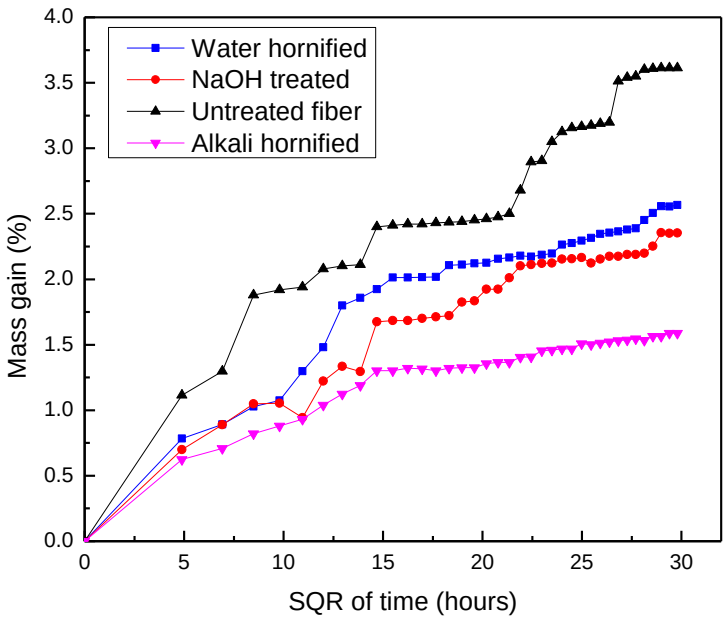
CHAPTER 5

RESULTS & DISCUSSIONS

5.1 EXPERIMENTAL RESULTS

5.1.1 Exposure in Freezing Environment

Specimens were immersed at 0°C in the cryostat bath. They were dispersed for around 45 days. The percentile mass gain was recorded at different time intervals. The mass gain at near saturation was 1.587895%, 2.352021%, 2.567501% & 3.613452% for the alkali hornified specimen, NaOH treated specimen, water hornified specimen and untreated specimen, respectively. Hence, the mass gain is least in the alkali hornified specimen and highest in the untreated specimen (Figure 5.1).



(a)

Figure 5.1 Water uptake for chemically treated and untreated Agave Americana reinforced specimen as a function of square root of immersion time at 0°C

5.1.2 Exposure in Controlled Humidity and Temperature Environments

Figure 5.2 represents the mass gain of the Untreated, Water hornified, NaOH treated and alkali hornified specimens when immersed in the water bath at 25°C, 50°C & 75°C. The chemical treatments partly attributed to the disappearance of the hydrophilic nature of the agave Americana reinforced specimen.

It is observed that, the mass gain because of the water uptake is maximum at high temperature. The mass gain is maximum in the untreated fibre's specimen & least in the alkali hornified specimen.

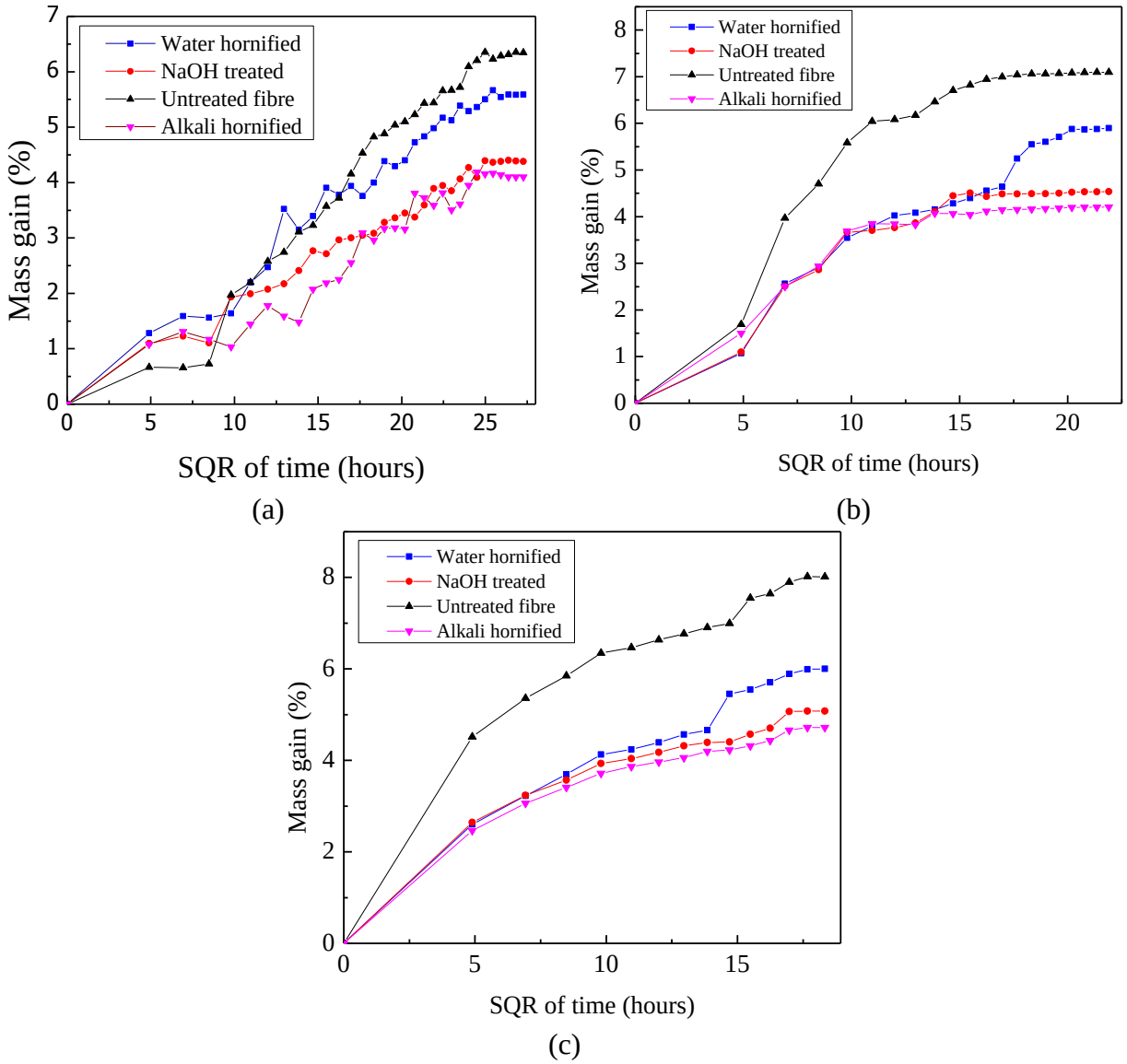


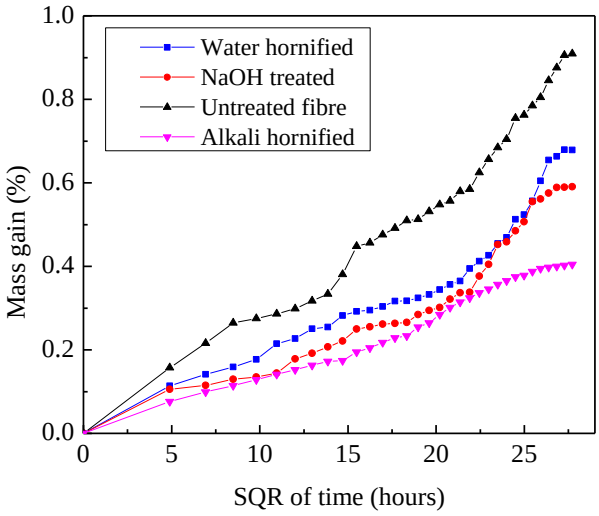
Figure 5.2 Water uptake for chemically treated and untreated Agave Americana reinforced specimen as a function of square root of immersion time at (a) 25°C, (b) 50°C and (c) 75°C

