AN INVESTIGATION ON MECHANICAL PROPERTIES OF SILK FIBRE REINFORCED POLYPROPYLENE COMPOSITES

A PROJECT REPORT

Submitted by

A. GOWRI	0810203304
B. GOWRI PRIYA	0810203305
S. JANANI	0810203306

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(An Autonomous Institution affiliated to Anna University of Technology, Coimbatore)

BONAFIDE CERTIFICATE

Certified that this project report "AN INVESTIGATION ON MECHANICAL PROPERTIES OF SILK FIBRE REINFORCEED POLYPROPYLENE COMPOSITES" is the bonafide work of **A.Gowri (0810203304)**, **B.Gowri Priya (0810203305)**, **S.Janani (0810203306)** who carried out the project work under my supervision

SIGNATURE

DR.J.SRINIVASAN

HEAD OF THE DEPARTMENT

FASHION TECHNOLOGY
KUMARAGURU COLLEGE
OF TECHNOLOGY
(An Autonomous Institution
Affiliated to Anna University of
Technology)
Saravanampatty,
Coimbatore.

SIGNATURE

Mr.G.RAJKUMAR
Assistant professor

SUPERVISOR

FASHION TECHNOLOGY
KUMARAGURU COLLEGE
OF TECHNOLOGY
(An Autonomous Institution
Affiliated to Anna University of
Technology)
Saravanampatty,
Coimbatore.

Certified that the candidates with the University Register No. 0810203304, 0810203305, 0810203306 were examined by us in the project viva-voce examination held on .....12-04-2012...

12.4.12
(INTERNAL EXAMINER)

(EXTERNAL EXAMINER)

ABSTRACT

Fibre reinforced composites have gained popularity nowadays because of their processing advantages and good technical properties like strength, stiffness, elastic modulus, creep rate, damping. Natural fibre reinforced composites are best alternative one for synthetic composites.

The natural fibres such as hemp, ramie, coir, pineapple leaf fibre etc are used as reinforcement materials in the composites. Only few literatures are available on silk fibre reinforced composite materials. Hence an attempt is made in this project work to study the mechanical properties of silk fibre reinforced polypropylene composites.

The silk hard waste is collected from shuttle less loom silk fabric weaving mills. The silk threads are cleaned from non silk materials like cotton leno threads. The pure silk threads are converted into fibrous form using an opening machine.

The silk fibres are blended with polypropylene in blend ratio of 50:50 silk / polypropylene. The blended fibres are carded three times to get well oriented web. A composite is made in the compression moulding machine using six layers of super imposed webs. The process parameters like temperature, time and pressure in the compression moulding machine are optimized using box Benken design using Design Expert software.

The mechanical properties of the composites like tensile strength, impact strength and flexural strength are determined using ASTM standards. The thermal conductivity and water uptake properties are also determined.

From this work it is found that the optimised process parameters for the silk reinforced polypropylene composites are 177°C temp, 7 mins and 35 Bar pressure. Composite boards are prepared in the optimised process conditions. The actual values of tensile, flexural and impact strength are compared with the predicted values of the design expert software. The result shows that there is a very good correlation between actual and predicted values.

TABLE OF CONTENTS

Chapter I : Introduction

Chapter II : Literature Review

Chapter III : Materials And Methods

Chapter IV : Results And Discussions

LIST OF TABLES

LIST OF FIGURES

CHAPTER I

INTRODUCTION

Natural fiber reinforced polymer composites have raised great attention and interest among materials scientists and engineers in recent years due to the considerations of developing an environmental friendly material and partly replacing currently used glass or carbon fibers in fiber reinforced composites. The use of synthetic polymers, toxin, substances and non biodegradable products, because of their hazards after effects on environment and human health, helps to re-establish the needs of natural fiber.

Polymer composites have been subjected to increasing interest, study, and utilization for some decades. However, the increase in environmental concern has pointed out how it is also necessary to reduce and rationalize the use of polymeric materials, not only due to their non-biodegradability, but also because their production requires large amount of oil as raw material which is notoriously not renewable. All these issues have induced mankind to look for alternatives (La Mantia & Morreale, 2006).

Natural fiber reinforced polymer composites materials are almost replacing materials such as ceramics, metals, glasses, etc. Moreover, in the last two decades, organic natural fillers have gained importance over the inorganic fillers due to their low densities, low cost, non abrasiveness, easy recyclability, bio-degradability and renewable nature

In India, the non-woven and composite industry is in its initial stage. As the price of petro-based materials like polypropylene and polyester continues to be high, and eco-concern is increasing among the consumers, new technologies are emerging; natural fibers are being used, creating non-woven composites with attributes never seen before.

The silk hard waste from shuttle less weaving mills are collected and separated from non silk materials and cotton leno threads. Then the silk hard waste is converted into fibrous form. The opened silk fibres are blended with polypropylene. They used for web formation. The web is formed in a carding machine and bonding will be done at optimized process parameters in a compression moulding machine.

1.1 BACKGROUND:

A composite is a material made by combining two or more dissimilar materials in such a way that the resultant material is endowed with properties superior to any of its parental ones. Fibre-reinforced composites, owing to their superior properties, are usually applied in different fields like defence, aerospace, engineering applications, sports goods, etc. Nowadays, natural fibre composites have gained increasing interest due to their eco-friendly properties. A lot of work has been done by researchers based on these natural fibres.

Natural fibres such as jute, sisal, silk and coir are inexpensive, abundant and renewable, lightweight, with low density, high toughness, and biodegradable. Natural fibres such as silk have the potential to be used as a replacement for traditional reinforcement materials in composites for applications which requires high strength to weight ratio and further weight reduction. Silk has been an important fabric in the textiles industry due to its lustre and superb mechanical properties. Silk yarn are easily available as the waste product of textile industry, so the composite is cost effective and perfect utilization of waste product.

1.2 OBJECTIVES OF THE PROJECT:

The main objective of the project work is utilization of natural silk fibre waste for wealth creation. A number of synthetic fibres are being used as composite materials. These synthetic fibres are hazardous to human skin and also they are not eco friendly and biodegradable. The main objectives of the project are:

- To utilize spun silk selvedge waste generated during shuttle less weaving and to produce silk reinforced polypropylene composites
- To optimize the process parameters like Temperature, Time and Pressure in compression molding machine
- To study the mechanical properties like Tensile strength, Shear strength and Flexural strength of the silk reinforced polypropylene composites.
- To study the thermal and water uptake properties of silk reinforced polypropylene composites.
- To study the acoustical properties of silk reinforced polypropylene composites and to compare with commercially available products.

CHAPTER II

REVIEW OF LITERATURE

2.1 SILK FIBER

2.1.1 INTRODUCTION:

Silk is a natural protein fiber, some forms of which can be woven into textiles. The best-known type of silk is obtained from the cocoons of the larvae of the mulberry silkworm Bombyx mori reared in captivity (sericulture). The shimmering appearance of silk is due to the triangular prism-like structure of the silk fiber, which allows silk cloth to refract incoming light at different angles, thus producing different colors.

Silks are produced by several other insects, but only the silk of moth caterpillars has been used for textile manufacturing. There has been some research into other silks, which differ at the molecular level. Silks are mainly produced by the larvae of insects undergoing complete metamorphosis, but also by some adult insects such as web spinners. Silk production is especially common in the Hymenoptera (bees, wasps, and ants), and is sometimes used in nest construction.

2.1.2 STRUCTURE OF SILK

The secondary structure of silk is an example of the beta pleated sheet. In this structure, individual protein chains are aligned side-by-side with every other protein chain aligned in an opposite direction. The protein chains are held together by intermolecular hydrogen bonding, that is hydrogen bonding between amide groups of two separate chains. This intermolecular hydrogen bonding in the beta-pleated sheet is in contrast to the intra molecular hydrogen bonding in the alpha-helix.

The hydrogen on the amide of one protein chain is hydrogen bonded to the amide oxygen of the neighbouring protein chain. The pleated sheet effect arises from the fact that the amide structure is planar while the "bends" occur at the carbon containing the side chain.

Fortunately, the "side" chain R groups in silk are not very bulky. The basic primary structure of silk consists of a six amino acid unit that repeats itself. The sequence where every other unit is glycine in silk is: -gly-ala-gly-ala-gly-ala-. Although glycine and alanine make up 75-80% of the amino acids in silk, another 10-15% is serine and the final 10 % contain bulky side chains such as in tyr, arg, val, asp, and glu.

These amino acids with bulky side chains disrupt the regular patterns set by the gly-ala-ser. Different species of silkworms produce different portions of ordered and disordered regions. The disordered regions provide a small amount of elasticity since the ordered beta-pleated sheet is already fully extended and cannot stretch further without breaking.

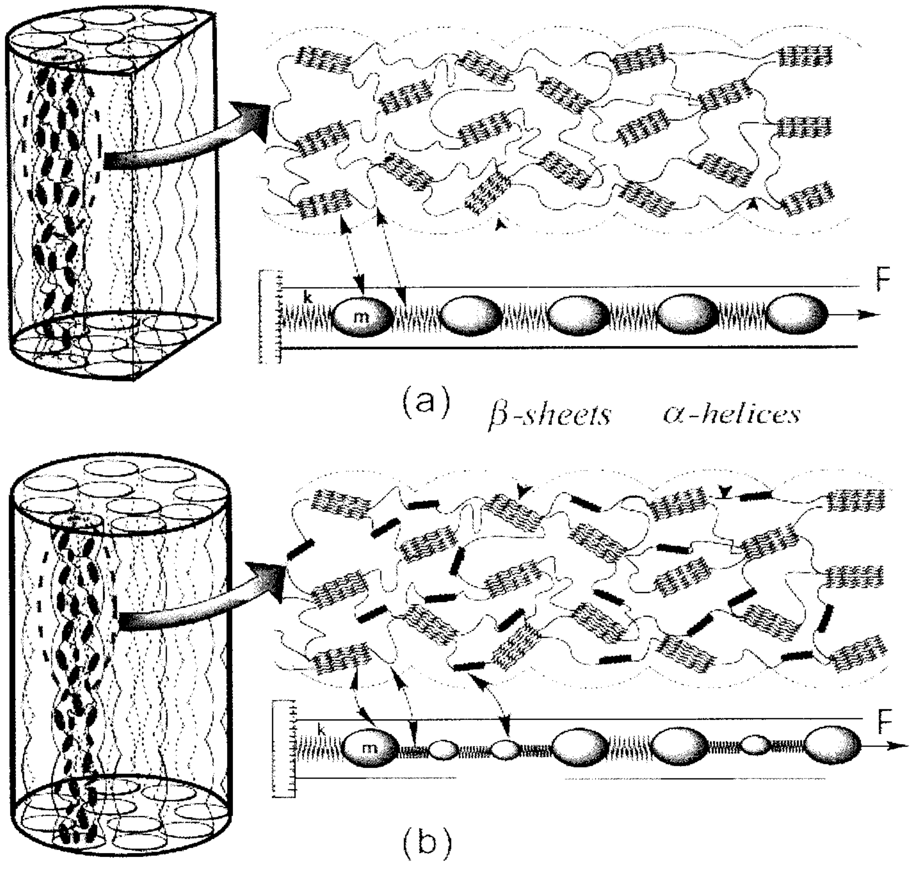
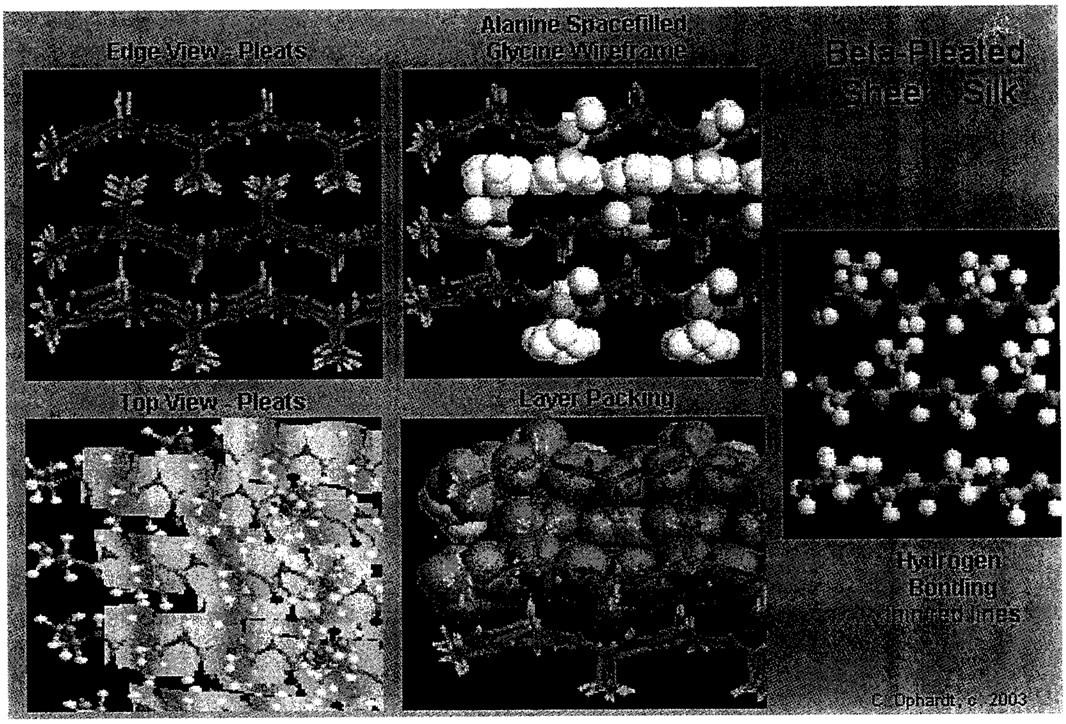


Fig:2.1 Shows structure of silk

The beta pleated sheet motif is found in many proteins along with the alpha helix structure. The chains may run parallel (all N terminals on one end) or anti-parallel (N terminal and C terminal ends alternate).

C. Ophardt, c. 2003

Fig:2.2 Shows both Helices and Sheets in same protein



C. Ophardt, c. 2003

Fig:2.3 Shows beta-pleated sheet of silk

2.1.3 PROPERTIES OF SILK

2.1.3.1 Physical properties

Density	1.34 g/cm^3
Fiber diameter	11.5 µm 3.57µm(spider) 12.9 µm(bombyx mori)
Linear density	0.014 tex (spider) 0.117 tex(bombyx mori)

Table: 2.1 Physical Properties of Silk

2.1.3.2 Chemical Properties

Silk is hygroscopic in nature. Under standard environmental condition (20° C (68°F), 65% R.H) silk absorbs about 10 W% of water. Water absorption causes fibre swelling, which has been reported to about 1.6% in the longitudinal direction and 18.7% crosswise. In the isoionic range (PH 4-5), swelling and reactivity are at their lowest.

As a protein fibre, silk is amphoteric because it possesses cationic and anionic groups on the side chain of various amino acids residues whose dissociation state depends on the surrounding PH conditions, this characteristics makes silk able to absorb and bind strong alkalis and mineral acids with the formation of salts moderately strong and weak acids undergo binding to silk via hydrogen bonds. Compared to wool, silk is more sensitive to acid and more resistant to alkali, due to the absence of acids-stable and alkali-unstable cystine bridges. This allows processing of silk under alkaline condition while dying should be better carried out a weakly acidic or neutral PH.

