Effect of Wood Strength on the Properties of Wood Saw Dust Reinforced Polymer Matrix Composites

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October, 2011 BUET, DHAKA

Effect of Wood Strength on the Properties of Wood Saw Dust Reinforced Polymer Matrix Composites

A Thesis submitted to Materials & Metallurgical Engineering Department, Bangladesh University of Engineering and Technology (BUET), Dhaka, in partial fulfillment of the requirements for the degree of **Master of Philosophy** in Material science.

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October, 2011

The thesis titled "**Effect of Wood Strength on the Properties of Wood Saw Dust Reinforced Polymer Matrix Composites**" submitted by Md. Faruk Hossain, Student No. 0409113018, Session- April 2009, has been accepted as satisfactory in partial fulfillment of the requirements for the degree of Master of Philosophy in Material Science on 26 October, 2011.

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Md. Faruk Hossain

ACKNOWLEDGEMENTS

At first, the author expresses his gratefulness to the most Merciful Allah for his kind help in successfully completing the thesis work.

It gives me immense pleasure to express my deep sense of gratitude to my supervisor Prof. Dr. Md. Aminul Islam for his invaluable guidance, motivation, constant inspiration and above all for his ever co-operating attitude that enabled me in bringing up this thesis in the present form.

I express my sincere gratitude to Prof. Dr. Mohammad Ali Choudhury**,** Department of Electrical and Electronics Engineering for providing the necessary facilities in his laboratory.

I want to thanks to the authority of the Department of Materials & Metallurgical Engineering of BUET, for funding and giving the opportunity and accessibility to different labs.

I acknowledge with appreciation the co-operation of Md. Harun-or-Rashid, Mr. Ashiqur Rahman, Md. Ahmed Ullah, Abdur Rahim and Mr. Yusuf Khan for their help at various stages of my research work.

I am greatly thankful to other staff members of the department and all my well wishers and friends for their inspiration and help.

Finally, I would like to extend my appreciation and gratitude to my beloved parents and family members for their advices and continuous moral support.

Thanks to all. May Allah bless all of you.

Md. Faruk Hossain

Dhaka, 2011.

CERTIFICATE

This is to certify that the thesis entitled "**Effect of Wood Strength on the Properties of Wood Saw Dust Reinforced Polymer Matrix Composites**", submitted by ***Md. Faruk Hossain (Roll Number: 0409113018)*** in partial fulfillment of the requirements for the award of ***Master of Philosophy*** in the department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka is an authentic work carried out under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to elsewhere for the award of any degree.

Date: 26 October, 2011
BUET, Dhaka

Professor Dr. Md. Aminul Islam
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Dedicated to my

Parents and Well wishers

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Nomenclature

GRM	Garjan medium
GRC	Garjan coarse
GMM	Gamari medium
GMC	Gamari coarse
KRM	Kerosene medium
KRC	Kerosene coarse
TGRM	Treated Garjon medium
TGRC	Treated Garjon coarse
TGMM	Treated Gamari medium
TGMC	Treated Gamari coarse
TKRM	Treated Kerosene medium
TKRC	Treated Kerosene coarse
P (o)	Pure polyester
MMC	Metal Matrix composite
CMC	Ceramic Matrix Composite
PMC	Polymer Matrix Composite
TGA	Thermo-Gravimetric Analysis
FTIR	Fourier Transform Infrared Radiation
SEM	Scanning Electron Microscope

List of Notations

cm	Centimeter
cc	Centimeter cube
ρ	Density
ρ_T	True density
ρ_A	Apparent density
E_m	Elastic modulus of matrix
E_f	Elastic modulus of fiber
E_L	Longitudinal Elastic modulus
E_T	Transverse Elastic modulus
g	Gram
g/cc	Gram per centimeter cube
GPa	Giga Pascal
kg	Kilogram
KV	Kilovolt
L	Length
m	Meter
m^3	Meter cube
mm	Millimeter
MPa	Mega Pascal
Pa	Pascal
s	Second
V	Volume
V_f	Volume fraction of fiber
V_m	Volume fraction of matrix
vol. %	Volume percentage
W	Weight
Wt%	Weight percentage
^oC	Degree Celsius
%	Percentage
µm	Micrometer
σ_f	Fiber tensile stress
σ_m	Matrix tensile stress
σ_{fu}	Fiber ultimate tensile stress
Є	Strain
α	Alpha
β	Beta
±	Plus minus