The mass gain of the alkali hornified specimen is least as compared to others i.e. 4.10085% at 25°C, 4.3154% at 50°C & % at 4.7215% at 75°C. The mass gain in NaOH treated specimen is 4.38215% at 25°C, 4.534625% at 50°C & 5.079405% at 75°C. The mass gain in water hornified specimen is 5.58687% at 25°C, 5.89667% at 50°C & 6.00067% at 75°C whereas the mass gain in untreated fibre specimen is 6.3423% at 25°C, 7.094787% at 50°C & 8.015235% at 75°C.

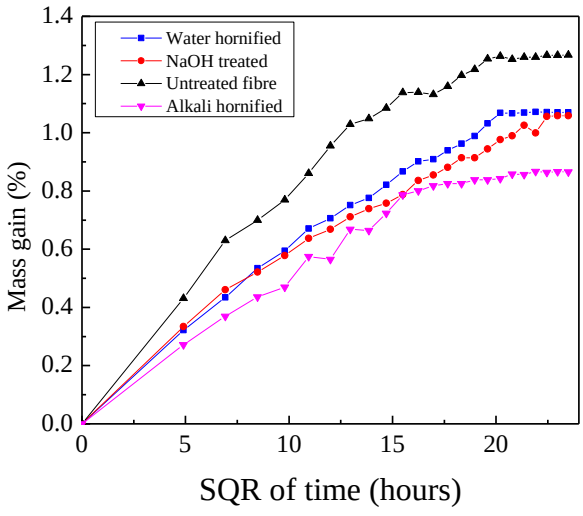
5.1.3 Environment Chamber at 70% R.H

After keeping specimens at 70% R.H with different temperature conditions i.e. 25°C, 50°C & 75°C. From the figure 5.3 it is observed that, at high temperature the mass gain is maximum. Moreover, the chemically treated specimens show resistant to water uptake as compare to the untreated fibre's specimen.

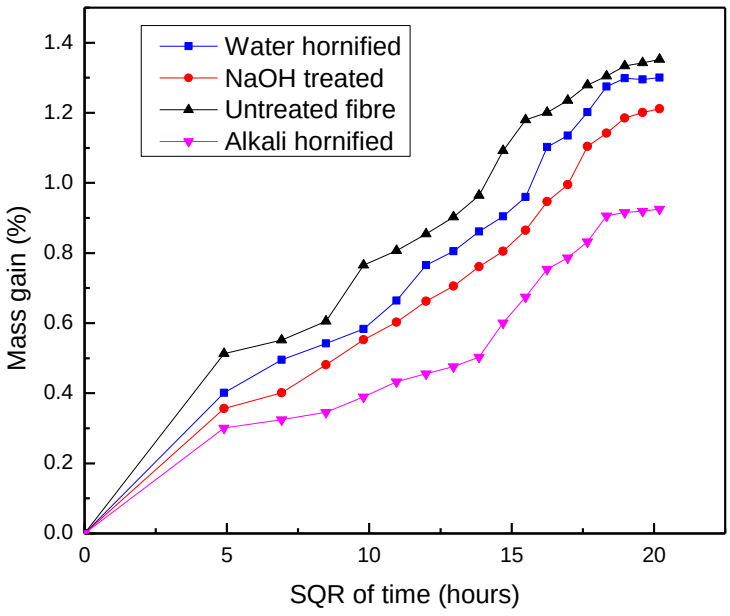
The results for the mass gain at 70% R.H for the: alkali hornified specimen is least i.e. % at 25°C, % at 50°C & % at 75°C, for NaOH treated specimen is 0.591% at 25°C, 1.058% at 50°C & 1.211% at 75°C, for water hornified specimen is 0.6788% at 25°C, 1.07% at 50°C & 1.3% at 75°C whereas for the untreated fibre specimen is 0.908945% at 25°C, 1.26717% at 50°C & 1.5324612% at75°C.



(a)



(b)

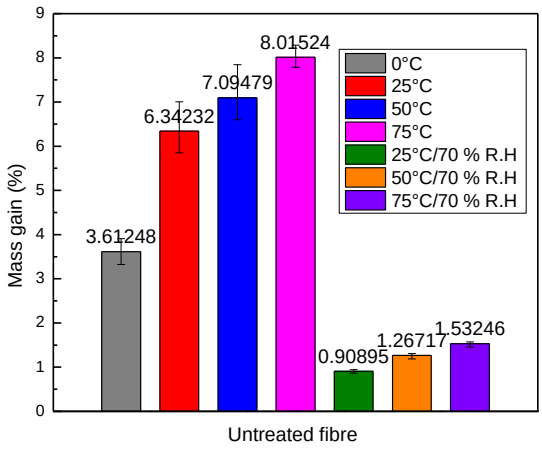


(c)

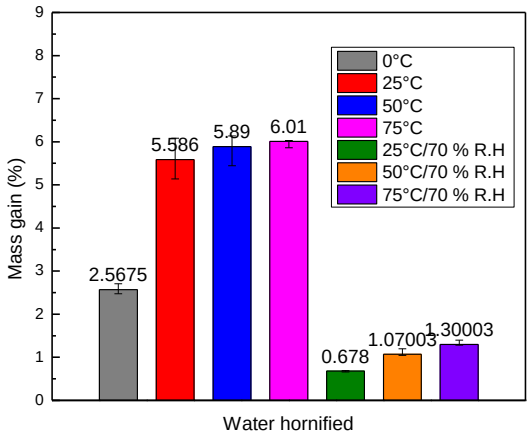
Figure 5.3 Moisture gain for chemically treated and untreated Agave Americana reinforced specimen as a function of square root of immersion time at (a) 25°C/70% R.H, (b) 50°C/70% R.H and (c) 75°C/70% R.H