Silk is able to absorb considerable amount of salts. This property has been exploited in the traditional mineral waiting process with tin-phosphate-silicate. A method is used for decades to increase silk weight in order to compensate for the last resulting from degumming.

Silk is also characterized by good chemical reactivity. Side chain groups of various amino acids residues can be exploited as reactive sites to bind selected chemical agents in order to obtain a effective and specific modification of the fibrous substrate.

2.1.3.3 Mechanical Properties

Primary and higher order structures of silk fibroin result in a fibre that exhibits outstanding mechanical properties. The mechanical properties of silk are a direct outcome of the chemical structure, the size and orientation of the crystalline domains, and the connectivity of these domains to the less crystalline and amorphous regions. Silk is unique in terms of strength and toughness among all the natural and synthetic fibres. Typical values of mechanical parameter are as follows: stress 710+_130 MPa, strain 26+_5% modulus 9.5+_1.6 GPa .interestingly, an increase in the rate of loading causes increased elongation and thus greater resistance to rupturing. This is a typical feature of all natural silk, including silk worm silk, which represent an exception to most fibrous material. In general, silk has an excellent ability to absorb energy at high rates of loading.

Tenacity	4.25 gram-force per denier
Tensile strength	260 MPa 180 MPa(spider)
Young's modulus, longitudinal	12.7 Gpa(N clavipes spider) 9.9 Gpa(bombyx mori) 34 Gpa(A aurantia spider)
Young's modulus. Transverse	0.579 GPa

Table: 2.2 Mechanical Properties of Silk

2.1.3.4 Thermal Properties:

Silk is virtually unaffected by temperature up to 140° C (284°F). Based on differential scanning calorimeter, thermo mechanical, and thermo gravimetric analyses, with increasing temperature up to about 200°C (392°F) silk display an exceptional thermal stability. At higher temperature, small molecular weight gas molecules i.e. CO, CO_2, NH_3, etc... Start evolving due to thermal degradation of side chain groups of amino acid residues. If the temperature is increased further, degradation proceeds faster, leading to main chain fission and, at above 300° C (572°F), to complete burning. The glass transition temperature is located at C. 175° C (347°F). Along with other protein fibers, such as wool, silk is considered to have low flammability.

2.2 POLYPROPELENE

2.2.1 INTRODUCTION

The most important property of polypropylene is its versatility. It can be tailored to many fabrication methods and applications. Excellent chemical resistance, the lowest density, highest melting point (in the family of olefin fibers), and moderate cost makes it an important fibre in industrial applications. However, the poor dyeability and texturizability have limited polypropylene's applications in conventional textile industry. With the introduction of fiber denier textured yarn its use in apparel sector is becoming inevitable along with its main outlet in the industrial sector.

When polypropylene was first introduced into the market in 1950s, the product is non-crystallisable, which result in low melting point. With the introduction of isotactic commercial polypropylene a considerable increase in the crystallinity and melting point was achieved. Polypropylene has registered continued worldwide market share growth in recent years and it is predicated that consumption will still grow as polypropylene increasingly are used as substitution for other materials such as glass, metal, and some engineering plastics.

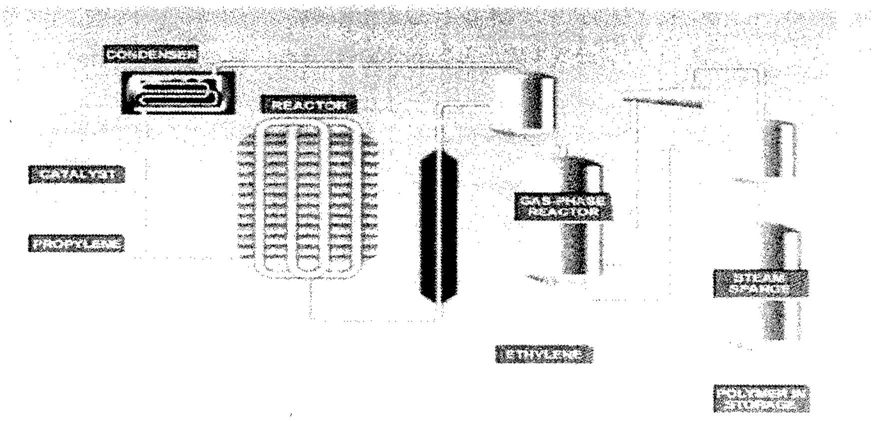
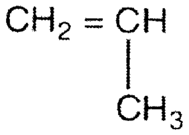


Fig: 2.4 Process of Polypropylene

2.2.2 STRUCTURE OF PP FIBRE

Polypropylene (PP) is a thermoplastic matrix material that received attention for the production of polymer composites (Fung *et al.*, 2003). PP was first product produced by G. Natta, *following the work of K. Ziegler, by the* polymerization of propylene monomer *in 1954. The macro molecule of PP* contains 10,000 to 20,000 monomer *units. The steric arrangement of the methyl* groups attached to every second *carbon atom in the chain may vary (Figure* 2.8). if all the methyl groups are *on the same side of the winding spiral chain* molecule, the product is referred *to as isotactic PP. A PP structure where* pendant methylene groups are attached *to the polymer backbone chain in an* alternating manner is known as syndiotactic *PP. The structure where pendant* groups are located in a random manner *on the polymer backbone is atactic* form (Billmeyer, 1971).



$$CH_2=CH-CH_3$$

Figure: 2.5 Polymer Molecule in Isotactic, Syndiotactic and Atactic

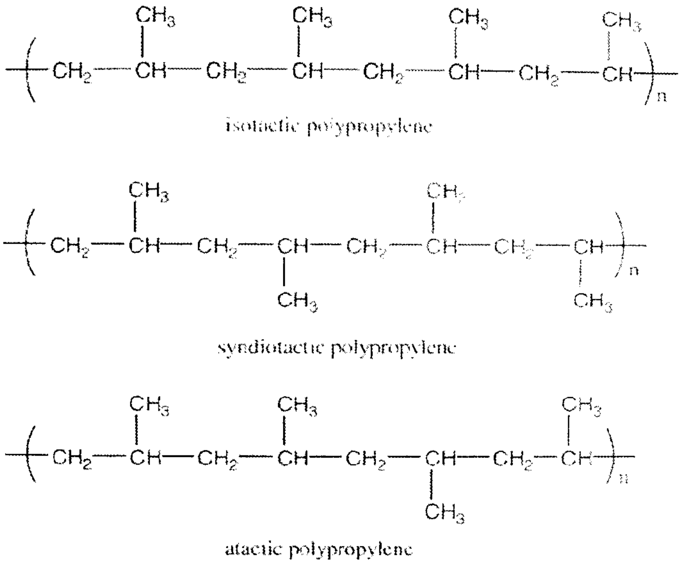


Figure: 2.6 Pr*opylene Monomer (Billmeyer, 1971)*

2.2.3 PROPERTIES OF PP FIBRE

- Fairly low physical properties
- Fairly low heat resistance
- Excellent chemical resistance, i.e. resistant to deterioration from chemicals, mildew,insects,perspiration, rot and weather
- Translucent to opaque
- "Living hinge" capability
- Low price
- Easy to process
- Good bulk and cover,
- Very lightweight (olefin fibres have the lowest specific gravity of all fibres)
- High strength(wet or dry)
- Abrasion resistant
- Low moisture absorption
- Stain and soil resistant
- Good washability, quick drying, unique wicking
- Resilient, mouldable, very comfortable
- Thermally bondable

The main drawbacks of PP fibres are listed below:

- Low melting temperature prevents it from being ironed like cotton, wool, nylon etc.
- Hard to be dyed after manufacturing, except after substantial treatment and modification.
- High crystallinity and poor thermal conductivity leads to limited texturizability. [Drawn polypropylene requires a contact time of 2 seconds in the heater compared to PET (POY)which requires only 0.4 seconds]
- Poor UV and thermal stability which requires addition of expensive UV stabilizers and antioxidants to overcome this problem
- Poor adhesion to glues and latex
- Flammable which melts and burns like wax.

Heat setting removes the residual strains and produces a defect-free and stable crystalline structure to make fibre/fabrics dimensionally stable. It also improves the percentage of overall crystallinity. The smectic structure changes to more perfect monoclinic structure during the process of heat setting if the temperature is above 70°C. At 145°C the conversion is almost complete. In comparison to predominantly smectic form, the monoclinic form does not experience any major change in crystalline structure during the course of drawing and heat setting.

2.2.3.1 Physical Properties

The general physical properties of PP fibres are shown in Table 1. Polypropylene fibres are produced in a variety of types with different tenacities designed to suit varying market requirement. Fibres for general textile uses have tenacities in the range of 4.5-6.0 g/den. High tenacity yarns up to 9.0 g/den are produced for the use in ropes, nets and other similar applications. High performance PP fibres have been made with high strength and high modulus. The techniques include ultra-drawing, solid state extrusion and crystal surface growth. The filaments with tenacities over 13.0 g/den can be made.

The degree of orientation achieved by drawing influences the mechanical properties of PP filaments. The greater the degree of stretch, the higher the tensile strength and the lower the elongation. Commercial PP monofilaments have an elongation at break in the region of 12-25%. Multifilament sand staple fibres are in the range of 20-30% and 20-35%, respectively.

Moisture regain	<0.1%
Refractive indexNnD^{23}	1.49
Thermal conductivity	0.95 Btu-in/ft^2.hr.°F
Coefficient of linear thermal Expansion	4.0 x 10^{-5}/°F
Heat of fusion	21 cal/g
Specific heat	0.46 cal/g.C
Density of melt at 180Oc	0.769 g/cc
Heat of combustion	19,400 Btu/lb
Oxygen index	17.4

Decomposition temperature range	328-410^{0}C
Dielectric constant (0.1 M Hz)	2.25
Dissipation factor (0.1 M Hz)	<0.0002
Specific volume resistivity	>1016.cm

Resistance at 20^{0}C	Concentration %	Residual Tenacity % (after 4 days)
Hydrochloric Acid	34	100
Nitric Acid	65	90
Sulphuric Acid	95	100
Formic Acid	75	100
Glacial Acetic Acid	-	100
Potassium Hydrate	40	95
Sodium Hydrate	40	100
Trichloroethylene		80
Perchloroethylene		80
Toluene		98
Benzene		80
Sodium Hypochlorite (5% active chlorine)		85
Hydrogen Peroxide (12 Parts)		90
Sulphuric Acid	94	100
Glacial Acetic Acid		100
Sodium Hydrate	40	100

Table: 2.3 Physical Properties of Polypropylene

2.2.3.2 Chemical Properties

Acids –concentrated	Good-Fair
Acids-dilute	Good-fair
Alcohols	Good
Alkalis	Good-Greases
Aromatic hydrocarbons	Fair
Greases and Oils	Good-Fair
Halogenated hydrocarbons	Good-poor
Halogens	Poor
Ketones	Good

Table: 2.4 Chemical Properties of Polypropylene

2.2.3.3 Mechanical Properties

Abrasive resisitance –ASTM D1044	13-16mg/1000cycles
Coefficient of friction	0.1- 0.3
Hardness –rockwell	R80-100
Elongation at break (%)	150-300, for biax film>50
Izod impact strength (J m^{-1})	20-100
Tensile modulus (GPa)	0.9-1.5, for biax film 2.2-4.2
Tensile strength (MPa)	25-40, for biax film 130-300

Table: 2.5 Mechanical Properties of Polypropylene

2.2.3.4 Electrical Properties

Dielectric constant@1MHz	2.2-2.6
Dielectric strength (k V mm^{-1})	30-40
Dissipation factor @ 1MHz	0.0003 – 0.0005
Surface resistivity (Ohm/sq)	10^{13}
Volume resistivity (Ohm cm)	10^{1}

Table: 2.6 Electrical properties of polypropylene

2.2.3.5 Thermal Properties

Polypropylene fibres have a softening point in the region of 150oC and a melting point at 160-170oC.At low temperatures of -70oC or lower, PP fibres remain their excellent flexibility. At high temperature (below 120oC) PP fibres nearly remain their normal mechanical properties. PP fibres have the lowest thermal conductivity of all commercial fibres. In this respect, it is the warmest fibre of all. The thermal conductivity of common textile fibres is shown in the table below:

Material	Thermal conductivity relative to air
Air	1.0
PP	6.0
Wool	6.4
Acetate	8.6
Viscose	11
Cotton	17.0

Table: 2.7 Thermal Conductivity of Textile Fibres

2.2.3.6 Other Properties

In general, PP fibre has excellent chemical resistance to acids and alkalis, high abrasion resistance and resistance to insects and pests. PP fibre is also easy to process and inexpensive compared to other synthetic fibres. Its low moisture absorption helps quick transporting of moisture.

The main drawbacks of PP fibres are:

1. Low melting temperature which prevents it from being ironed like cotton, wool, nylon etc
2. Hard to be dyed after manufacturing except being modified
3. High crystallinity and poor thermal conductivity leads to limited texturizability. [Drawn polypropylene requires a contact time of 2 seconds in the heater compared to PET which requires only 0.4 seconds]
4. Poor UV and thermal stability which requires addition of expensive UV stabilizers and antioxidants to overcome this problem,
5. Poor resilience compared to PET and Nylon
6. Creeping due to its low Tg (-15 to -20^{o}C)

7. Poor adhesion to glues and latex
8. Flammable which melts and burns like wax.

2.2.4 PROCESSABILITY

Processability of a polymer is highly dependent on its rheological properties, which have close relationship with its molecular weight, molecular weight distribution, temperature and shear rate. PP resins are generally categorized according to their melt flow rates (MFR), which is the amount of material that comes through a standard die hole for ten minutes. Polymers with higher molecular weight have lower MFR and higher viscosity (under a given temperature). Commercial polypropylene has a wide range of MFR from 0.25 to 800. MFR is a very important parameter for both melt-blown and Spun bond processing. PP melts exhibit non-Newtonian viscosity, normal stress in shear flow, excessive entrance and exit pressure drop, die swell, melt fracture and draw resonance. PP melts are more visco elastic than PET and nylon melts. The flow pattern and stability of PP melts are

Properties Of PPSF

PPSF has following unique properties:

1. Lowest specific gravity of 0.91 (provides excellent covering).
2. Negligible Moisture absorption (0.05%).
3. Provides very warm feel (warmer than wool and acrylic).
4. Fabric containing PPSF wicks away moisture from skin to atmosphere due to the capillary action.
5. PPSF is resistant to many acids, alkalies and solvents.
6. PPSF is resistant to attack of mildew/moths/insects and a wide range of bacteria.
7. Not prone to static charge generation.
8. PPSF is dope- dyed in various shades/colours and there will not be any variation between fibre lots of the same shade.
9. Not subject to light degradation, if UV Stabilized.
10. Highly resilient fibre.

11. PPSF is available in round cross-section and also in Trilobal shape (for superior sparkle/lustre.)

Sl.No.	Property	2.5 Denier	15 Denier
1.	Fibre Denier	2.5 ± 0.1	15-16
2.	Fibre Tenacity (gm/denier)	5.5-6	3.3-3.8
3.	% Oil Pick-up	0.4-0.5	0.5-0.6
4.	Crimps per cm	5.4-5.8	3.6-4.0

Table: 2.8 Typical Fibre Properties

2.2.5 FIBRE APPLICATIONS

Finer Denier PPSF Denier range of 2.5 to 3 in Natural white is used for spinning yarns which go into Filter Fabrics. Also, finer denier PPSF is used in non-woven Geo-Textile and in Hygienic Products.