Abstract

The use of wood dust for reinforcing polymer has already been considerably studied both from a scientific and a commercial point of view over the last decades. Because, these materials are attractive for their reduced environmental pollution and globally pleasant aesthetical properties. Wood dust is attractive filler for thermoset polymers, mainly because of it low cost, low density and high-specific properties. It is biodegradable and nonabrasive during processing. The present research work has been undertaken with an objective to explore the effects of wood strength, i.e. the types of woods were used to prepare the dust particle as reinforcing materials in polymer composites. In this regards, mechanical, electrical and thermal behaviours of the resulting composites reinforced with dust particle of various woods were investigated. For more detail study 0, 5 and 10 wt% were used to know the effects of proportions of wood particle on composite properties. Similarly, wood particle of two different sizes (medium and coarse) were used to know the effects of wood particle sizes on the properties of the developed composites. Initiative has also been taken to investigate the effects of chemical treatment (treatment with 10% NaOH solution) of wood particle on composite properties.

Among three woods Garjan, Kerosene and Gamari resulted, respectively, the highest, medium and the lowest tensile strengths. Experimental results revealed that saw dust particle of higher strength wood resulted relatively higher tensile strength of the developed composites. For all cases, tensile strengths of the developed composites were found to decrease with increase in the wood particle contents. Compared to medium size particle, coarse saw dust particle caused more deterioration in the tensile strengths. On the other hand, chemically treated wood particle resulted relatively higher strengths. With increase in the wood particle contents more wood particle stacking and air bubbles were found during the study of fracture surface under the SEM, which have been thought to be responsible for the reduced tensile strength of the composites.

For pure woods, wood strengths or densities have not been found to influence their breakdown voltages. In this case, size of the individual pore was found to influence the breakdown voltage. Similar to tensile strength, breakdown voltage was found to decrease with increase in the proportion of the wood particle. FTIR results suggested that chemical treatment removed part of the lignin of the wood particle. Increase in the breakdown voltage of the composites reinforced

with the chemically treated wood particle suggested that lignin has relatively poor breakdown voltage compared to that of the wood fiber.

Pure polyester has been found to have lower thermal conductivity compared to that of any wood and that wood strength or true density has been found to influence the thermal conductivity of the as-received pure very mildly. With increase in the dust particle content, thermal conductivities of all composites have been found to increase for both the untreated and chemically treated wood particle.

Thermo-Gravimetric Analysis (TGA) was used to know the decomposition of composites as a function of increasing temperature. TGA results revealed that wood particle have no significant influence on the thermal stability of the pure polyester up to 350^{o}C, however, the thermal stability of the composite increased slightly at further higher temperature. This high temperature thermal stability was again improved with higher proportion of wood particle. Chemical treatment improved the thermal stability of the composite. In this case improved thermal stability has been found from around 150^{o}C. This observation suggested that lignin has lower thermal stability compared to that of wood fiber.

Water absorption of both pure polyester and wood particle reinforced composites has been found to increase with increase in time. Either increase in the proportion of wood particle content in the composite or particle size of the wood dust increased the water absorption. Chemical treatment improved almost all properties. However, in the case of water absorption, it has been found to reduce the water absorption resistance of the composites, which indirectly suggest that lignin has better water absorption resistance than that of wood fiber.

Chapter 1

INTRODUCTION

1.1 INTRODUCTION

Composite is one of the most advanced and adaptable engineering materials known to men. Progresses in the field of materials science and technology have given birth to these fascinating and wonderful materials. Composites are heterogeneous in nature, created by the assembly of two or more components with fillers or reinforcing fibers and a compactable matrix. The matrix may be metallic, ceramic or polymeric in origin. It gives the composites their shape, surface appearance, environmental tolerance and overall durability while the fibrous reinforcement carries most of the structural loads thus giving macroscopic stiffness and strength [1].