5.2 COMPARISON OF MASS GAIN AT DIFFERENT EXPOSURE ENVIRONMENTS

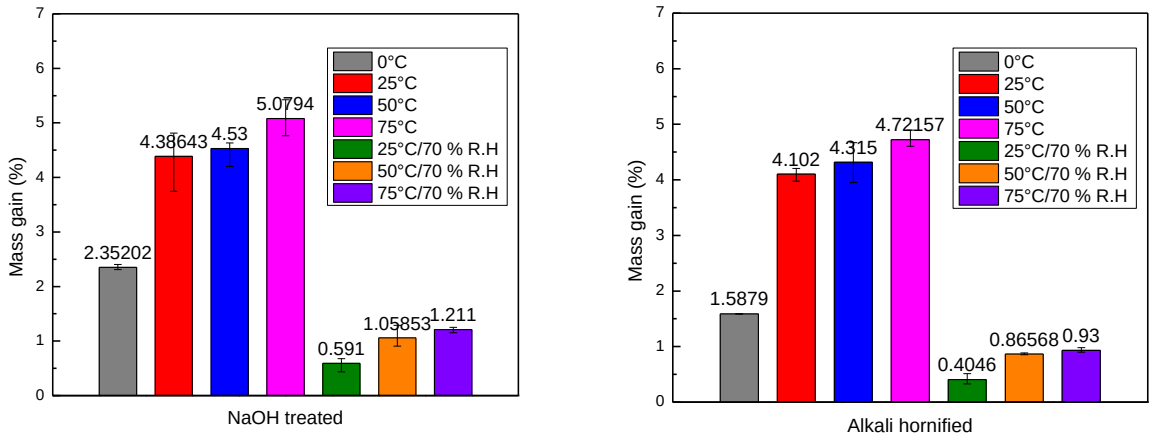
Figure 5.4 compares the final mass gain at that at environmental conditions separately as a result of each type of fibre treatment. One can easily see the effect of elevated temperatures on the rate of diffusion and final mass gain. At high temperatures the mass gain is maximum and the rate of saturation is also faster when compared to the low temperature. All the bars in a single graph are plotted for the mass gain for same time duration of different exposure conditions.



(a)



(b)



(c) (d)

Figure 5.4 Mass gain at different environmental conditions for (a) Untreated fibre (b) Water hornified (c) NaOH treated, and (d) Alkali hornified specimens

5.2 MOISTURE DIFFUSION ANALYSIS

Inverse Fickian Moisture diffusion theory is being applied first to calculate the diffusivity of homogenized fibre matrix system composite sheets from the experimental mass gain curves. Once the diffusivity is found, the diffusion based finite element modelling (FEM) procedure was used to analyze the theoretical mass gain. The FEM results were compared with the experimental results subsequently.

5.2.1 Calculation of Diffusion Coefficients

Moisture diffusion theory is being applied on the unidirectional agave Americana reinforced composite sheets. A 3-D model of the ARCS is created and mass gain percentage is obtained by finite element modelling. Further, the modelling results are compared with the experimental results.

5.2.2 Determination of Diffusion Coefficients

Moisture concentration is a function of time t, and distance from the surface. To carry out moisture diffusion analysis in finite element modeling, diffusion coefficient must be known. The procedure stated in (Starik et al., 2002) was employed to calculate the diffusivity of composite sheet

On the experimental mass gain curve, slope (S) is calculated as:

$$S = \frac{\frac{M_i}{M_{max}} \times c}{t} \tag{6.1}$$

Where M_i is the minimum mass gain, M_{max} is the maximum mass gain, c is the thickness and t stands for SQR of time (hours). Using this slope, the diffusivity (D) can be obtained by

$$D = \pi \times \frac{1}{16} \times \frac{(S)^2}{3600} \tag{6.2}$$

The edge correction factor (f_{ssc}) is calculated by,

$$f_{SSC} = 1 + 0.54 \times \left(\frac{c}{b}\right) + 0.54 \times \left(\frac{c}{a}\right) + 0.33 \times \left(\frac{c \times c}{b \times a}\right) \tag{6.3}$$

Where a, b, c are length, width and thickness of the specimen, respectively. The effective diffusion coefficient (D_{eff}) is calculated by

$$D_{eff} = D \times (f_{SSC})^2 \tag{6.4}$$

Where f_{ssc} stands for edge correction factor and D is the diffusivity

Table 5.1 The effective diffusion cofficient, calculated from the Equation 6.4

Sr. No.	Water Hornified Specimen (m^2/sec)	NaOH treated Specimen (m^2/sec)	Untreated Specimen (m^2/sec)	Alkali Hornified Specimen (m^2/sec)
0°C (Cryostat bath)	1.145×10^{-12}	9.94×10^{-13}	1.55×10^{-12}	1.76×10^{-12}
25°C (Water bath)	1.168×10^{-12}	9.25×10^{-13}	1.094E-12	1.51×10^{-12}
50°C (Water bath)	1.537×10^{-12}	2.28×10^{-12}	2.57×10^{-12}	3.56×10^{-12}
75°C (Water bath)	4.02×10^{-12}	6.54×10^{-12}	7.66×10^{-12}	6.58×10^{-12}
25°C/70%R.H (Environmental Chamber)	3.91×10^{-13}	3.59×10^{-13}	5.43×10^{-13}	5.84×10^{-13}
50°C/70%R.H (Environmental Chamber)	2×10^{-12}	2.12×10^{-12}	2.72×10^{-12}	2.12×10^{-12}
75°C/70%R.H (Environmental Chamber)	1.07×10^{-12}	1.03×10^{-12}	1.12×10^{-12}	1.02×10^{-12}

5.2.3 Finite Element Modelling

Once we determine the effective moisture diffusivity D_{eff} of the composite. The moisture diffusion is simulated inside a three-dimensional lamina representing with all of its edges suddenly exposed from dry state to the ambient moisture under an assumption that the moisture diffuses through the gaps in polymer structure following Fickian diffusion according to the relation.

$$\frac{\partial c}{\partial t} = D_{eff}\left(\frac{\partial^2 c}{\partial x^2} + \frac{\partial^2 c}{\partial y^2} + \frac{\partial^2 c}{\partial z^2}\right) \tag{6.5}$$

Here D_{eff} is the computed diffusivity of the composite.