Other major applications of finer denier PPSF are in thermal bonded fabrics, thermal insulations, Dref-2 yarns for Filter Cartridges, Blankets, carpet backing yarn etc.Coarser Denier PPSF of 10 to 15 Denier is used in floor non-woven carpets, automobile floor mats etc.

PPSF cut into smaller cut lengths like 6mm, 10mm, and 20mm, are used in the building construction industry and also in asbestos sheet/pipes.

2.3 COMPOSITES

Composite materials were divided into natural occurring composite and synthetic composites. Many natural occurring materials were classified as composite materials. Examples of natural composites were wood which was made up of cellulose molecules in a lignin matrix, teeth and bone which were composed of hydroxyapetite in a matrix of collagen, insect exoskeleton and to name a few. Synthetic composites were concrete which a combination of stone and cement, asphalt and also fibreglass which was a glass fibre reinforced unsaturated polypropylene matrix.

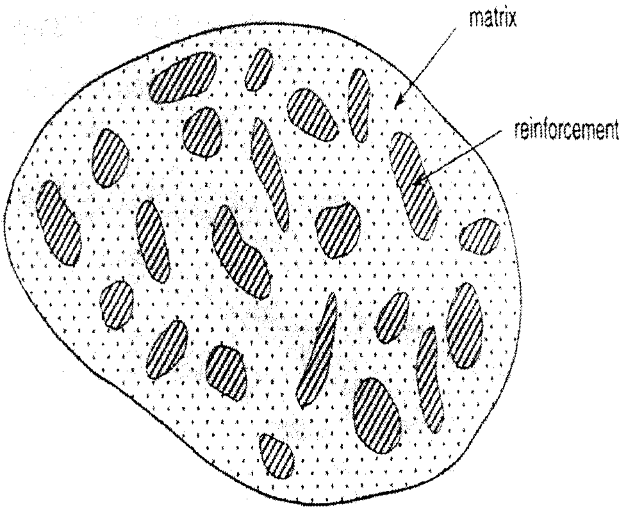


Figure: 2.7 Composite materials (Berthelot, 1999)

2.3.1 Definition of Composite

A typical composite material is a system of materials composing of two or more materials (mixed and bonded) on a macroscopic scale. Generally, a composite material is composed of reinforcement (fibres, particles, flakes, and / or fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed properly, the new combined material exhibits better strength than would each individual material. As define by Javitz, "Composites are multifunctional material systems that provide characteristic not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form. Kelly [2] very clearly stresses that the composition should not be regarded simple as a combination of two materials. In the broader significance; the combination has own distinctive properties. In terms of strength or resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them. the composites are compound materials which different from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their shortcomings', in order to obtain an improved material. Van suchetclan [4] explains composite materials as heterogeneous materials consisting of two or more solid phases which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property composite.

Nowadays composite materials have recently gained much attention due to their superior properties and wide variety of applications. Composites also one of the fastest growing industries and continue to demonstrate a marked impact to the materials world. Composite can have many meanings and arbitrary classifications (Askeland, 1988; Schwartz, 1992). There appeared to be no completely acceptable universal definition for a composite material. The Oxford dictionary for example, defined a composite as something made up of distinct parts (or constituents). In the broadest definition, any product composed of two or more materials whether metallic, organic, or inorganic and having recognizable interface between them was a composite (Schwartz, 1992; Reinhart & Clement, 1987). According to Jordan (2002), the noun composite was derived from the Latin verb componere which mean to put together. Therefore composite materials were considered as a material which was formed when two or more chemically distinct constituents are combined together on a macro scale (Schwartz, 1992).

Schwartz (1992) further stressed that the constituents in a composite material were physically identified and exhibit an interface between one another. A more detail description of a composite material was given by Agarwal & Broutman (1990) who defined a composite as a material that consisted of one or more discontinuous phases which were usually hard and strong embedded in a continuous phase. The continuous phase was called the matrix while the discontinuous phase was termed the reinforcement material. According to Hepler (1963), composite material consisted of three basic elements: fibre, matrix and the fibre-matrix interface. Each of these elements must have appropriate characteristics and function both individually and collectively in order for the composite to attain the desired superior properties. Meanwhile, Milewski & Katz (1987) described composite as materials that contain a strong, stiff fibre reinforcing material supported by a more flexible binder or matrix material.

2.3.2 Classifications of Composite Materials

Composite materials can be classified in different ways. Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high temperature performance of the composite. A typical classification is given in As per the classifications the composites are basically divided in to two broad classes. They are:

i. Particle-reinforced Composites

ii. Fibre- reinforced Composites

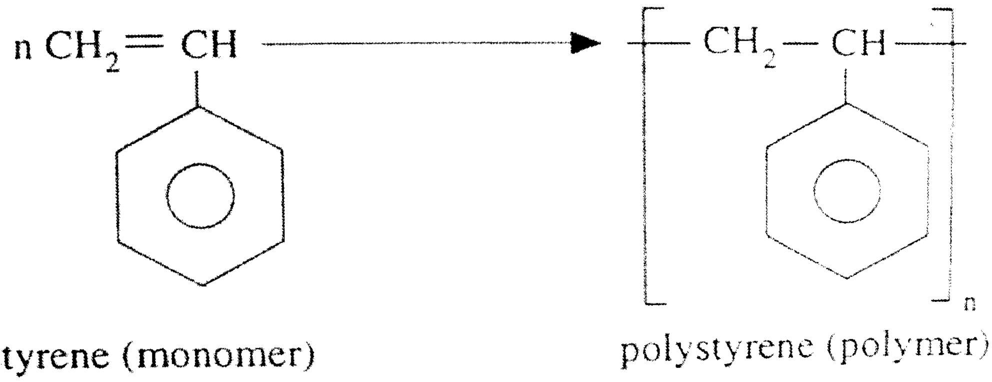


Figure: 2.8 Formation of the polymer polystyrene (Ebewele, 2000)

Modification of organic polymer through the incorporation of additive yields, with few exceptions, multiphase system containing the additive embedded in the

- Improving and controlling of processing characteristics
- Overall cost reduction

Important types of modified polymer systems include polymer composite, polymer-polymer blends and polymeric foam (Xanthos, 2005).In general, parameters affecting the properties of polymer composites include (Xanthos, 2005):

- The properties of the additives (inherent properties, size, shape)
- Composition
- The interaction of components at the phase boundaries, which is also associated with the existence of a thick interface, known also as the interphase, this is often considered as a separate phase, controlling adhesion between the components
- The method of fabrication.

Polymeric composites are increasingly gaining importance as substitute materials for metals in applications within the aerospace, automotive, marine, sporting goods and electronic industries. Their light weight and superior mechanical properties make them especially suited for transportation applications (Seavey, 1999).

2.3.3 NATURAL FIBER COMPOSITES

Natural fiber has attracted worldwide attention as potential reinforcement for composites because of their easy availability, easy processability, Low density, Light weight, non abrasivity, and Lower cost and above all eco-friendly characteristics **Silk Fiber Composite** Composition, structure and material properties of silk fibre produced by **spiders, silkworms, scorpions, mi**tes and flies may differ widely depending on **the specific source and the uncontrollable reel**ing conditions of those insects.

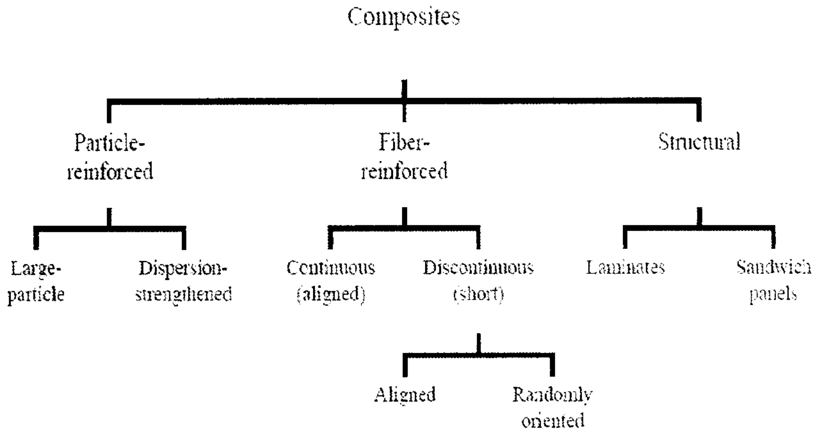


Figure: 2.9 Type of reinforced composites

Spinning under controlled conditions will have more uniform cross-sectional area of silk fibre, reproducible molecular alignment and fewer micro-structural flaws. The size and weight of cocoons decrease with an increase in temperature and cocoons can bear efficiently both external static forces and dynamic impact loadings.

Normal compact cocoon exhibits a high ability of elastic deformation with an elastic strain limit higher than 20 % in both longitudinal and transverse directions. Anisotropic properties mainly due to the non-uniform distribution and orientations of silk segments and the inner layer of cocoon has low porosity (higher silk density) and smaller average diameter of silk, therefore, there is an increase in elastic modulus and strength from outside to inside layers. That is, the thinner the silk, the higher the elastic modulus and tensile strength and the maximum values at the innermost layer.

On the other hand, temperature above the glass transition temperature, the cocoon and its layers become softer and softer and behave similar to a rubber-like material. Silk fibre have higher tensile strength than glass fibre or synthetic organic fibre, good elasticity, and excellent resilience. They resist failure in compression, stable at physiological temperatures and sericin coating is water-soluble proteinaceous glue. silks are insoluble in most solvents, including water, dilute acid and alkali.

Reactivity of silk fibre with chemical agents is positively correlated to the largeness of internal and external surface areas . When fabricating silk-based composites, the amount of resin gained by fibre is strongly related to the degree of swelling of the non-crystalline regions, that is, the amorphous regions and the micro-voids inside the fibre. So the composite made from silk fibres are have high elastic modulus ,tensile strength and furthermore it is cost effective as it's a waste material of silk industry.

2.3.4 HAND AND MACHINE LAY-UP

This is the simplest way of manufacturing composites fibre. In this method the fabric layers are placed on a mould until the desired thickness is reached. A gel coat is applied on the mould for better quality surface. Prepegs are very suitable for hand lay-up techniques to avoid any wetting process. Prepegs are yarns or fabrics that are already impregnated or melted with resin. A roller is used to remove the entrapped air, control the thickness and guarantee good wet out and smooth surface. The curing usually takes place at room temperature or under heat to speed up the process. Usually Polyester and Epoxy resin is used for hand lay-up.

Machine lay-up is automated from the hand lay-up. Computer controlled automatic tape lying machines are used to lay down fibre to fabric. This process provides consistency and increased speed.

2.3.5 VARIOUS MOLDING TECHNIQUES

Some of the most common industrial manufacturing processes for polymers include:

- Spray-up molding
- Transfer molding
- Rotational molding
- Injection molding
- Blow molding
- Vaccum forming
- Extrusion
- Pultrusion
- Compression molding

2.3.5.1 Spray-up molding

Chopped fibres and resins are simultaneously deposited on a mold using spraying equipment. Gel coat is applied by spray gun. Curing takes place at room or elevated temperatures. Polyester and epoxy resins are used.

2.3.5.2 Transfer molding

Transfer molding is different to compression molding because the plastic is not fed directly into the die cavity. Instead it is fed into a chamber outside the die. Here it is preheated before a piston forces it through a system of runners and gates that allow it into the die cavity. When the polymer is cured through heat and pressure it is ejected from the mould. The advantage of this process is that the runner system allows the simultaneous production of many small, intricate parts and there is no 'flashing' around the finished article.

2.3.5.3 Rotational molding

Rotational molding is a unique process for manufacturing thermo-softening polymers as it produces hollow items e.g. Lawnmower grass-catchers, balls and some types of children's toys. A split mould is filled with exactly the right quantity of polymer which is then tumbled in the mould once it is bolted together. The die is rotated in a heated chamber until the polymer is completely melted. The die is removed from the chamber and continues to rotate until the polymer is set. The die halves are separated and the article removed.

2.3.5.4 Injection molding

In this process thermo softening polymer powder, or granules, are placed into a feed hopper connected to a heating chamber. When the fluid state is attained, due to the heat, a piston or 'screw type' mechanism forces the plastic through a nozzle and into the die. These are often water-cooled to hasten setting such that when the plastic becomes solid again the die is opened and the component is ejected. Any flashing is then removed. Modern technology and sophisticated metal machining and finishing techniques mean that large objects with fine tolerances are easily produced.

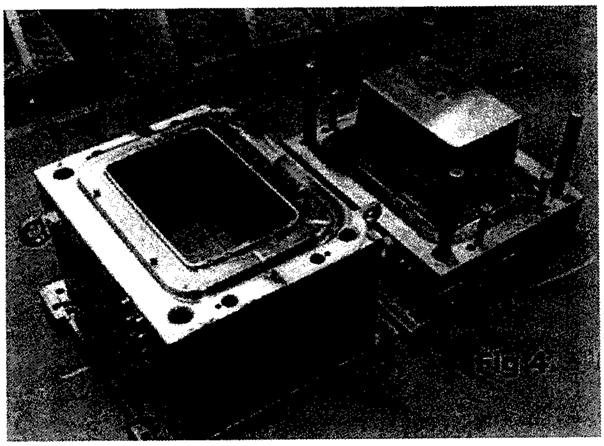
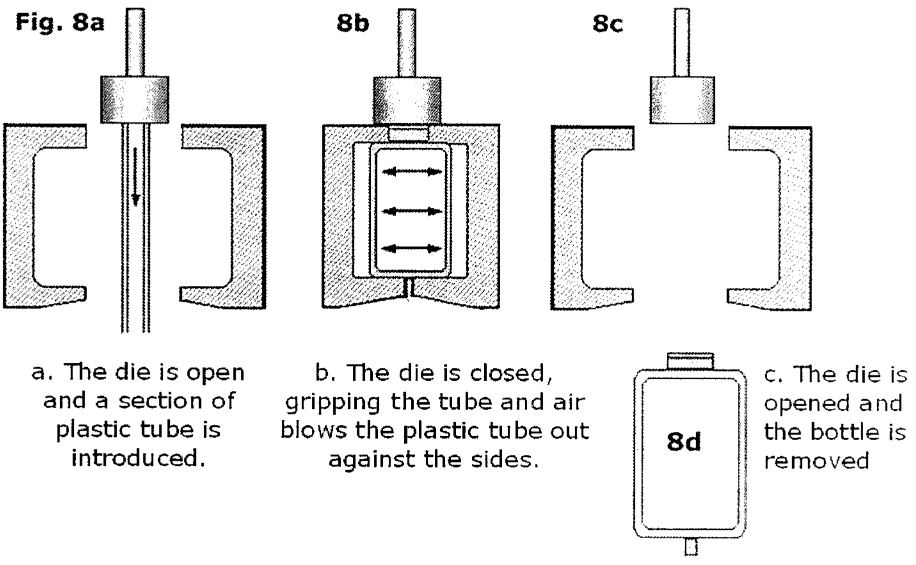


Fig: 2.10 Injection molding

2.3.5.5 Blow molding

Thermoplastic polymer articles, such a soft drink bottles made from PET (polyethyleneteraphthalate), are formed by blow molding. A hot, thin extruded tube (Figure 8a) of the polymer is gripped in a die as an internal blasts of air forces it out against the sides of the mould (Figure 8b). It is held in the die until it cools and is then released (Figure 8c). The polymer assumes the shape of the die (Figure 8d).



a. The die is open and a section of plastic tube is introduced.

b. The die is closed, gripping the tube and air blows the plastic tube out against the sides.

c. The die is opened and the bottle is removed

Fig: 2.11 BLOW MOLDING

With the die closed and the tube pinched, the air pressure, which has blown the bottle to shape, is maintained until the plastic is cooled to room temperature.