Wood is a natural polymeric composite and consists primarily of cellulose, hemicellulose and lignin [2]. The cellulose fiber provides strength and the lignin is the "glue" that bonds and stabilizes the fiber. There is also a good potential for the use of natural fibers/particles as reinforcing fillers in polymeric materials. The utilization of ligno-cellulosic materials in the production of polymeric composites is attractive particularly because of low cost/high volume applications. Bio-degradable ligno-cellulosic fillers possess several advantages compared to inorganic fillers, such as lower density, greater deformability, lower abrasiveness and cost [3–12]. The main drawbacks of wood particles are their relative low degradation temperature and their hygroscopicities, which weaken their adhesion with hydrophobic polymers. However, wood fibers show very good mechanical properties such as tensile strength and Young’s moduli [13].

The past decade has seen fast and steady growth of wood plastics industry. Among many reasons for the commercial success, the low cost and reinforcing capacity of the wood fillers provide new opportunities to manufacture composite materials. The main application areas of wood flour filled composites are the automotive and building industries in which they are used in structural applications as fencing, decking, outdoor furniture, window parts, roofline products, door panels, etc. [14, 15]. The elastic modulus of wood fibers is approximately 40 times higher than that of polyethylene and the strength about 20 times higher [16]. The increased interest in the use of wood as filler and/or reinforcement in thermosets are due to the many advantages. Low density, high stiffness and strength, and low price are some of these advantages [17-19]. The environmental awareness of people today is forcing the industries to choose natural materials as substitutes for non-renewable materials. Wood has been used as building and engineering material since early times and offers the advantages of not just being aesthetically pleasing but also renewable, recyclable and biodegradable [20].

1.2 OUTLINES OF THE PRESENT STUDY

Three different woods such as Garjan (high strength), Kerosene (medium strength) and Gamari (low strength) have been bought from local market and saw dust has been made from BUET carpentry shop. Saw dust was screened to get particle of various finenesses, e.g. fine, medium and coarse. Fine particle has only been used to measure true density and medium and coarse particle have been used to prepare composites with unsaturated polyester matrix. 50% of the medium and coarse particle has been chemically treated and then composites have been made. For all cases composites were made by die casting method.

Optical microscope was used to find out the macro as well as the microstructure of these composites. Standard tensile samples have been made from the developed composites and they have been tested in the Universal tensile testing machine to know their mechanical behaviours. Tensile fracture surfaces were observed under the SEM to analyze the fracture morphologies, crack initiation sites, reason of lower strength, etc. Thermal conductivity of the developed composites has been measured by Sale’s method. In this study, breakdown voltage was also measured by high voltage testing machine (available in Electrical and Electronics Engineering Department). Fourier transform Infrared Spectroscopy (FTIR) have been used to find out the functional groups of the woods, polyester and their composites. Thermo-Gravimetric Analysis (TGA) has been used to evaluate the thermal degradation of these composite and finally water absorption test was also conducted to evaluate the physical degradation of these composites.

In the third chapter, experimental techniques are described that were used during the study. Initially, apparent and true density measurements are depicted. Next fabrication technique is described and finally several methods that were used to characterize the wood, pure polyester and composites that contents untreated and treated saw dust.

In the fourth chapter, the experimental results are discussed and analyzed (comparisons with similar studies will be given).

Finally, in chapter five general conclusions and future works are given based on the obtained results.

Chapter 2

LITERATURE REVIEW

2.1 COMPOSITES

Composite materials (or composites for short) are engineered materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure. The different materials work together to produce a new material, which combines all of the properties of the previously separate materials. Within the composite it is still possible to easily tell the different materials apart. They do not tend to blend or dissolve into each other. Nature made the first composites in living things. Wood is a composite of cellulose fibers held together with a glue or matrix of soft lignin. In engineering materials, composites are formed by coatings, internal additives and laminating [21].

Composites are made up of individual materials referred to as constituent materials. There are two categories of constituent materials, matrix and reinforcement. At least one portion of each type is required. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements impart their special mechanical and physical properties to enhance the matrix properties.

The reinforcing material and the matrix material can be metal, ceramic, or polymer. Composites are used because overall properties of the composites are superior to those of the individual components. For example, polymer/ceramic composites have a greater modulus than the polymer component, but aren't as brittle as ceramics. The following are some of the reasons why composites are selected for certain applications.