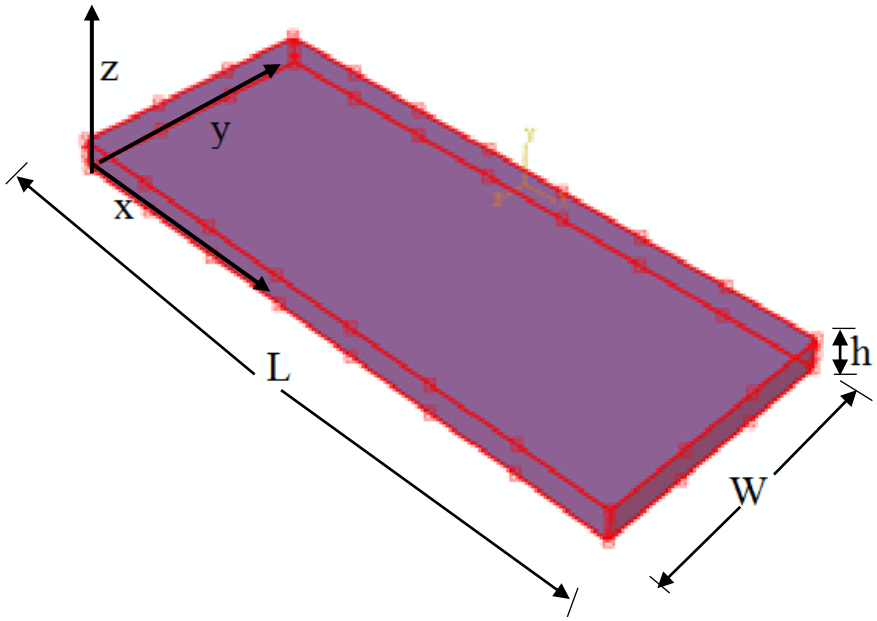


Figure 5.5 Dimensioning of the specimen

Figure 5.5 shows the dimensions of the specimen. Here, *L* is the length of the specimen along the *x* axis, *W* is the width of the specimen along y axis and h is the height of the specimen along the *z* axis.

Moisture boundary: The initial moisture concentration (c) inside the composite is null. The moisture all the six moisture exposed *planes* attains the boundary moisture (c_{amb}) as soon as it is exposed to the moist environment.

Initial Condition $c = 0 \quad (0 \le x \le L, 0 \le y \le W, 0 \le z \le h, \forall t = 0)$

Boundary Condition $c = c_{amb} \quad [(x = 0, L), (y = 0, w), (z = 0, h) \forall t > 0)$

The final weight gain , $M(\Omega_m, t)$, by the FRPC sheet due to moisture ingression at time *t* is calculated through the volume integral of the moisture concentration at that time. That is

$$M(\Omega_m, t) = \int_{\Omega_m} c(x, y, z, t)\, dV \qquad (6.6)$$

5.3 COMPARISON BETWEEN NUMERICAL (FEM) AND EXPERIMENTAL WORK

5.3.1 Cryostat Bath at 0°C

Figure 5.6 shows the comparison between the FEM and the experimental work at 0°C in the cryostat bath.

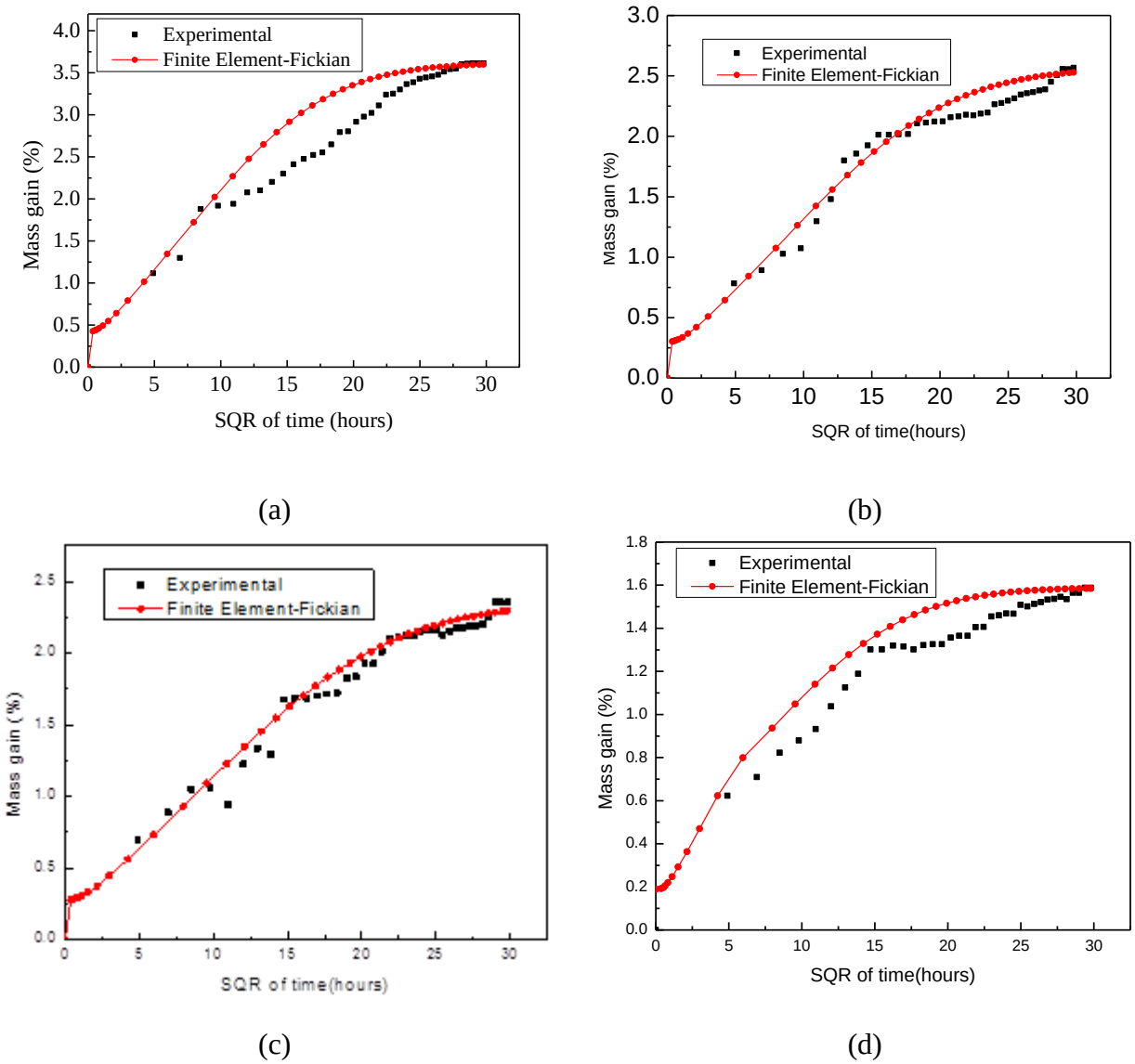


Figure 5.6 Comparative graphs between FEM and experimental work at 0°C (a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

5.3.2 Water Bath at 25°C

Figure 5.7 shows the comparison between the FEM and the experimental work at 25°C in the water bath.