2.3.5.6 Vacuum forming

Vacuum forming relies upon air pressure to form a shape. A heated sheet of polymer is clamped above a mould and the air in the mould is evacuated leaving a partial vacuum. The air pressure above the mould forces the plastic sheet down into the mould to form the shape which is removed when cooled. This process is often used for transparent canopies and covers over lit signs such as those seen in service stations or fast food outlets.

2.3.5.7 Extrusion

Extrusion involves the use of powder or granules, mixed with dyes as required, which are placed into a feed hopper connected to a heating chamber. When the fluid state is attained due to the heat, a piston or 'screw type' mechanism forces the plastic through a nozzle and into the mould or die. The dies are often water-cooled to hasten setting such that when the plastic becomes solid again the die is opened and the component is ejected. Any flashing is then removed.

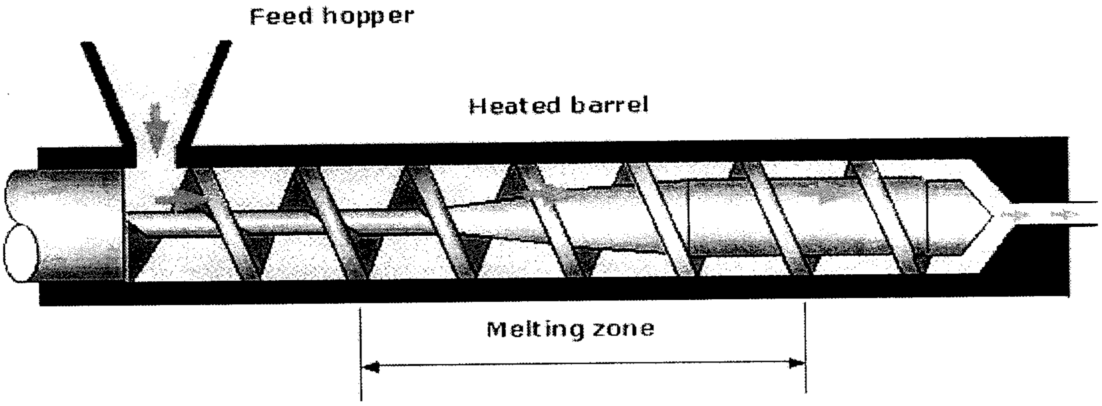


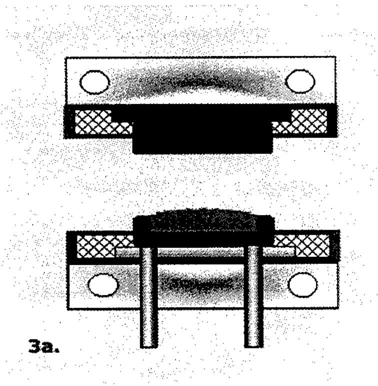
Fig: 2.12 Extrusion

2.3.5.8 Pultrusion

Pultrusion is similar to extrusion except that thermosetting polymers are used and the composition of the composite polymer requires a greater force to move it through the die. For this reason, a pulling force is incorporated into the forming process to overcome resistance during manufacture of the article.

2.4 COMPOSITE MOLDING

According to ASTM International Handbook Committee, 1973, composite material was defined as a macroscopic combination of two or more distinct materials, having a recognized interface between them. However, because composites were usually used for their structural properties, the definition were restricted to include only those materials that contain reinforcement (such as fibers or particles) supported by a binder (matrix) material. Composite material is also defined as a heterogeneous mixture of two or more heterogeneous phases which were bonded together (Kelly, 1994).The earliest example of a composite material were traced back in the third millennium BC when Egyptians made bricks from clay reinforced with straw (Matthews & Rawling, 1999).The Mongolians though produced bows which were made from a combination of wood, animal tendons and silk. As early as1908, the first composite materials were applied for the fabrication of large quantities of sheets, tubes and pipes for electronic purposes. In 1986 for example, aeroplane seats and fuel-tanks were made of natural fibers with a small content of polymeric binders. Today, a renaissance in the use of composite material in technical applications was taking placed in the automobile and packaging industries (Bledzki & Gassan, 1999).



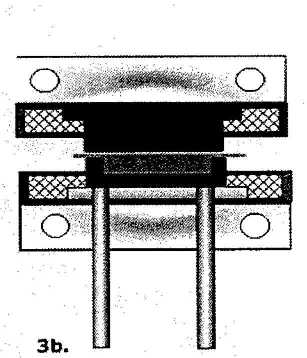


Fig: 2.13a .With the dies apart, the prepared polymer 'dough' is placed into the cavity.

Fig: 2.13b.With the die closed, the article is formed and the small amount of flashing on each side will be removed later.

2.4.1 Introduction to fibre reinforced composite

Textile –reinforced composite materials (TRCM) or part of the general class of engineering materials called composite materials. It is usual to divide all engineering materials into four classes: metals, polymers, ceramics and composites. A rigorous definition of composite materials is difficult to achieve because the first three classes of homogeneous materials are sometimes heterogeneous at submicron dimensions (e.g. precipitates in metals).

A useful working definition is to say that composite materials are characterized by being multiphase materials within which the phase distribution and geometry has been deliberately tailored to optimize one or more properties. This is clearly an appropriate definition for textile-reinforced composites for which there is one phase, called the matrix, reinforced by a fibrous reinforcement in the form of a textile.

2.4.2 Classification of composite materials based on matrix

According to the nature of the matrix, composite materials are classified as metal matrix composites, ceramic matrix composites and polymer matrix composites.

2.4.2.1 Metal matrix composites (MMCs)

Metal matrix composites (MMCs), like all composites, consist of at least two chemically and physically distinct phases, suitably distributed to provide properties not obtainable with either of the individual phases. Generally, there are two phases, e.g., a fibrous or particulate phase, distributed in a metallic matrix. For example, continuous Al_2O_3 fiber reinforced Al matrix composites used in power transmission lines.

As the reinforcement in MMCs serves to reduce the coefficient of thermal expansion (CTE) and increase the strength and modulus in the composites, most of these materials have been developed for the aerospace industries and some are being used in other application such as automobile engines.

Compared to monolithic metal, MMCs have (Composite-by-design, 2009):

- Higher strength-to-density ratios
- Higher stiffness-to-density ratios
- Better fatigue resistance
- Better elevated temperature properties
  - Higher strength
  - Lower creep rate
- Lower coefficients of thermal expansion
- Better wear resistance

However, MMC has the following disadvantages compared to metal itself and the corresponding PMC (Chung, 2003; Composites-by-design, 2009):

- Higher cost of some material systems
- Relatively immature technology
- Complex fabrication methods for fibre-reinforced systems (except for casting)
- Limited service experience

2.4.2.2 Ceramic matrix composite

Ceramic matrix composites (CMCs) combine reinforcing ceramic phases with a ceramic matrix to create materials with new and superior properties (Ceramic Industry, 2009). Ceramic matrix composites (CMCs) have been developed to overcome the intrinsic brittleness and lack of reliability of monolithic ceramics, with a view to introduce ceramics in structural parts used in severe environments, such as rocket and jet engines, gas turbines for power plants, heat shields for space vehicles, fusion reactor first wall, aircraft brakes, heat treatment furnaces, etc. It is generally admitted that the use of CMCs in advanced engines will allow an increase of the temperature at which the engine can be operated and eventually the elimination of the colding fluids, both resulting in an increase of yield. Further, the use of light CMCs in place of heavy super alloys is expected to yield significant weight saving.

Although CMCs are promising thermostructural materials, their applications are still limited by the lack of suitable reinforcements, processing difficulties, sound material data bases, lifetime and cost (Naslain, 2009).

2.4.2.3 Polymer matrices

The matrices in composite materials have the role of transferring the mechanical loading to the fillers (reinforcing materials) and to protect them from the outside environment. The matrices must therefore be quite flexible and offer good. compatibility with the fillers. In addition they must have a low density to keep in the composite's high specific mechanical characteristics (Berthelot, 1999). Polymers are used as the matrix phase for the composites namely polymer matrix composites. The polymer matrices are divided into two types, thermosetting resins and thermoplastic resins (Lubin & Peters, 1998). These two types of matrix have the property of being able to be molded or manufactured in order to yield either a finished product or a semi-finished product whose form can be modified (Berthelot, 1999).

A. Thermosetting resins

Thermosetting resins have a network structure formed exclusively by covalent bonds. Thermosets have a high density of cross-links and are consequently infusible, insoluble, thermally stable, and dimensional stable under load. The major commercial thermosets include epoxies, polyesters, and polymers based on formaldehyde. The formaldehyde-based resins, which are the most widely used thermosets, consist essentially of two classes of thermoset. These are the condensation products of formaldehyde with phenol (or resorcinol) (phenoplasts or phenolic resins) or with urea or melamine (aminoplastics or amino resins) Some basic properties of the selected thermosetting resins are shown.

Thermosetting resins can be processed only once. In fact polymerization by applying heat in the presence of catalyst, these resins lead to a geometric structure that can be destroyed only by a considerable application of thermal energy. It may soften to some extent at elevated temperature. This characteristic is sometimes used to create a bend or curve in tubular structure, such as filament wound tubes. Thermosetting resins are brittle in nature and are generally used with some forms of the filler or reinforcement.

Thermoset polymeric matrix composites can substitute efficiently for steel and concrete in civil constructions, showing greater resistance to oxidation for the former and better freeze thaw for the latter. This behavior could lead to structural component with improved weathering capability (Tyberg *et al.*, 1999).

Properties	Resin materials		
	Polyester	Phenolic	Epoxide
Density (kg/cm$_3$)	1,200	1,200	1,100-1,500
Tensile strength (MPa)	50-80	40	60-80
Tensile elongation (%)	2-5	2.5	2-5
Flexural strength (MPa)	90-130	90	100-150
Heat deflection temperature (^{o}C)	60-100	120	290

Table: 2.9 General characteristic of thermosetting resin (Berthelot, 1999)

Advantages of thermoset composites

These resin materials could be one-part or two-part system and are generally in the liquid state at room temperature. These resin systems are then cured at elevated temperature or sometime at room temperature to get final shape. Manufacturing methods for processing thermoset composites provide the following advantages (Reigner and Sander, 1979)

Processing of thermoset composites is easy because the initial resin system is in the liquid state.

1. Fillers are easy to wet with thermoset, thus voids and porosities are less.
2. Heat and pressure requirements are less in the processing of thermoset composites than thermoplastic composites, thus provide energy savings.
3. A simple low-cost tooling system can be used to process thermoset composites.

Disadvantages of thermoset composites

Thermoset composite materials also have some disadvantages which are (Reigner and Sander, 1979):

- Thermoset composite processing requires a lengthy cure time and thus results in low production rates than thermoplastics.
- Once cured and solidified, thermoset composite parts cannot be reformed to obtain other shapes.
- Recycling of thermoset is an issue.

Physical and chemical treatment may be wedded to enhance the adhesion between reinforcement and matrix.

B. Thermoplastic resins

Almost 85% of polymer products produced worldwide are thermoplastic. Over 70% of the total production of thermoplastics is accounted for by the large volume, low cost commodity resin: polyethylene (PE) of different densities, isostatic polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC). Next in performance and cost are acrylics, acrylonitrile-butadiene-styrene (ABS) terpolymers, and high-impact polystyrene (HIPS) (Xanthos, 2005). Table 2.2 lists the densities, tensile strengths, impact strengths, and maximum-use temperatures for some selected engineering thermoplastics.

Thermoplastic materials are, in general, ductile and tougher than thermoset materials and are used for wide variety of non-structural applications. Thermoplastics can be re-melted by heating and solidified by cooling, which render them capable of repeated reshaping and reforming. Thermoplastic can be either amorphous or semi-crystalline as shown in Figure 2.5 and Figure 2.6. In amorphous thermoplastics, molecules are randomly arranged, whereas in the crystalline region of semi-crystalline plastics, molecules are arranged in an orderly fashion (Mazumdar, 2002).

Materials	Density (g/cm³)	Tensile strength (MPa)	Impact strength Izod (J/m)	Max-use temperature (°C)
Polypropylene	0.9	35	27.5	135
Polyphenylene Oxide	1.06-1.10	53.82-66.24	267	80-105
Polyphenylene Sulphide	1.34	69	16	260

Table: 2.10 Properties of selected engineering thermoplastics (Smith, 2004)

Fig: 2.14 Molecular arrangements in amorphous polymers (Mazumdar, 2002)

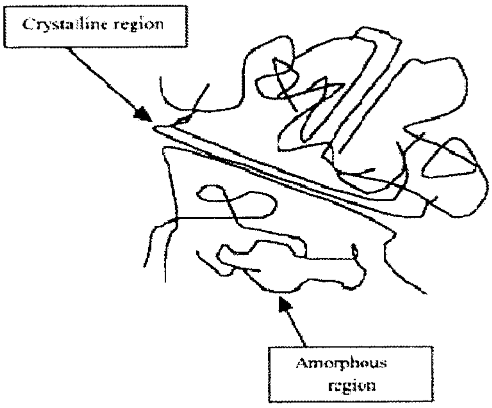


Fig: 2.15 Molecular arrangements in semi-crystalline polymers

2.5 FIBER ORIENTATION

Fibre orientation is another important parameter that influences the mechanical behaviour of short-fibre composites .This is because the fibre in such composites are rarely oriented in a single direction, which is necessary for the fibres to offer maximum reinforcement effects. As a result, the degree of reinforcement in a short-fibre composite is found to be strongly dependent on the orientation of each individual fibre with respect to the loading axis. Changes in fibre orientation take place continuously and progressively during the processing of short-fibre composites.

The changes are related in a complex way to the geometrical properties of the fibres, the viscoelastic properties of the matrix, and the change in shape of the material which is produced by the processing operation. In these operations, the polymer melt will undergo both elongation or extensional flow and shear flow. The effect of these flow processes on the fibre orientation is illustrated in Figure Thomas *et al.* (1996) reinforced polystyrene with benzoylated sisal fibres. The influences of fibre length, fiber content as well as fibre orientation on the composite produced were evaluated.

Randomly oriented composite specimens were prepared by injection molding while the unidirectional oriented composite specimens were prepared by a combination of injection molding and compression molding. The specimens were then observed under an optical stereomicroscope to confirm fibre orientation. A longitudinally oriented composite (the fibres are oriented parallel to the test direction) was found to show the best mechanical properties. This was followed by randomly and transversely oriented composites ie, the fibres are oriented transverse to the test direction.

Advantages of thermoplastic composites

The advantages of thermoplastic composites include (Lubin & Peters, 1998):

1. The processing for thermoplastic composites can be faster than for thermoset composites since curing reaction is required. Thermoplastic

2.5.1 Fillers/Reinforcements

In general, filler are defined as materials that are added to the formulation to lower the compound cost. For effective utilization of fillers, a complete understanding of individual characteristics is essential. Each class of the fillers appears to exhibit specific characteristics which make them especially suited for the given application (Lutz & Grossman, 2000). Fillers, being much stiffer and stronger than the polymer, usually increase its modulus and strength. Thus, mechanical property modification may be considered as their primary function, although their presence may significantly affect thermal expansion, transparency, thermal stability, etc (Xanthos, 2005).