- ✓ High strength to weight ratio (low density high tensile strength)
- ✓ High creep resistance
- ✓ High tensile strength at elevated temperatures
- ✓ High toughness

Typically, reinforcing materials are strong with low densities while the matrix is usually a ductile, or tough, material. If the composite is designed and fabricated correctly, it combines the strength of the reinforcement with the toughness of the matrix to achieve a combination of desirable properties not available in any single conventional material. The downside is that such composites are often more expensive than conventional materials. Examples of some current application of composites include the brake-shoes and pads, tires and aircraft in which nearly 100% of the structural components are composites.

Two main components are existed in the composites. There are-

a. **Matrix -**metal, ceramic, polymer that surrounds the fiber.
b. **Reinforcing Fiber-** metal, ceramic, polymer and wood embedded in the composite.

2.1.1 Types of Composites on the Basis of Matrices

Three types composites are available according to origin are given below-

- Metal Matrix Composites (MMC)
- Ceramic Matrix Composites (CMC)
- Polymer Matrix Composites (PMC)

2.1.2 Metal Matrix Composites

A metal matrix composite (MMC) is composite material where, one component is a metal and other may be a different metal or another material, such as a ceramic or organic compound. It can say that metal matrix composite (MMC) is a material consisting of a metallic matrix combined with a ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) dispersed phase.

MMCs are made by dispersing a reinforcing material into a metal matrix. The reinforcement surface can be coated to prevent a chemical reaction with the matrix. For example, carbon fibers are commonly used in aluminium matrix to synthesize composites showing low density and high strength. However, carbon reacts with aluminium to generate a brittle and water-soluble compound Al_4C_3 on the surface of the fiber. To prevent this reaction, the carbon fibers are coated with nickel or titanium boride [69].

Several properties of Metal Matrix Composites (MMC) are existed. These properties increase the uses of MMCs which are pointed below-

- High strength even at elevated temperatures

- High stiffness (modulus of elasticity)
- High thermal conductivity
- Excellent abrasion resistance
- Good creep resistance
- High wear resistance
- High thermal stability
- Low coefficient of thermal expansion

MMC is used in different categories of the works. Several of them are presented below [22-24]

- Carbide drills are often made from a tough cobalt matrix with hard tungsten carbide particles inside.
- Some tank armors may be made from metal matrix composites, probably steel reinforced with boron nitride. Boron nitride is a good reinforcement for steel because it is very stiff and it does not dissolve in molten steel.
- Modern high-performance sport cars, such as those built by Porsche, use rotors made of carbon fiber within a silicon carbide matrix because of its high specific heat and thermal conductivity.
- Aluminium metal matrix composite cylinder liners in some of their engines.
- Specialized bicycles have used aluminum MMC compounds for its top of the range bicycle frames for several years.
- Aluminum matrix composites (AMC) are used for manufacturing automotive parts (pistons, brake components), brake rotors for high speed trains, bicycles, golf clubs, electronic substrates, cores for high voltage electrical cables.
- Magnesium matrix composites are used for manufacturing components for racing cars, lightweight automotive brake system, and aircraft parts for gearboxes, transmissions, compressors and engine.
- Titanium matrix composites are used for manufacturing structural components of the F-16 jet’s landing gear, turbine engine components (fan blades, actuator pistons, synchronization rings, connecting links, shafts and discs), automotive engine components, drive train parts, general machine components.
- Copper matrix composites are used for manufacturing hybrids modules, electronic relays, electrically conducting springs and other electrical and electronic components.

- Some equipment in particle accelerators such as radio frequency quadrupoles or electron targets uses copper MMC compounds such as Glidcop to retain the material properties of copper at high temperatures and radiation levels.

2.1.3 Ceramic Matrix Composites (CMC)

Ceramic Matrix Composites is the matrix made of a ceramic material embedded between the fibres. Typical fibrous materials include carbon, silicon carbide, aluminium oxide and mullite. Aluminium oxide, zirconium oxide and silicon carbide are chiefly used as matrix components.

Ceramic Matrix Composite (CMC) is a material consisting of a ceramic matrix combined with a ceramic (oxides, carbides) dispersed phase. This composite have had a stormy development wherever the combination of breakage resistance and strength of conventional technical ceramics such as aluminium oxide, silicon carbide or zirconium oxide prove inadequate. Because even small production errors or scratches on the surface of conventional ceramics can lead to cracks forming, their use in many applications had always been impossible. Typical applications are brake disks in the automotive industry, emergency rail systems for the transrapid, emergency running systems for rapidly rotating machines or friction pads for clutches [70].