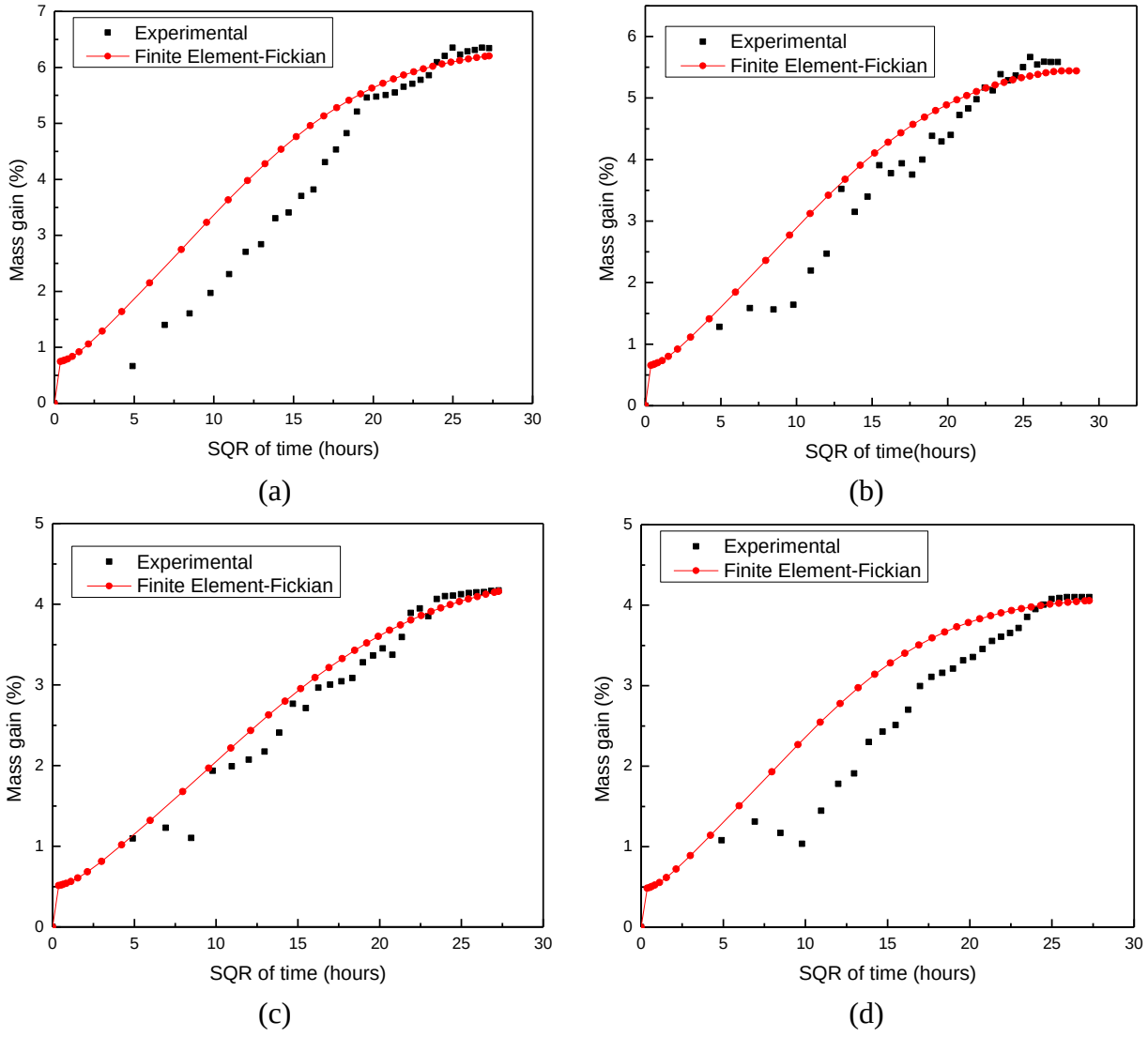


Figure 5.7 Comparative graphs between FEM and experimental work at 25°C (a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

5.3.3 Water Bath at 50°C

Figure 5.8 shows the comparison between the FEM and the experimental work at 50°C in the water bath.

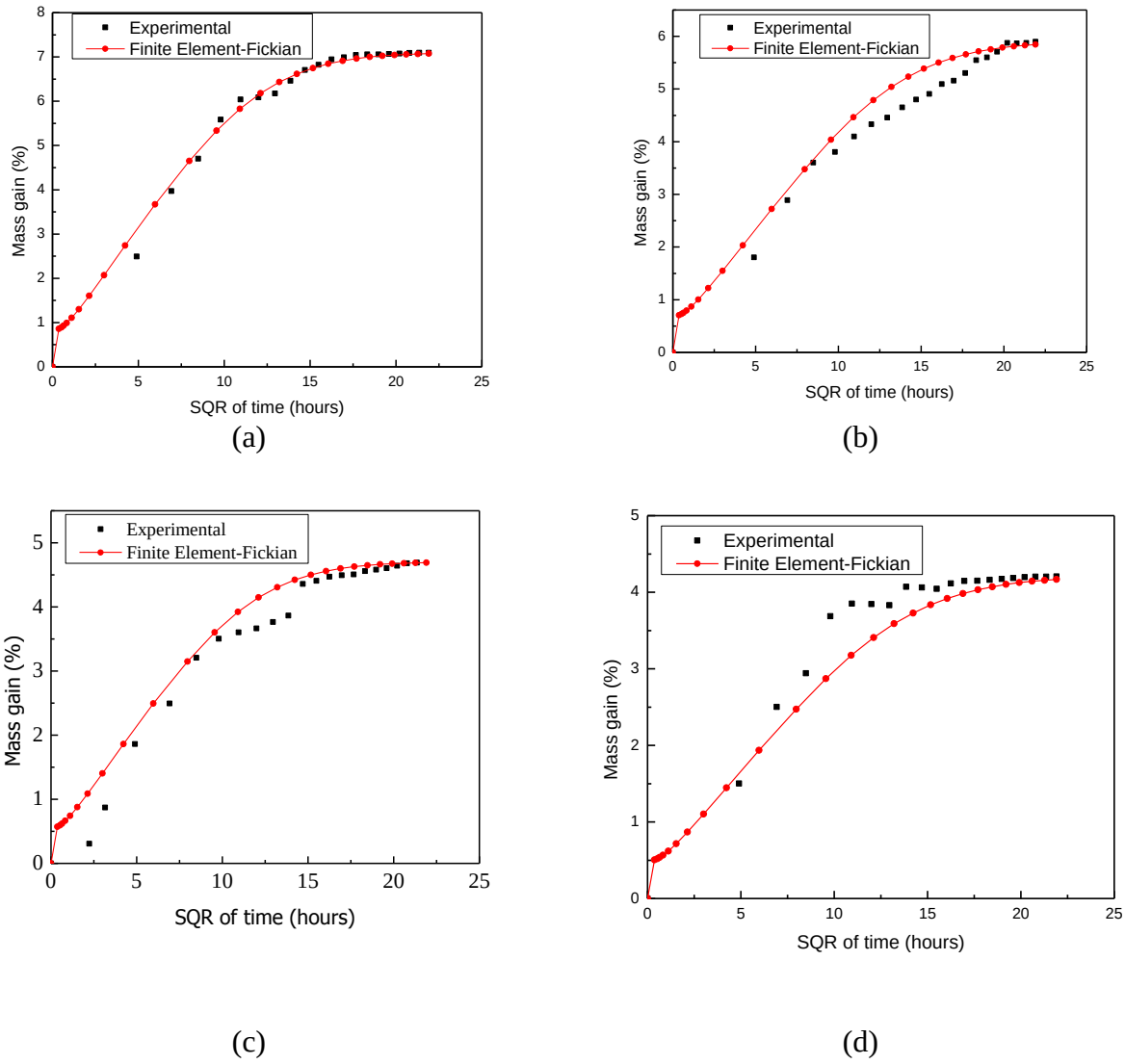


Figure 5.8 Comparative graphs between FEM and experimental work at 50°C (a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

5.3.4 Water Bath at 75°C

Figure 5.9 shows the comparison between the FEM and the experimental work at 75°C in the water bath.