Selecting of fillers is not just an art but a science and various factors would have to be considered in the choice such as (Shenoy, 1999)

- Cost and availability
- Wettability or compatibility with the polymer
- Effect on polymer flow characteristics
- Physical properties

2.5.2 Classification of filler/reinforcement

A. Fibres reinforcement

Fibre reinforced materials have been around for thousands of years. The earliest fibrous reinforcements were straw or horsehair for toughening mud in order to make bricks. Modern composites are reinforced with fibres such as glass, carbon or Kevlar. There are also particulate reinforcements using materials such as silica flour, glass beads and sand and there are now even lamellar reinforcement's possible (De & White, 1996).

Fibres for composite materials can come in many forms, from continuous fibres to discontinuous fibres (Figure 2.9), organic fibres to inorganic fibres while the properties of fibre-reinforced composites are strongly dependent on the way the fibres are laid in the composites. The important thing in fibre-reinforced composites is that the fibres carry the load and the composite strength is greatest along the axis of the fibre. Long continuous fibres in the direction of the load result in a composite with the properties far exceeding the matrix resin itself. The same material chopped in the short length yields lower properties than continuous fibres. Depending on the types of application (structural or non-structural) and manufacturing method, the fibre form is selected. For structural applications, continuous fibres or long fibres are recommended; whereas for non-structural applications, short fibres are recommended.

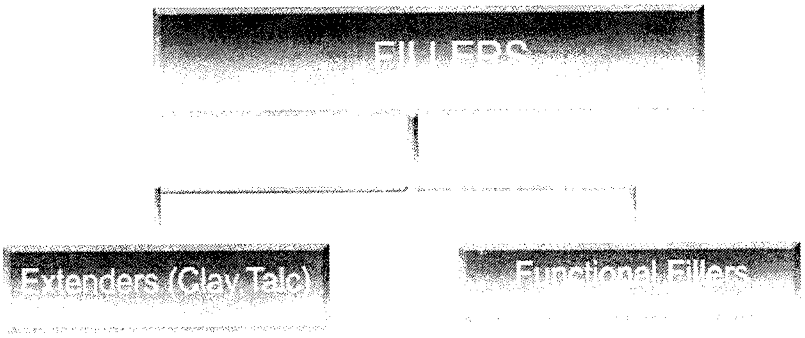


Fig: 2.16 Types of fillers

i. Extenders

The extender fillers primarily occupy space and are mainly used to lower the formulation cost[1,8,9].

Properties of extender fillers

In general, ideal extender filler should

- Be spherical to permit retention of anisotropic properties.
- Have an appropriate particle size distribution for particle packing.
- Cause no chemical reactivity with the polymer or the additives
- Have low specific gravity.
- Have desirable refractive index and colour.
- Be low in cost.

ii. Functional fillers

Functional fillers on the other hand have a definite and required function in the formulation apart from lowering the formulation cost. However, some of the extender fillers when reduced to a finer particle size and/or surface treated would perform as functional filler. Fillers that are functional in one polymer may be merely extenders in another polymer. Such factors seriously complicate the task of establishing sharp boundary lines between extenders and functional fillers in terms of their generic composition; however, on the performance basis they can be separated as shown in the fig: 2.17

Fillers: Types, Properties, and Performance

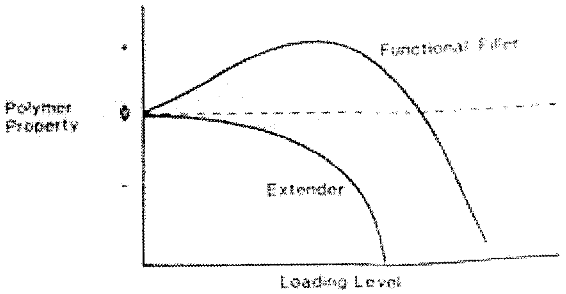


Figure 1 Characteristics of functional and extender fillers

Therefore, the extender fillers basically lower the formulation cost and increase the flexural modulus, whereas the functional fillers provide at least one specifically required function in the formulation.

2.5.3 CLASSIFICATION BASED ON TYPE

A. Particulate fillers

Particulate fillers are divided into two types, inert fillers and reinforcing fillers. The term inert filler is something of a misnomer as many properties may be affected by incorporation of such filler. For normal uses, such fillers should be quite insoluble in any liquids that the polymer compound is liable to come into contact. Each type of filler may differ in the following ways:

- Average particle size and size distribution.
- Particle shape and porosity.
- Chemical nature of the surface.
- Impurities such as grit and metal ions.

The common observation is that the finer the particle sizes the higher the values of the tensile strength, modulus and hardness. Coarser particles will tend to give less strong compounds than the virgin material (i.e. material without the filler), but if the particle size is fine there is an enhancement of the mechanical properties and the phenomenon is called as reinforcement.

Impurities in the fillers can have serious effects on the polymer compound. Coarse particles lead to points of weakness in soft polymers and will therefore fail under stresses below that which might be expected. In general, the phenomenon of reinforcement appears to depend on three factors:

composites only require heating, shaping and cooling.

2. The properties are attractive, in particular, high delamination resistance and damage tolerance, low moisture absorption and the excellent chemical resistance of semi-crystalline polymers.
3. In light of environmental concern, thermoplastic composites offer advantage. They have very low toxicity since they do not contain reactive chemicals (therefore storage life is infinite). It is possible to remelt and dissolve such thermoplastics; their composites are also easy recycled or combined with other recycled materials in the market for molding compound.

F. Disadvantages of thermoplastic composites

The disadvantages of thermoplastic composites include:

1. Thermoplastic composites require heavy and strong tools for processing. Moreover, the cost of the tools is high. For example the injection molding equipment costs more than $50,000, whereas the mandrel equipment for filament winding process costs less than $500.

- An extensity factor – the total amount of surface area of filler per unit volume in contact with the polymer.
- An intensity factor – the specific activity of the filler – polymer interface causing chemical and/or physical bonding.
- Geometrical factors such as structure and porosity of the particles.

B. Rubbery fillers

Rubbery fillers are often incorporated into rigid thermoplastics to improve their toughness. The increase in toughness of the material leads to significant increase in the fracture resistance of the material.

C. Fibrous fillers

Fibrous fillers have been long used in plastic materials. Fibrous fillers are often embedded in the laminar form. The fibers have higher modulus than the resins in which they are embedded so that when the composite of resin plus the fiber is strained in the plane of the fibrous layer the bulk of the stress is taken up by the fiber. As a result of this, both the strength and the modulus are enhanced when compared to the unfilled resin.

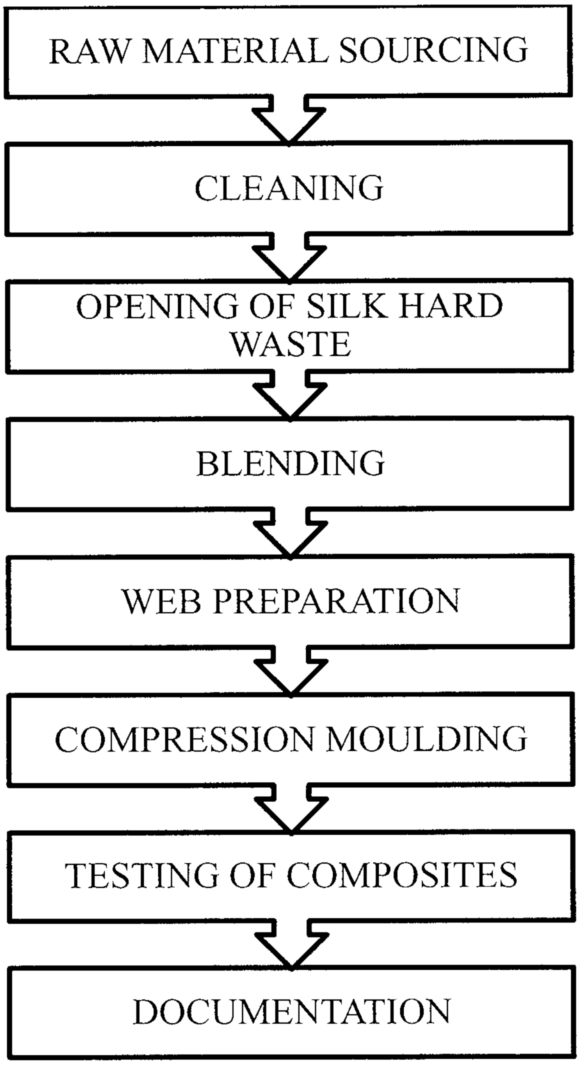
CHAPTER III

MATERIALS AND METHODS

3.1 INTRODUCTION

This chapter explains the methodologies, materials, evaluation techniques used to study the composite properties and sound absorption properties of silk and polypropylene fibre reinforced composites.

3.2 METHODOLOGY



3.3 RAW MATERIAL SOURCING

The silk selvedge waste is collected from M/s Ethnic fashion, Bangalore. The properties of the silk fibres are given in the following table.

SILK

LENGTH (mm)	75.6
FINENESS (Tex)	3.7
TENSILE STRENGTH	6.67g/d

POLYPROPYLENE

LENGTH (mm)	51
FINENESS (Tex)	2.5
TENSILE STRENGTH	5.5-6g/d

Table: 3.1 Physical Properties of silk & polypropylene

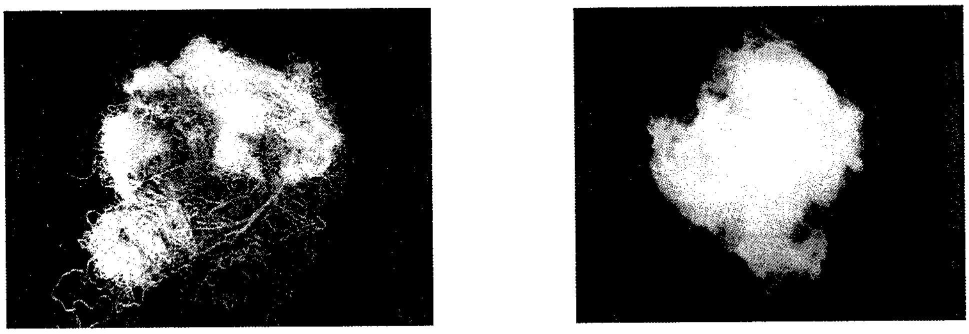


Fig: 3.1 silk & polypropylene

3.4 CLEANING

The shuttle less loom silk selvedge consists of non silk materials like cotton leno threads. The selvedge waste is occurred in strip form in the weaving machine. The horizontal threads are silk threads. The vertical threads are cotton threads. These cotton leno threads are removed from the silk threads.

3.5 OPENING OF SILK HARD WASTE

The cut silk threads are converted into fibre form using carding machine which contains lickerin, doffer, and cylinder. The silk threads are fed into a carding machine which is opened into fibrous form by beating action.

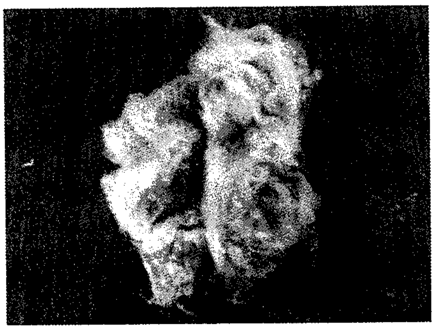


FIG: 3.2 Opened hard silk waste

3.6 BLENDING

Blended fibres/blends combine 2 or more fibre substances into a single fibre strand or yarn with modified or changed properties and appearance of the fibre, yarn or fabric. Each fibre has separate set of physical and aesthetic characteristics inherent in its design. The blended fibres are made to create the kind of fibre required to meet the specific needs of the industry. Fibre blends have their own characteristics depending upon the type and the percentage of the specific fibres used. Each fibre in a blend adds not only favourable properties but also undesirable properties. Fibre blends however utilize the advantages of all fibre to counteract the disadvantages of each single fibre. Man-made fibres can be blended with one or more other fibers, either natural or man-made. The silk and polypropylene fibres are mixed manually in the blend ratio of 50:50. They are well opened by hand to get a homogeneous blend.

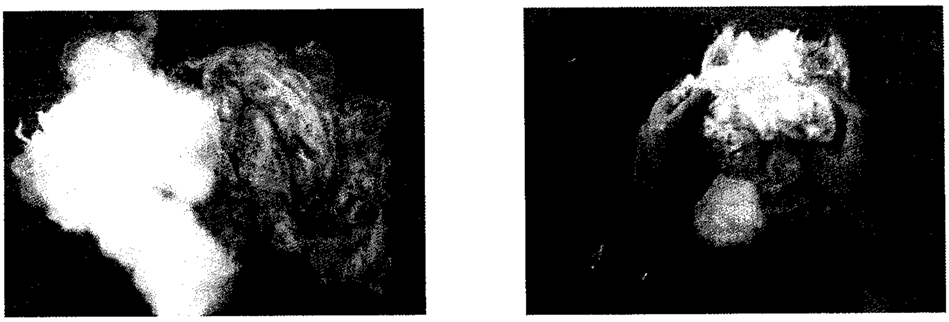


Fig: 3.3 shows the mixing of fibres

3.7.1 WEB FORMATION

CARDING

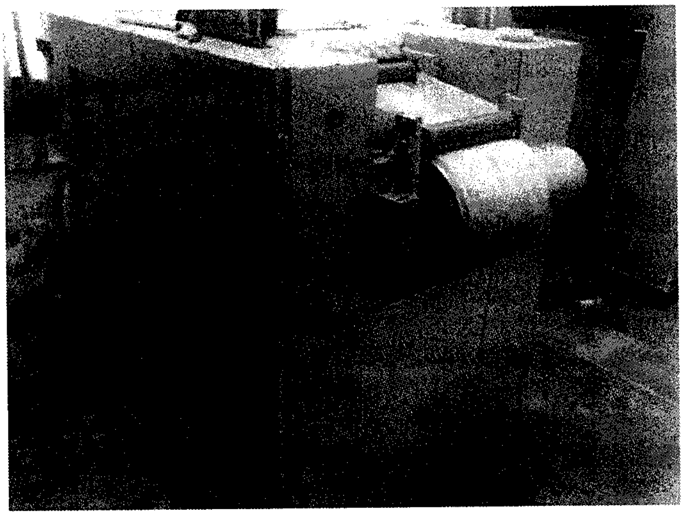
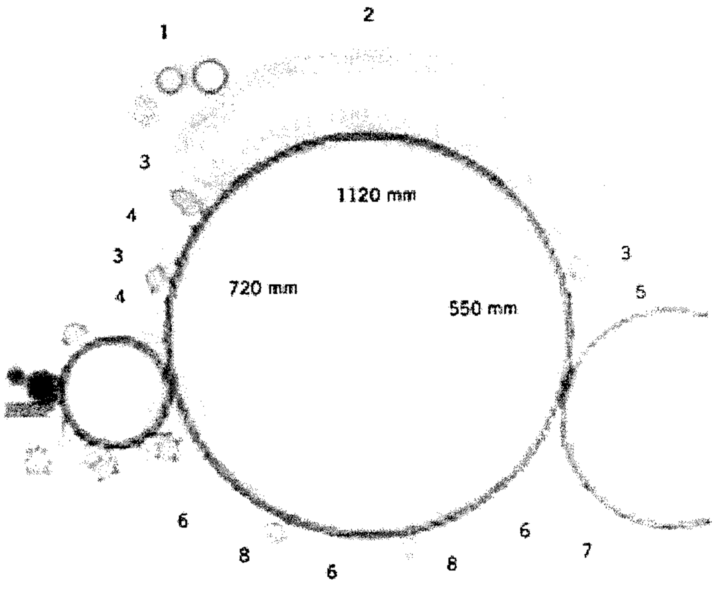


Fig: 3.4 carding machine



The blended silk and polypropylene fibres are carded three times to get a well oriented web. The carding process is done using tritex mini carding machine at TIFAC core, Kumaraguru College of Technology.