Typical properties of Ceramic Matrix Composites are focused briefly.

- High mechanical strength even at high temperatures
- High thermal shock resistance
- High stiffness
- High toughness
- Low density
- High corrosion resistance even at high temperatures

CMCs have different applications considering their properties, some of them are described below [25, 26].

- Silicon carbide matrix composites are used for manufacturing combustion liners of gas turbine engines, hot gas re-circulating fans, heat exchangers, rocket propulsion components, filters for hot liquids, gas-fired burner parts, furnace pipe hangers, immersion burner tubes.

- Alumina and alumina-silica (mullite) matrix composites are used for manufacturing heat exchangers, filters for hot liquids, thermo-photovoltaic burners, burner stabilizers, combustion liners of gas turbine engines.
- Carbon-Carbon Composites are used for manufacturing high performance braking systems, refractory components, hot-pressed dies, heating elements, turbojet engine components.
- A combustor made of SiC with a special SiC fiber of enhanced high-temperature stability was successfully tested for 15,000 hours. SiC oxidation was slowed down heavily by the use of an oxidation protection coating of several layers of oxides.

2.1.4 Polymer Matrix Composites (PMC)

A polymer matrix composite (PMC) as Fig. 2.1 is a composite material consisting of a polymer imbedded with a reinforcing phase such as fibers or powders. By this definition an alloy or blend could be a composite, but the accepted use of polymer is to mean a material with a continuous resin matrix and a controlled distribution of a reinforcing material. Even with this definition some polymer materials, such as liquid crystal polymers, meet the definition, but they are not normally considered to be composites. From the commercial standpoint, composites are made from matrices of epoxy, unsaturated polyester and some other thermosets, and a few thermoplastics [27].

The importance of PMC processes derive from the growing use of this class of material, especially fiber-reinforced polymers (FRPs),

- FRP composites can be designed with very high strength-to-weight and modulus-to-weight ratios.
- These features make them attractive in aircraft, cars, trucks, boats, and sports equipment.

Figure 2.1: Polymer matrix composite [76].

Critical functions of the polymer matrices are given below-

- Bind the fibers together

- Protect the fibers from environment and abrasion
- Separate and disperse the fibers throughout the composite
- Transfer force between the individual fibers

2.1.5 Polymer

The engineering materials known as plastics are more correctly called polymers. This term comes from Greek words poly, which means “many” and meros means “part”. Polymers are substances composed of long-chain repeating molecules (mers). In the most cases the element carbon forms the backbone of the chain (an organic material). The atoms in the repeating molecule are strongly bonded (usually covalent), and the bonds between molecules may be due to weaker secondary bonds or similar covalent bonds [71].

Two types of polymer

a. Thermoplastics
b. Thermoset polymers

2.1.6 Thermoplastic polymer

A thermoplastic polymer is one which melts or flows when heated. Thermoplastic polymers are usually not highly crosslinked, and act much like molecular solids, low melting and boiling points, low strength, ductile. Examples include polyethylene and polypropylene, nylon, polyamide etc [28]. Properties of thermoplastics as a matrix are given briefly-

- Extremely strong bonds within chain molecules
- Chains held by weak Vander Waals Forces
- Molecules can slide past one-another on heating
- Polymer can be reversibly softened

2.1.7 Thermoplastic polymer matrices

Thermoplastic resins have received considerable attention in the past few years as the matrix material in organic resin-based composites. Whereas thermosetting materials date back 5000 years, when Egyptians used a straw-reinforcing agent in a clay matrix to form bricks, thermoplastic composites are relatively recent. Although their use in advanced composites is not widespread, thermoplastic composites are used extensively in commercial applications ranging from automobiles to durable goods.

Thermoplastic polymers are usually linear molecules with no chemical linkage between the molecules. The molecules are held together by weak secondary forces, such as vander Waals or hydrogen bonding, and as such are readily deformed by the application of heat or pressure. Thermoplastic resins can be amorphous, that is, structure less, or semicrystalline, in which some of the molecules form an ordered array. A material is usually considered semicrystalline if as little as 5% of the polymer is in the crystalline form. Semicrystalline resins exhibit a higher modulus, but amorphous materials are tougher and are usually more solvent sensitive but can be processed at lower temperatures.