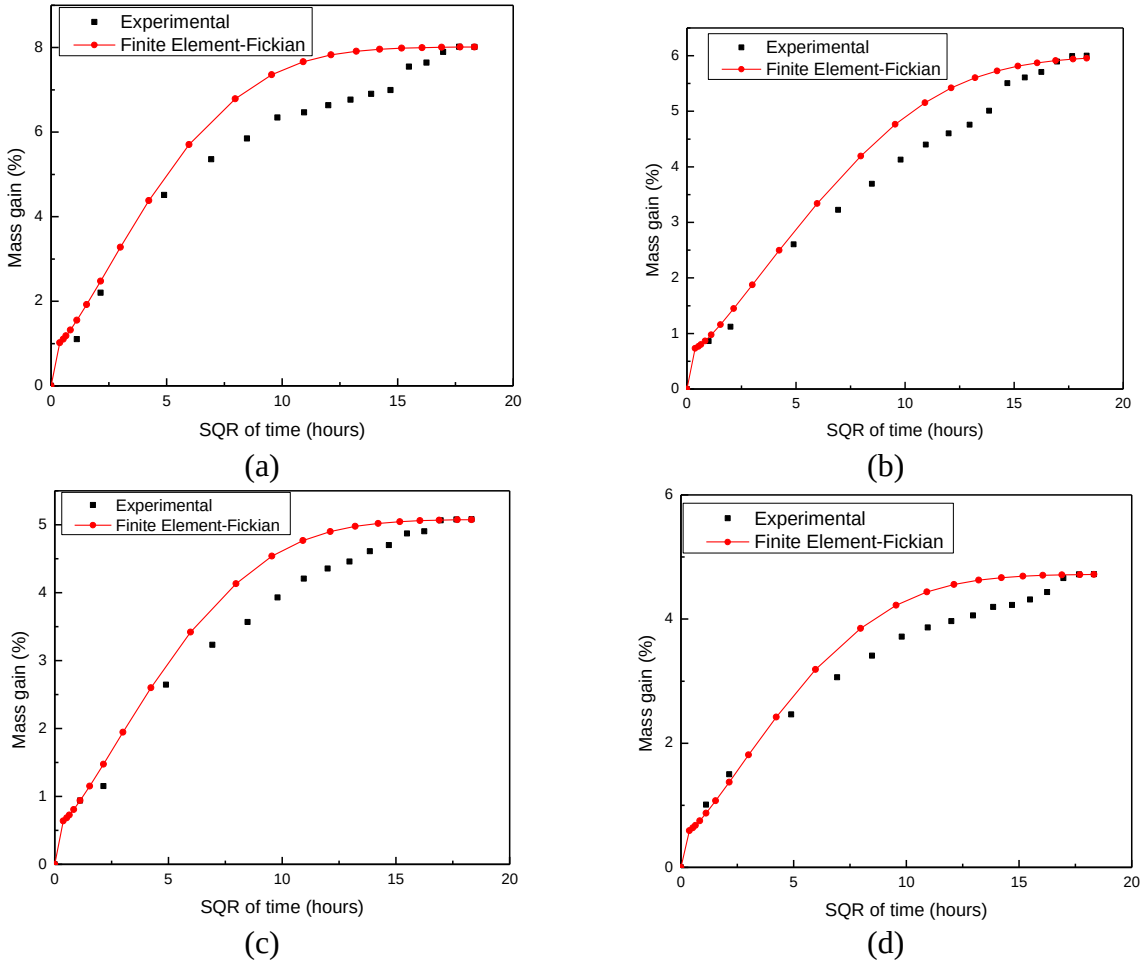


Figure 5.9 Comparative graphs between FEM and experimental work at 75°C (a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

5.3.5 Environment Chamber at 25°C/ 70% R.H

Figure 5.10 shows the comparison between the FEM and the experimental work at 25°C/ 70% R.H in the environment chamber.