The six webs are super imposed one above the other to get an average GSM of 1300.

3.8 OPTIMISATION OF PROCESS PARAMETERS BY BOX BEHNKEN METHOD

In statistics, Box–Behnken designs are experimental designs for response surface methodology, devised by George E. P. Box and Donald Behnken in 1960, to achieve the following goals:

- Each factor, or independent variable, is placed at one of three equally spaced values. (Atleast three levels are needed for the following goal.)
- The design should be sufficient to fit a quadratic model, that is, one containing squared terms and products of two factors.
- The ratio of the number of experimental points to the number of coefficients in the quadratic model should be reasonable (in fact, their designs kept it in the range of 1.5 to 2.6).
- The estimation variance should more or less depend only on the distance from the centre (this is achieved exactly for the designs with 4 and 7 factors), and should not vary too much inside the smallest (hyper)cube containing the experimental points.

The design with 7 factors was found first while looking for a design having the desired property concerning estimation variance, and then similar designs were found for other numbers of factors.

Each design can be thought of as a combination of a two-level (full or fractional) factorial design with an incomplete block design. In each block, a certain number of factors are put through all combinations for the factorial design, while the other factors are kept at the central values. For instance, the Box–Behnken design for 3 factors involves three blocks, in each of which 2 factors are varied through the 4 possible combinations of high and low. It is necessary to include centre points as well (in which all factors are at their central values).

In this table, *m* represents the number of factors which are varied in each of the blocks. The design for 8 factors was not in the original paper. Designs for other numbers of factors have also been invented (at least up to 21). A design for 16 factors exists having only 256 factorial points.

Most of these designs can be split into groups (blocks), for each of which the model will have a different constant term, in such a way that the block constants will be uncorrelated with the other coefficients.

SL.No.	Temperature	Time	Pressure
S1	175	15	45
S2	175	7	45
S3	175	15	35
S4	180	7	40
S5	165	15	40
S6	175	7	35
S7	180	11	35
S8	175	11	40
S9	180	11	45
S10	180	15	40
S11	165	7	40
S12	165	11	35
S13	165	11	45

Table: 3.2 Optimisation of Process Parameters

3.9 COMPOSITE MANUFACTURING

Compression moulding machine is used to produce the silk fibre reinforced polypropylene composites. The process parameters involved in the compression moulding are temperature, pressure, time and cooling time. Three parameters are selected for optimisation of the composites i.e. temperature, time and pressure. Design Expert software is used for designing the experiments. The Box behnken three factor three level design is used to analyse the output value i.e. tensile strength. 13 composites are prepared for the study. They are

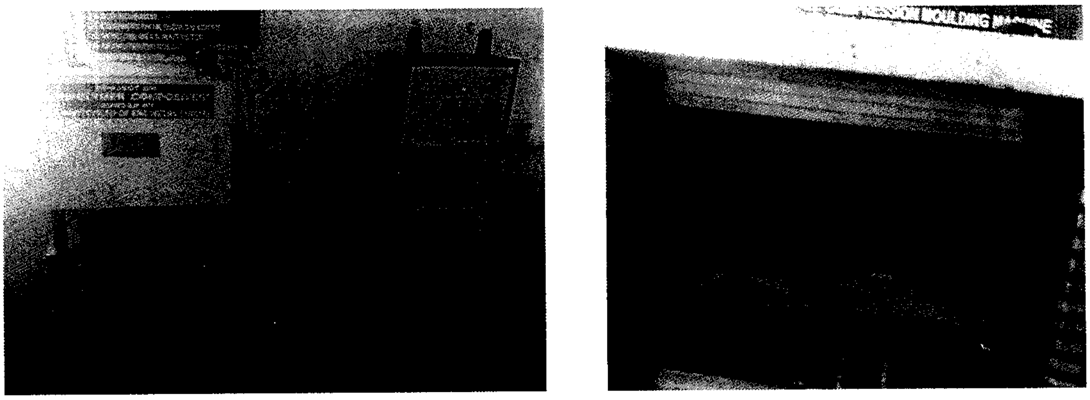


Fig: 3.5 compression moulding machine

3.10 TESTING OF SILK AND POLYPROPYLENE FIBER COMPOSITE

The composites are prepared by different parameters (temperature, pressure, time)as mentioned above for silk and wool composite. The prepared composite were tested in terms of mechanical and sound absorption properties. In order to test such processes the following test methods are used.

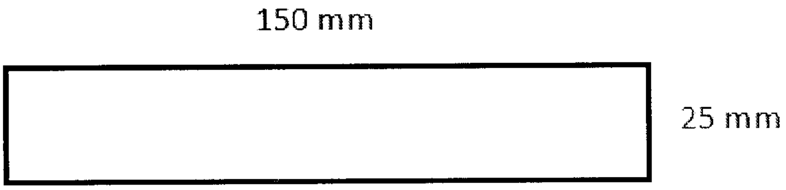
3.10.1 Tensile Strength Test

The breaking strength is a measure of the resistance of the material to a tensile load. The composite tensile strength is usually measured in tensile tester with constant rate of loading.

Sample Preparation

Sample size for testing the tensile strength is following,

- Length = 150 mm
- Width = 25 mm



Test Procedure

Fix the sample in between two jaws and bottom jaw is movable one. After the sample is fixed the bottom jaw is moving at the principle of Constant Rate of Loading (CRL).

The tensile tester shows the data in breaking load in Newton and Elongation at Break.

3.10.2 FLEXURAL STRENGTH

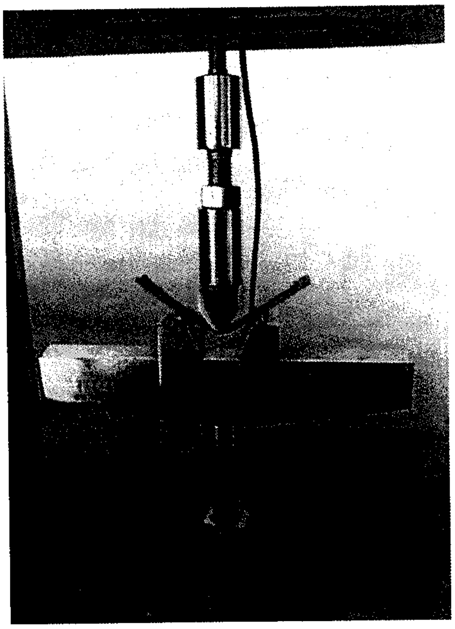


Fig: 3.6 Flexural Strength

The UCT 10kN is fully controlled from the accompanying PC with the professional Tram-QA software that can run virtually any test fully automatically and at the same time automatically collect testing results and store them in a database. Tram-QA offers the user a fast and effective way of generating and presenting a quality report. Additionally, Tram-QA offers a data filing and retrieving system giving the user instant access to reports on any previously tested samples. Much more than just the strength test results can be included in the quality report.

The Tram-QA software offers a simple way to incorporate all relevant quality parameters in a spreadsheet that can be set up for virtually any application. It is ideally suited to quality assurance and process control as well as research and development. It is capable of performing a wide range of complex testing procedures and can be set up for relevant international or national standards.

The bench-mounted machine is compact, and it can be supplied with different stroke lengths, and optional a second testing positions above the travelling beam. The machine is built around a two-spindle design. This machine design provides optimum stiffness resulting in accurate deformation measurements. Testing is controlled from the computer and a test is started by pressing a single key after keying in the sample identification, if any. During the testing the load-deformation curve is simultaneously generated on the monitor.

The machine is supplied with grips and loading plates to suit the products to be tested. Automatic positioning of the loading beam virtually eliminates the waiting time between tests. Additionally it incorporates a facility to connect it to an external device such as a balance so that results can be directly transferred to system for further processing.

SPECIFICATIONS:

Load cell Capacity	10000 N
Weight	145 kg
Max stroke length	200 mm
Daylight between columns	450 mm
Vertical daylight	650 mm
Frame stiffness	50 kN/mm
Resolution	0.05 N
Supply Voltage	230V 50Hz or 115V 60Hz
Speed Range	0.001mm/min - 500mm/min
Height	1260 mm
Width	750 mm
Depth	310 mm
Force measuring error	Less than 1% of actual value from max. force down to 1/1000 of max. force
Second testing space	Optional second testing space above the travelling beam.
Additional sensors	Optional Thickness gauge, Extensometer, extra load cells, balance etc. can be connected
Loading speed control	Selectable load- or travelling speed. Programmable loading sequence for semiautomatic testing as well as automatic fast return.

Table: 3.3 Specifications of Flexural Strength

3.10.3 Thermal Conductivity of Bad Conductors

Procedure

Formula: The coefficient of thermal conductivity of a bad conductor

$$K = \frac{Msd\,(r + 2l)\ (d\theta/dt)}{\pi r^2\,(2r + 2l)\,(\theta_1 - \theta_2)} \quad \text{Watt/ Metre/ Kelvin}$$

The Lees disc set up is arranged as shown in fig. Steam is passed through the chamber. As heat gets conducted into the brass disc through the bad conductor, it gets heated up. The temperature is noted from time to time. At one stage the temperature becomes steady. When the temperature becomes steady for atleast 10 minutes the steady temperature is noted ($\theta_2{}^0C$)

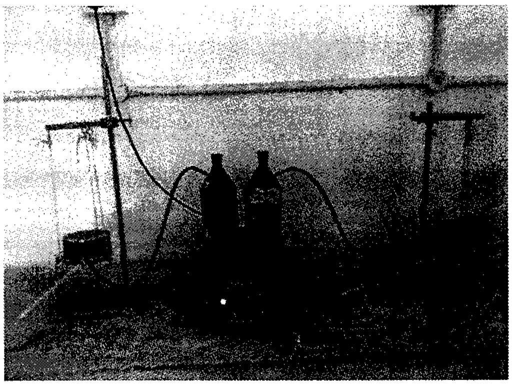


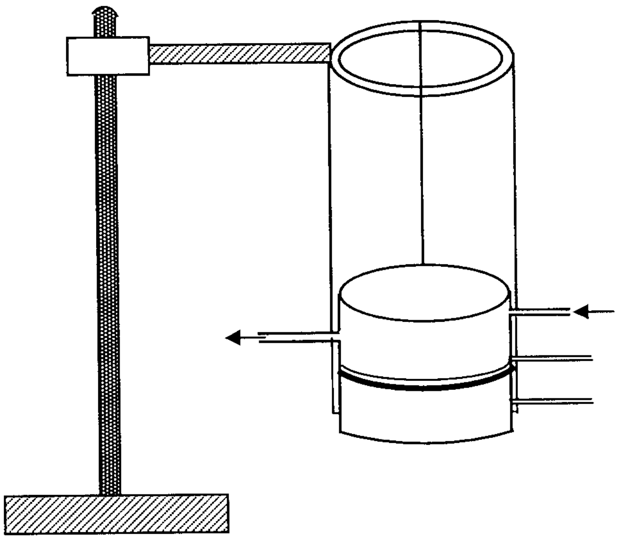
Fig: 3.7 Shows the Lee's Disc Apparatus

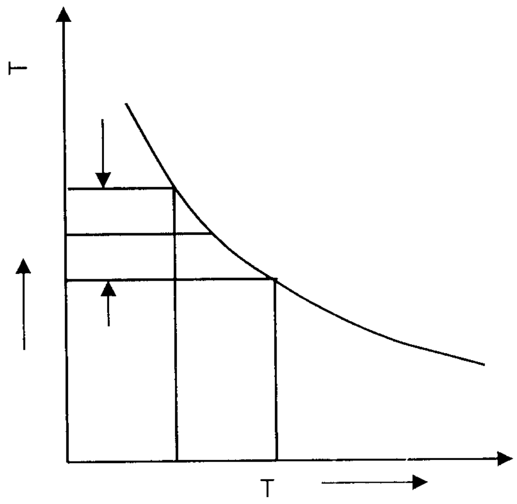
The temperature of steam is noted ($\theta_1{}^0C$). Now the cardboard is removed and the brass disc is heated in direct contact with the steam chamber until the temperature rises by about 5^0C above the steady temperature

The disc is now separately suspended from the ring after removing from the steam chamber. Temperatures are noted in steps of 30secs from $(\theta_2 + 5)\ ^0C$ to $(\theta_2 - 5)\ ^0C$ and the values are tabulated (Table). A graph is drawn with temperature on the Y axis and the time on the X axis.

A horizontal line is drawn corresponding to steady temperature $\theta_2\ ^0C$. The time dt for a fall of temperature of $\theta_2\ ^0C$ is found by taking two points one degree above 2 and the other one degree below

Note down the mass (M) of the brass disc B as noted over it or determine its mass using balance. It's diameter is found and hence the radius (r) is determined using vernier callipers. The thickness (l) of the brass disc is found using vernier callipers and thickness of the bad conductor (d) with a screw gauge.





$$\text{L east Count for screw gauage} = \frac{\text{Pitch}}{\text{Total number of head scale division}}$$

$$\text{Pitch} = \frac{\text{Distance move on pitch scale}}{\text{Number of rotation made head scale}}$$

CALCULATIONS

FORMULA

The coefficient of thermal conductivity of a bad conductor

$$K = \frac{Msd\,(r + 21)\;(d\theta/dt)}{\pi r^2\,(2r + 21)\,(\theta_1 - \theta_2)} \text{ Watt/ Metre/ Kelvin}$$

Where,

M - Mass of the disc placed over the experimental disc X 10^{-3} (Kilogram)

S -Specific heat of the material of the disc (Joule/Kilogram/Kelvin)

d - Thickness of the bad conductor in (metres) r - Radius of the brass disc in (metres)

l - Thickness of the brass disc in (metres)

θ_1 - temperature of the steam in (Degree Celsius)

θ_2 -Steady temperature in (Degree Celsius)

$d\theta/dt$ - rate of heat radiation of the brass disc at θ_2 (Kelvin/ second)

3.10.4 Fabric Thickness Gauge

Introduction

Determination of thickness of fabric samples in laboratory is usually carried out with the help of a precision thickness gauge. In this equipment, the fabric whose thickness is to be determined is kept on a flat anvil and a circular pressure foot is pressed on to it from the top under a standard fixed load. The Dial Indicator directly gives the Thickness in mm.