Although in a broad sense, a composite is a material that contains two or more distinct phases, this discussion is restricted to those materials in which one of the phases, usually the fibers, serves as a reinforcing or strengthening agent (see Composite Materials, survey). Materials in which a relatively inexpensive material, such as talc or calcium carbonate, is added to reduce the overall cost of the expensive thermoplastic polymer are not included. Advanced or high performance composites contain more than 50 wt % fibers, whereas other composites or commodity composites (reinforced plastics) contain 20–40 wt % reinforcing agent. The distinction is real, and different resin properties, hence different chemistries, are required for these materials. The different types of thermoplastic polymers matrices used in composites are Acrylic, Poly (methyl methacrylate) (PMMA), Polyethers, Polycarbonates, Polyamide, Polyethylene, Polyvinyl chloride, Teflon [28].

Many advantages of thermoplastic polymers are found, some of them are given below-

- Less expensive due to fast cycle times
- More complex designs are possible
- Wider range of properties due to copolymerization
- Recyclable since they can be molded, extruded and reused
- No need to add reinforcing agents, stabilizers or cure systems
- Less energy and closer and more economical control of product quality are possible

Some disadvantages of thermoplastic polymers are considered. In this context, they are used in limited services. These disadvantages are described below-

- Higher creep
- Poor chemical and heat resistance

- High compression set and
- Low thermal stability

2.1.8 Thermoset polymer

A thermoset polymer is one that doesn't melt when heated. Thermoset polymers "set" into a given shape when first made and afterwards do not flow or melt, but rather decompose upon heating. They are often highly cross linked polymers, with properties similar to those of network covalent solids- i.e., hard, strong and brittle. Examples include bakelite, formica and the various super glues etc [27].

2.1.9 Thermoset Polymer Matrices

Thermoset polymer matrices are usually formed form low-viscosity liquids that become cross-linked by combination with a catalyst or by the application of some external form of energy, such as heat or radiation etc. These are synthetic polymer reinforcements that have been developed and used in many applications, including glass–reinforced polyester radar domes on aircraft and graphite-epoxy doors on the space shuttle. Thermoset polymers are the most widely used matrix material in composite materials. The different types of thermoset polymer matrices used in composites are: Bis-Maleimides (BMI), Epoxy (Epoxide), Phenolic (PF), Polyester (UP), Polyimide, Polyurethane (PUR), Silicone [27].

Advantages of thermoset polymers are given below-

- Well established processing and application history
- Overall, better economics than thermoplastic polymers
- Better high temperature properties
- Good wetting and adhesion to reinforcement

Disadvantages of thermoset polymers compared to thermoplastics are presented below-

- Resins and composite materials must be refrigerated
- Long process cycles
- Reduced impact –toughness
- Poor recycling capabilities
- More difficult repair ability

2.1.10 Thermoset and Thermoplastic Polymer Matrix Composites

Comparisons between thermoset and thermoplastic composites are given below [71]

Properties	t/set	t/plastic
Young's Modulus (GPa)	1.3-6.0	1.0-4.8
Tensile strength (MPa)	20-180	40-190
Max service temp (ºC)	50-450	25-230
Fracture toughness, ($MPa^{1/2}$)	0.5-1.0	1.5-6.0

2.2 TYPES OF COMPOSITES ACCORDING TO REINFORCING

According to reinforcing, three types of composites are given below-

- Particle reinforced composites
- Fiber reinforced composites
- Structural composites

2.2.1 Particle Reinforced Composites

Particles used for reinforcing include ceramics and glasses such as small mineral particles, metal particles such as aluminum, and amorphous materials, including polymers and carbon black as shown in Fig. 2.2. Particles are used to increase the modulus of the matrix and to decrease the permeability of the matrix. These are also used to decrease the ductility of the matrix and to produce inexpensive composites. Reinforcers and matrices can be inexpensive materials and are easily processed.