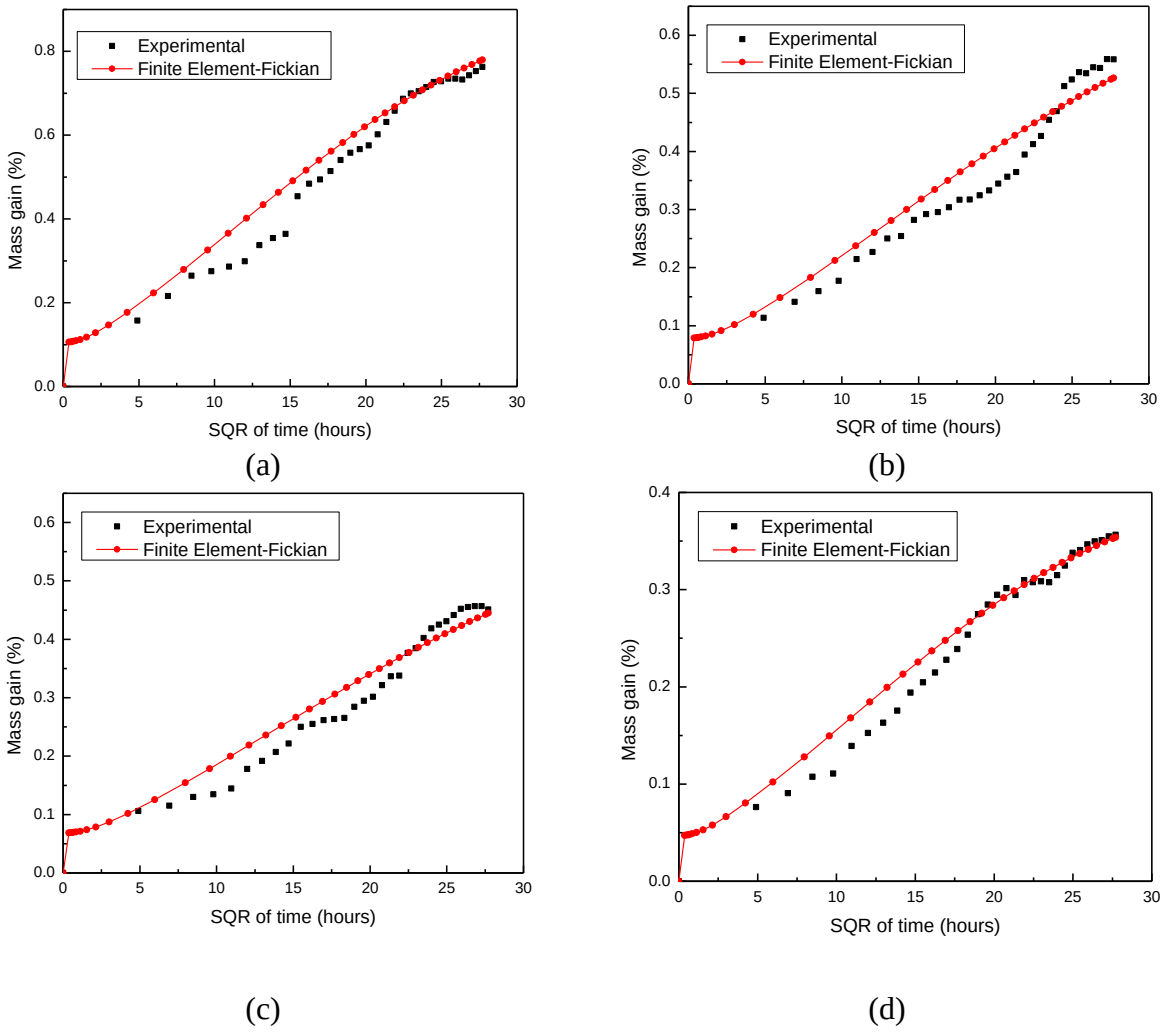


Figure 5.10 Comparative graphs between FEM and experimental work at 25°C/ 70% R.H
(a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

5.3.6 Environment Chamber at 50°C/ 70% R.H

Figure 5.11 shows the comparison between the FEM and the experimental work at 50°C/ 70% R.H in the environment chamber.

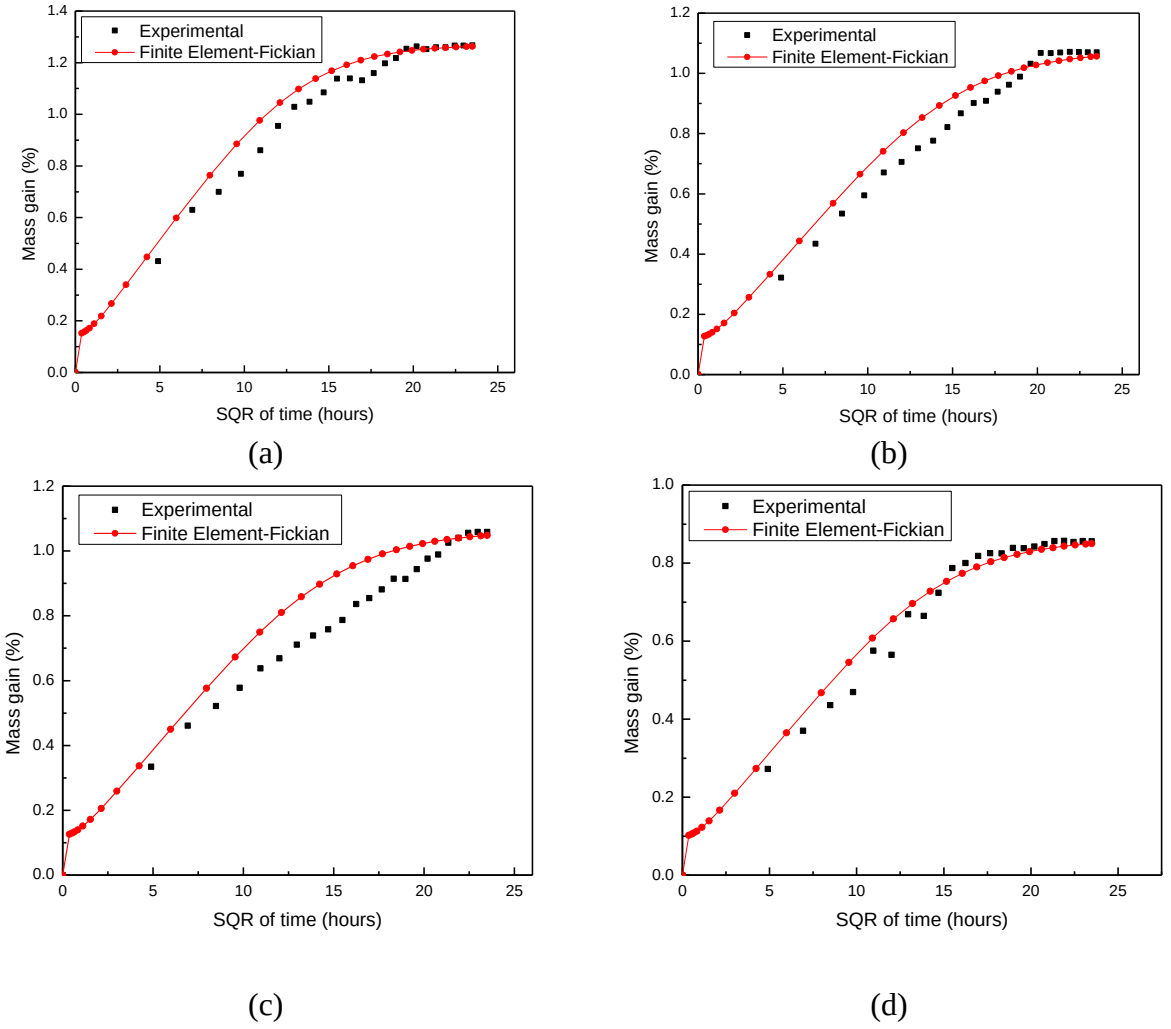


Figure 5.11 Comparative graphs between FEM and experimental work at 50°C/ 70% R.H (a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

5.3.7 Environment Chamber at 75°C/ 70% R.H

Figure 5.12 shows the comparison between the FEM and the experimental work at 75°C/ 70% R.H in the environment chamber.