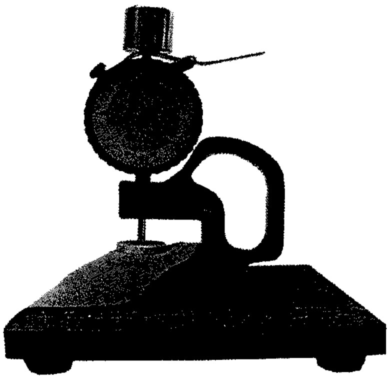


Fig: 3.8 Thickness Gauge

Fabric Testing Instrument determines the thickness of jari, yarn, fabric, paper, and film samples etc accurately. At B-Tex Engineering Fabric Thickness Gauge is calibrated, of extreme fine quality and accurately and conveniently performs testing pertaining to thickness measurement. In Fabric Thickness Gauge, the fabric whose thickness is to be determined is kept on a flat anvil and a circular pressure foot is pressed on to it from the top under a standard fixed load. Then the Dial Indicator directly gives the thickness in mm.

Specifications of Fabric Thickness Gauge

Range of measurement	: 0 - 10 mm
Least count of dial gauge	: 0.01 mm

Diameter of anvil : 60 mm
Diameter of pressure foot : 10 mm & 25 mm
Overall Dimensions of
Instrument : 115(W) x 185(D) x 180(H) mm
: 1.9 Kg. (4.18 lbs)
Net Weight of the Instrument

Fabric Thickness Gauge confirms with the following international standards

ASTM D 1777

3.10.5 Scanning Electron Microscopy (SEM)

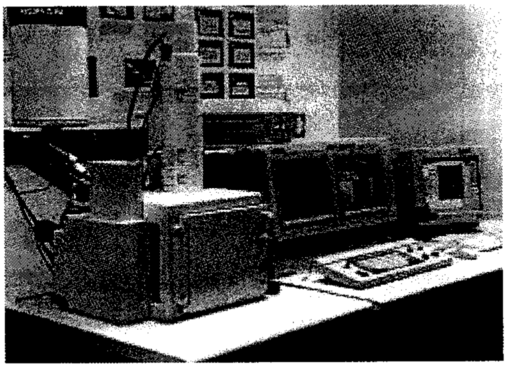


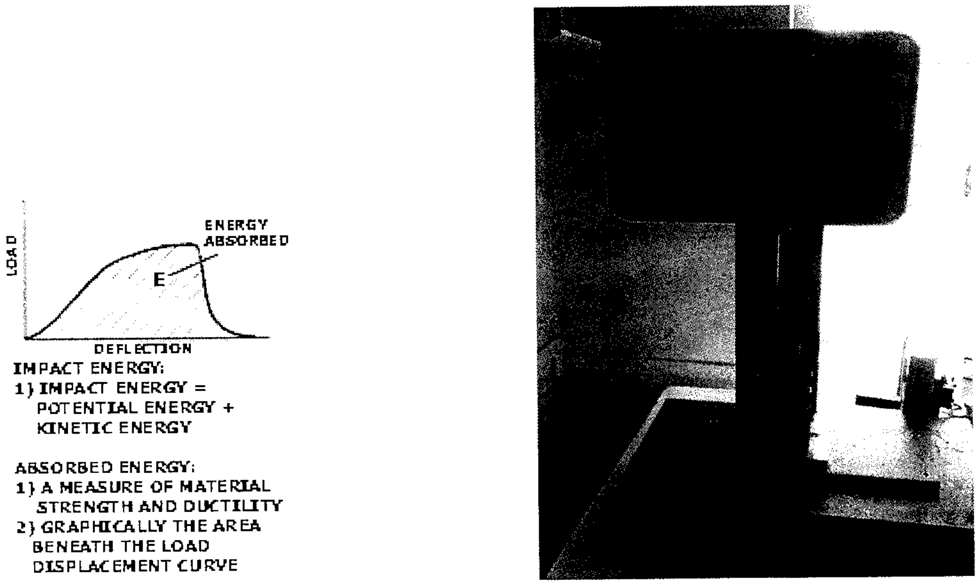
Fig: 3.9 SEM

A typical SEM instrument, showing the electron column, sample chamber, EDS detector, electronics console, and visual display monitors. The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample.

3.10.6 IMPACT TESTING

Impact testing is testing an object's ability to resist high-rate loading. An impact test is a test for determining the energy absorbed in fracturing a test piece at high velocity. Most of us think of it as one object striking another object at a relatively high speed.

Impact resistance is one of the most important properties for a part designer to consider, and without question, the most difficult to quantify. The impact resistance of a part is, in many applications, a critical measure of service life. More importantly these days, it involves the perplexing problem of product safety and liability.



IMPACT ENERGY:
1) IMPACT ENERGY = POTENTIAL ENERGY + KINETIC ENERGY

ABSORBED ENERGY:
1) A MEASURE OF MATERIAL STRENGTH AND DUCTILITY
2) GRAPHICALLY THE AREA BENEATH THE LOAD DISPLACEMENT CURVE

Fig: 3.10 Impact Testers

One must determine:

1. The impact energies the part can be expected to see in its lifetime,
2. The type of impact that will deliver that energy, and then
3. Select a material that will resist such assaults over the projected life span.

The equipment offers a great opportunity to improve quality management through instantaneous data generation. In spite of the highly advanced and automatic testing procedure operation is easy.

Due to the modular design of the equipment and the versatility of the software, tailored systems can be offered at a moderate price. Special systems for board, sheet and slab manufacturing industries can also be supplied.

3.10.7 WATER UPTAKE

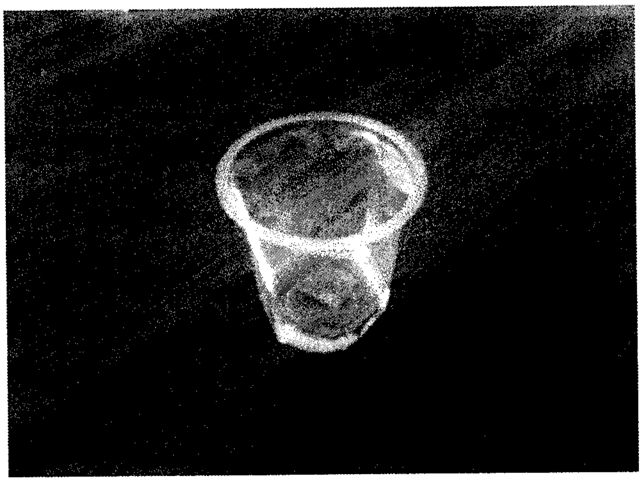


Fig: 3.11 Water up Take

Water absorption testing standards AS4459.3, Determination of water absorption, apparent porosity, apparent relative density and bulk density provides the means to classify ceramic tiles along with their method of manufacture according to AS 4662 Ceramic tiles - Definitions, classification, characteristics and marking. This is important when assessing the requirements that the ceramic tiles must achieve for them to be are considered the best commercial quality

The sample size of 1X1cm is taken, which is been placed in to a beaker consist of 100 ml of distilled water in it. And the sample is be weighted at regular interval of time until it reaches its constant weight.

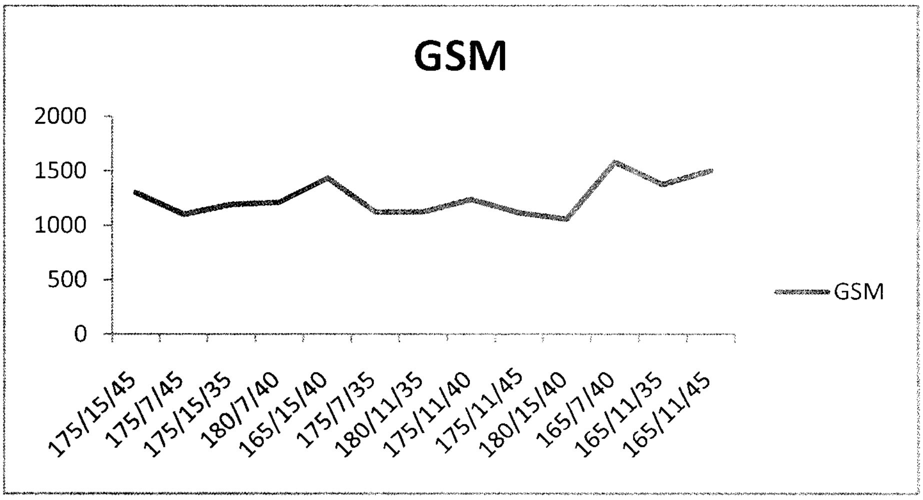
CHAPTER IV

RESULTS AND DISCUSSION

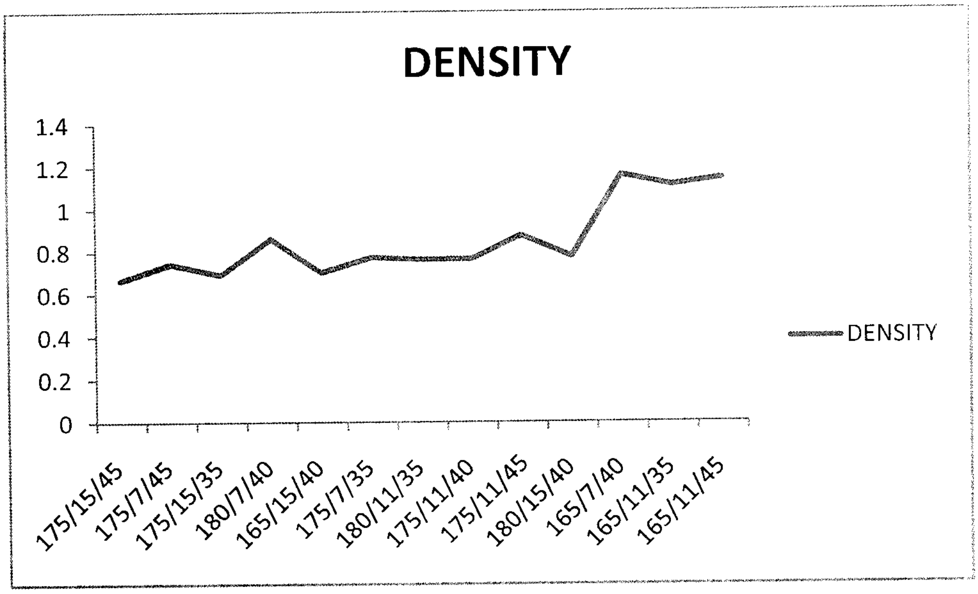
4.1 Testing results

TEMP/TIME/PRESSURE	THICKNESS	MASS(gm)	GSM	DENSITY	TENSILE STRENGTH	THERMAL CONDUCTIVITY
175/15/45	0.1612	12.980	1298	0.6654	15.24	0.000556
175/7/45	0.1226	11.010	1101	0.7421	31.37	0.000435
175/15/35	0.1424	11.900	1190	0.6906	14.27	0.000498
180/7/40	0.1162	12.100	1210	0.8605	26.75	0.000704
165/15/40	0.1692	14.340	1434	0.7007	29.29	0.000379
175/7/35	0.1200	11.210	1121	0.7720	25.75	0.000590
180/11/35	0.1942	9.240	9240	0.3932	15.47	0.000756
175/11/40	0.1338	12.380	1238	0.7646	22.67	0.000722
175/11/45	0.1054	11.150	1115	0.8742	29.22	0.000533
180/15/40	0.1128	10.560	1056	0.7736	37.67	0.000412
165/7/40	0.1126	15.780	1578	1.1581	15.86	0.000251
165/11/35	0.1176	13.740	1374	1.1089	14.55	0.000356
165/11/45	0.1894	14.980	1498	1.1424	22.15	0.000367

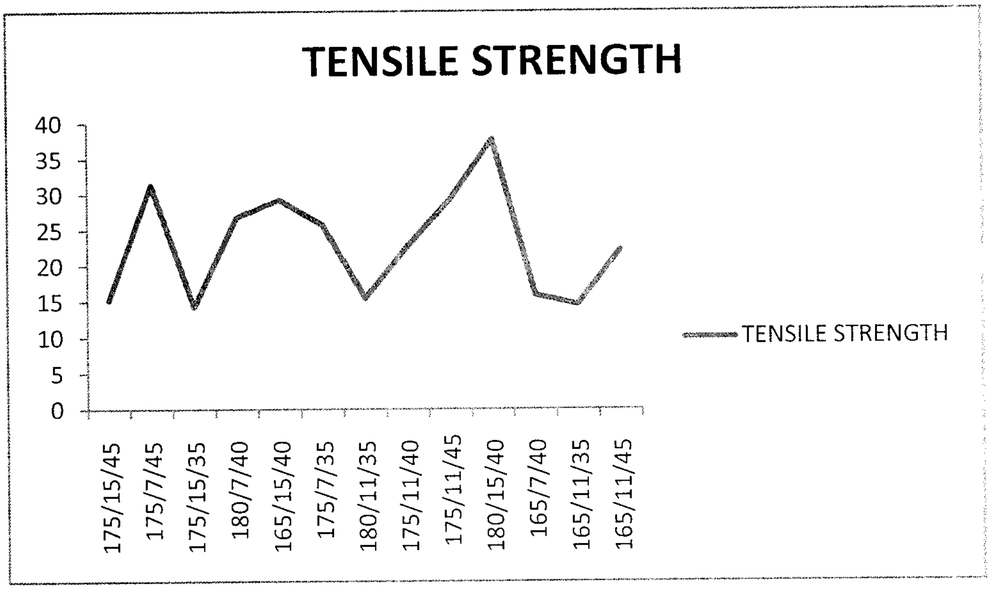
Table: 4.1 Result For GSM, Tensile Strength and Thermal Conductivity



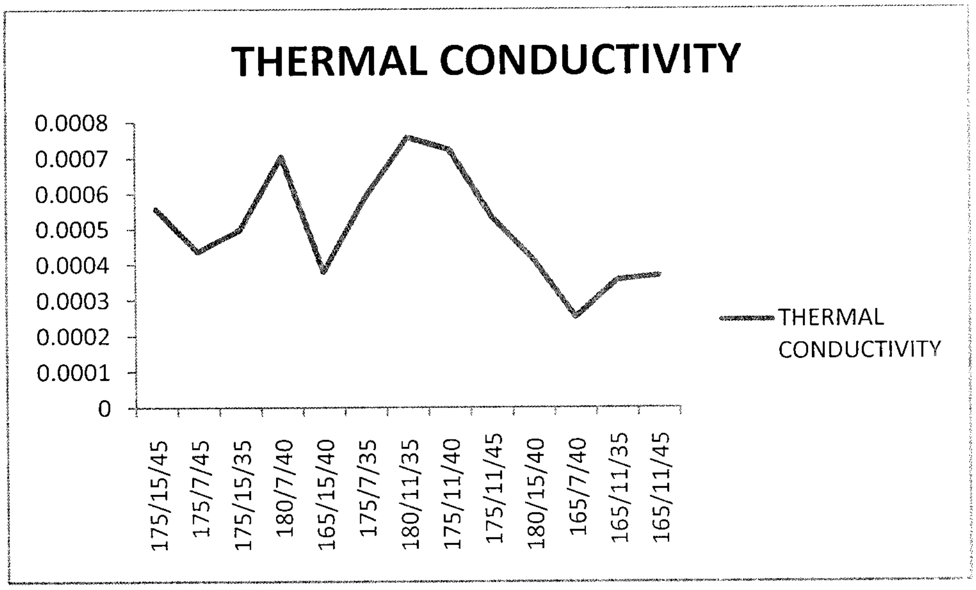
From the above graph the results shows the GSM of silk polypropylene composites. As a result we came to know that sample 11th gives the highest value and the sample 7th gives the lowest.