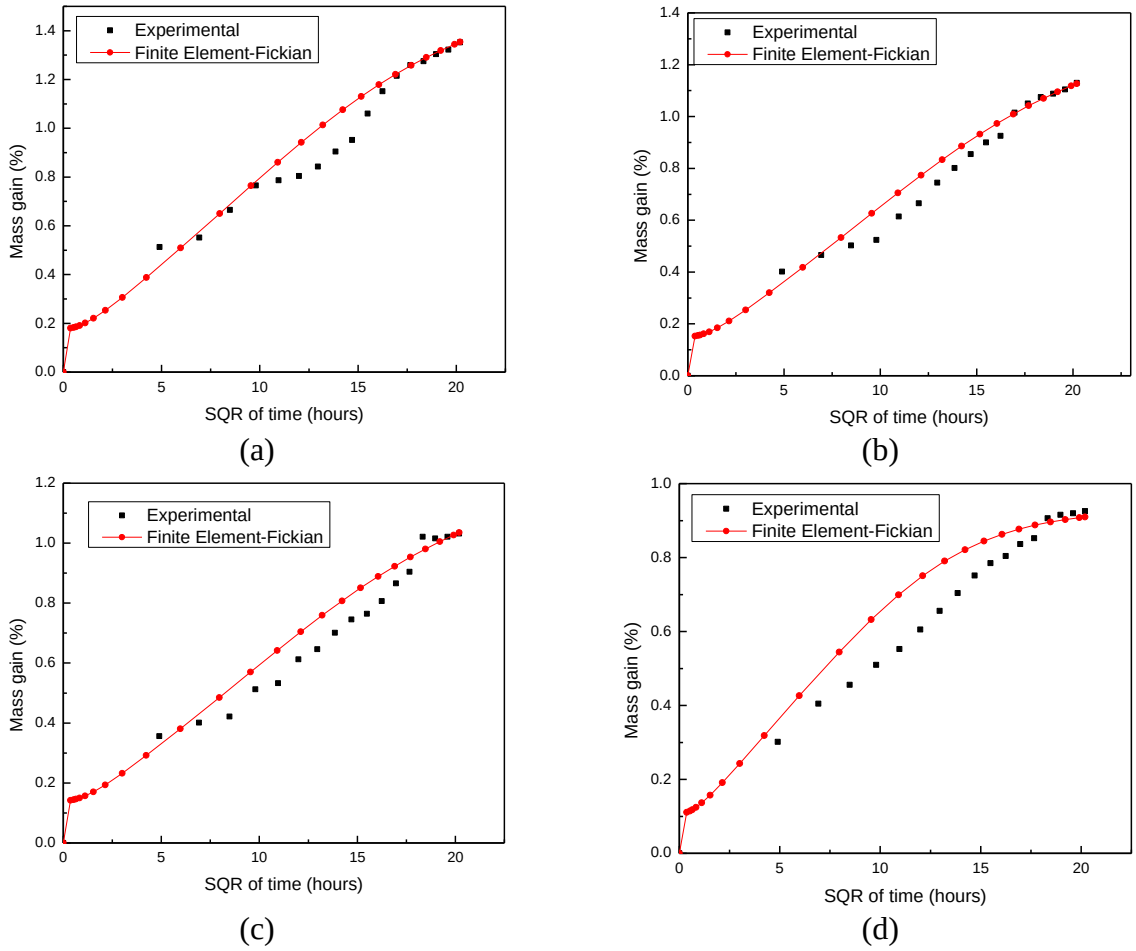


Figure 5.12 Comparative graphs between FEM and experimental work at 75°C/ 70% R.H
(a) Untreated fibre, (b) Water hornified, (c) NaOH treated and (d) Alkali hornified specimen

CHAPTER 6

CONCLUSIONS

6.1 CONCLUSIONS

The objective of this work is to observe the effect of different types of chemical treatments on the environment durability of Agave Americana fibre reinforced polymers.

Three different types of chemical treatments of the Agave fibres were done by alkali hornification, Water hornification and NaOH treatment. The key conclusions of this work are as follows:

1. All the treatment methods lead to improved moisture durability as compared to the untreated specimen.

2. Moisture diffusion is analyzed at 0°C in the cryostat bath, 25°C, 50°C and 75°C in the water bath and 25°C, 50°C & 75°C in the environmental chamber with 70 % R.H. The rate of mass gain increases with the increase in temperature. NaOH and alkali hornification results in a reduced diffusivity when compared to water hornification.

(i) At 0°C (cryostat bath) the specimen took 38 days to achieve saturation, the mass gain in the untreated fibre specimen is 3.613452%, water hornified specimen is 2.567501%, NaOH treated specimen is 2.352021% and alkali hornified specimen is 1.587895%.

(ii) At 25°C (Water bath) the specimen took 32 days to achieve saturation, the mass gain in the untreated fibre specimen is 6.342318%, water hornified specimen is 5.586867%, NaOH treated specimen is 4.382156% and alkali hornified specimen is 1.587895%.

(iii) At 50°C (Water bath) the specimen took 21 days to achieve saturation, the mass gain in the untreated fibre specimen is 7.094787%, water hornified specimen is 5.896867%, NaOH treated specimen is 4.534625% and alkali hornified specimen is 4.205462%.

(iv) At 75°C (Water bath) the specimen took 15 days to achieve saturation, the mass gain in the untreated fibre specimen is 8.015%, water hornified specimen is 6.00067%, NaOH treated specimen is 5.079% and alkali hornified specimen is 4.7215 %.

(v) At 25°C/70% R.H (Environmental chamber) the specimen took 34 days to achieve saturation, the mass gain in the untreated fibre specimen is 0.7625213%, water hornified

specimen is 0.558584%, NaOH treated specimen is 0.470908% and alkali hornified specimen is 0.404645%.

(vi) At 50°C/70% R.H (Environmental chamber) the specimen took 24 days to achieve saturation, the mass gain in the untreated fibre specimen is 1.26717%, water hornified specimen is 1.070029%, NaOH treated specimen is 1.05853% and alkali hornified specimen is 0.865675%.

(vii) At 75°C/70% R.H (Environmental chamber) the specimen took 18 days to achieve saturation, the mass gain in the untreated fibre specimen is 1.352461%, water hornified specimen is 1.300029%, NaOH treated specimen is 1.211555% and alkali hornified specimen is 0.925675%.

3. SEM micro structural investigations reveal that with the NaOH treatment and increase of the cycles in the alkali and water hornification improve the surface texture and porosity of structure.

4. The inverse calculation of the diffusion coefficients and a subsequent analysis of water uptake behaviour using finite element method were compared with experimental results. The analytical results show a good agreement with experiments. Hence, the analytical FEM method can be used effectively to reduce the number of experiments.

6.2 FUTURE SCOPE OF THIS WORK

Although significant efforts have been made in regard to reduce the moisture absorption in the natural fibres but still a lot of advancement can be done. Some suggestions are

- The effect of different concentrations of NaOH on the agave Americana fibre can be seen
- The number of cycles of the alkali hornification and the water hornification can vary and the specimen with different cycles can be prepared to study the moisture diffusivity
- The orientation of the fibre can vary in regard to study the effect on diffusivity
- Analysis on moisture diffusivity by using sealants and by controlling the direction of diffusivity in the specimen can also be studied.
- Preparation of hybrid composites with different fibre concentrations can also be done
- The effect of moisture uptake on the mechanical strength may be studied

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CURRICULUM VITAE

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