From the above graph the results shows the Density of silk polypropylene composites. As a result we came to know that sample 11th gives the highest value and the sample 7th gives the lowest.



The above graph indicates the tensile strength results of 13th samples in which 10th sample have the highest value and 3rd sample has the lowest.

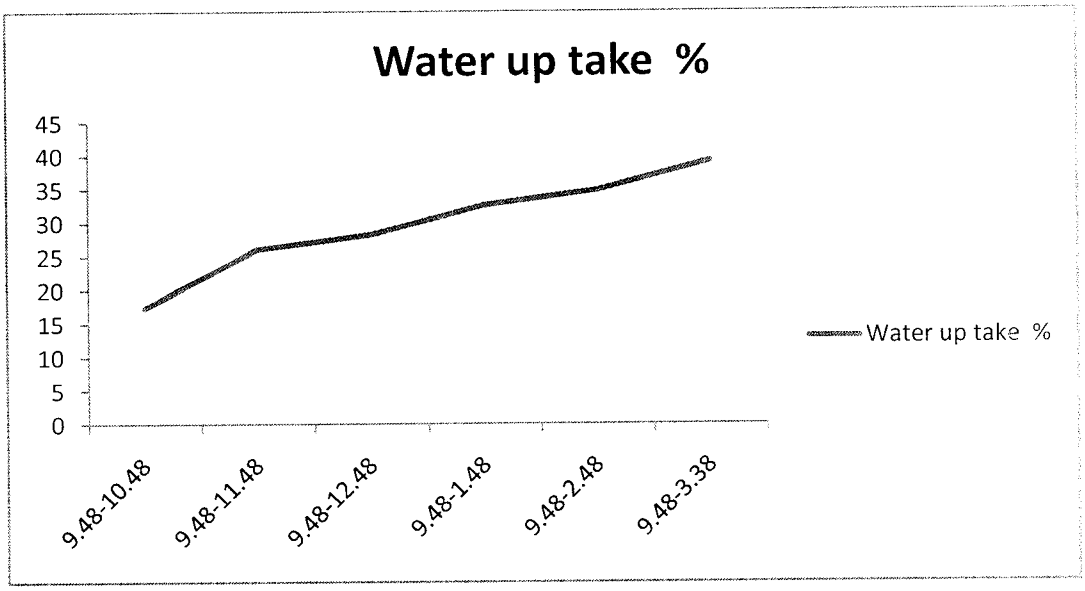


From this above graph of thermal conductivity of silk reinforced polypropylene 7th sample is having highest value of 0.000756 and the 11th sample got lowest 0.000251.

4.2 WATER UPTAKE

WATER UP TAKE (175°C/7mins/35bar)	
Minutes	**Water up take %**
60	17.391
120	26.086
180	28.26
240	32.6
320	34.78
380	39.13

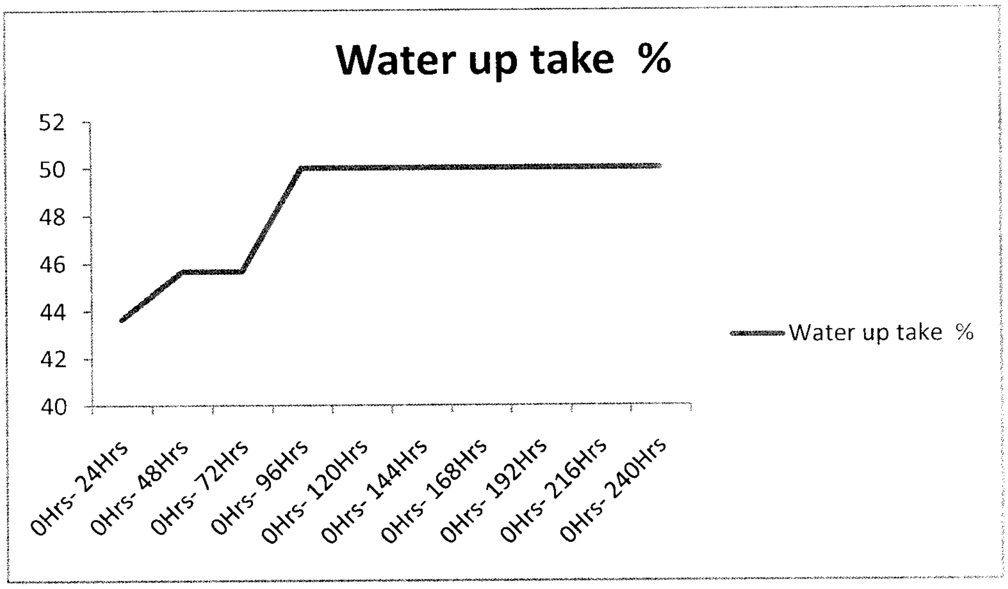
Table: 4.2 Water Up Take Every 1hr



As the result of water up take this graph shows the gradual increase in weight of the optimized sample. The weight of the sample after 60 mins of immersion in to distilled water is 17.391 the same sample is increased it weight up to 39.13 after 380mins.

WATER UP TAKE (175°C/7mins/35bar)	
Hours	**Water up take %**
24Hrs	43.65
48Hrs	45.65
72Hrs	45.65
96Hrs	50
120Hrs	50
144Hrs	50
168Hrs	50
192Hrs	50
216Hrs	50
240Hrs	50

Table: 17 Water Up Taker Every 24hr

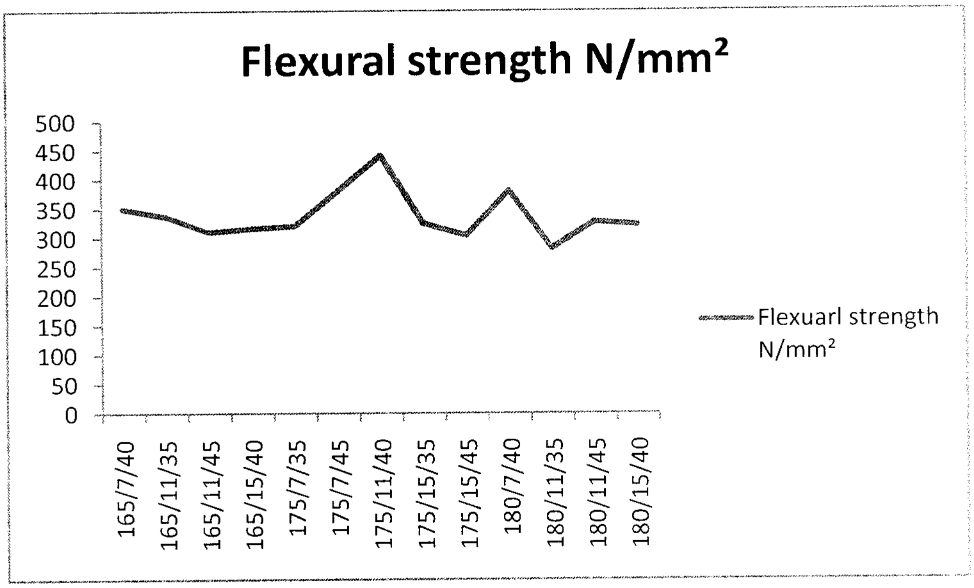


The above graph shows the result of random tests taken on the optimised sample with an interval of 24 hrs which shows the gradual increase in weight. The weight of the sample after 24hrs of immersion in to distilled water is 43.65 the same sample is increased it weight up to 50 after 96hrs. After the 96hrs the weight of the sample reminds constant.

4.3 FLEXURAL STRENGTH

Samples	Flexural strength N/mm^2
165/7/40	350.56
165/11/35	336.87
165/11/45	310.45
165/15/40	316.38
175/7/35	320.51
175/7/45	379.98
175/11/40	441.49
175/15/35	324.34
175/15/45	303.56
180/7/40	380.17
180/11/35	281.47
180/11/45	327.17
180/15/40	321.34

Table : 4.4 Flexural Strength

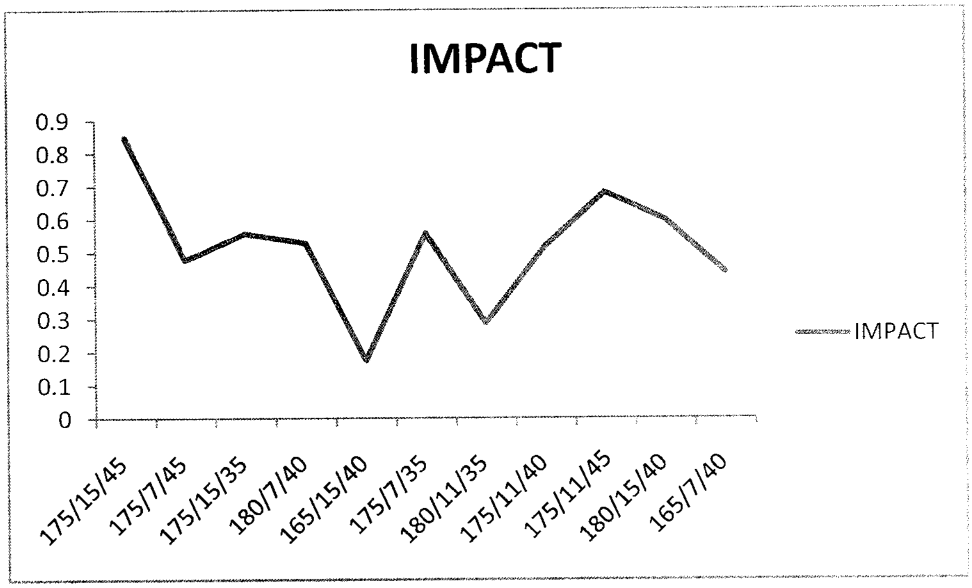


From the above graph the flexural strength of the 7th sample is higher and 11th sample is lower.

4.4 IMPACT TESTING

SPECIMEN NO	JOULES
1	0.849
2	0.478
3	0.558
4	0.529
5	0.175
6	0.558
7	0.286
8	0.518
9	0.680
10	0.598
11	0.439

Fig: 4.5 Impact values



From the above impact result we came to know that the sample 1st got the highest value and sample 5th got the lowest.

4.5 SEM

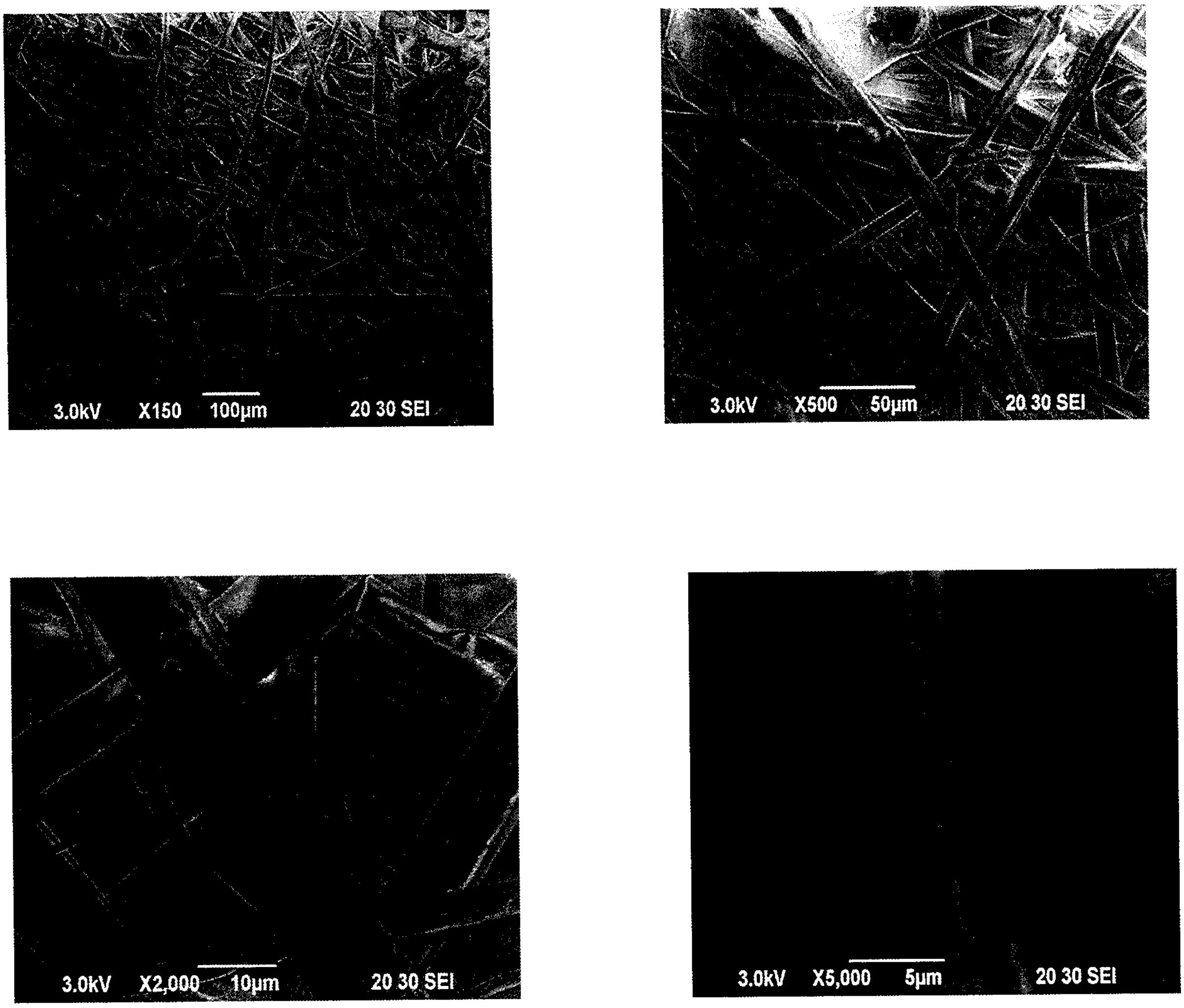


Fig: 31 SEM View

The above photo shows the clear view of the silk fibres. First fig: **31a is the 100µm of** the board which shows the clear **appearance and orientation of the silk fibres. The second fig:** 31b shows the 50 µm. Third fig: **31c is the 10µm of the fibres view. From the fourth fig:** 31d of 5µm shows the clear appearance **of the surface of a single fibre.**

CHAPTER V

CONCLUSION

- The three process parameters temperature, time and pressure in the Silk fibre reinforced polypropylene composites are optimised using BoxBehnhen design. The optimised process parameters are 175^0c, 7mins time and 35 Bar pressure.
- Composite boards are prepared at the optimised process conditions. The mechanical properties of the composites are 25.75 MPa for Tensile Strength, 320.51N/mm^2 for Flexural Strength, impact strength respectively.
- The water uptake for every 1hour shows the gradual increase in weight of the optimized silk reinforced polypropylene composite sample. The weight of the sample after 60mins is 17.391 the same sample is increased it weight up to 39.13 after 380mins.
- The random tests taken for water uptake on the optimised silk reinforced polypropylene sample with an interval of 24 hrs shows the gradual increase in weight. The weight of the sample after 24hrs is 43.65 the same sample is increased it weight up to 50 after 96hrs. After the 96hrs the weight of the sample reminds constant.
- Thermal conductivity of the silk reinforced polypropylene composites is 0.0005904

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