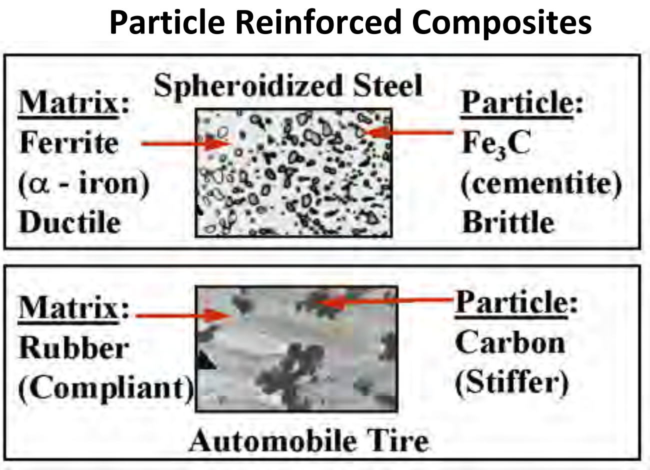


Figure 2.2: Particle-reinforced composites [72].

An example of particle reinforced composites is an automobile tire which has carbon black particles in a matrix of polyisobutylene elastomeric polymer. Another example is spheroidized steel where cementite is transformed into a spherical shape which improves the machinability of the material. Another example for particle-reinforced composite is concrete where the aggregates (sand and gravel) are the particles and cement is the matrix. Particle reinforced composites support higher tensile, compressive and shear stresses [72].

2.2.2 Fiber Reinforced Composites

Reinforcing fibers can be made of metals, ceramics, glasses, or polymers that have been turned into graphite and known as carbon fibers. Fibers increase the modulus of the matrix material. The strong covalent bond along the fiber's length gives them a very high modulus in this direction because of breaking or extending the fiber, the bonds must also be broken or moved. Fibers are difficult to process into composites which makes fiber-reinforced composites relatively expensive. Fiber-reinforced composites (as shown Fig. 2.3) are used in some of the most advanced and therefore most expensive, sports equipment, such as a time-trial racing bicycle frame which consists of carbon fibers in a thermoset polymer matrix. Body parts of race cars and some automobiles are composites made of glass fibers (or fiberglass) in a thermoset matrix**.**

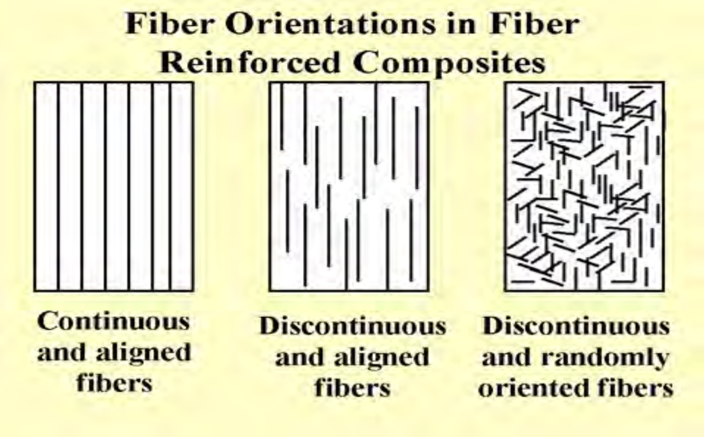


Figure 2.3: Fiber orientation in fiber reinforced composites [72].

The arrangement or orientation of the fibers relative to one another, the fiber concentration and the distribution, all have a significant influence on the strength and other properties of fiber-reinforced composites. Applications involving totally multidirectional applied stresses normally use discontinuous fibers, which are randomly oriented in the matrix material. Consideration of orientation and fiber length for particular composites depends on the level and nature of the applied stress as well as fabrication cost. Production rates for short-fiber composites (both aligned and randomly oriented) are rapid, and intricate shapes can be formed which are not possible with continuous fiber reinforcement [72].

2.2.3 Modulus of Fiber Reinforced Composites

Fibers have a very high modulus along their axis but have a low modulus perpendicular to their axis. If the fibers are all parallel, the modulus of a fiber reinforced composite depends upon which direction of measurement. The modulus of the entire composite (Fig. 2.4), matrix plus reinforcer, is governed by the rule of mixtures when measuring along the length of the fiber:

$E_c = E_f V_f + E_m V_m$

E_c is the modulus of the entire composite along the length of the fiber.

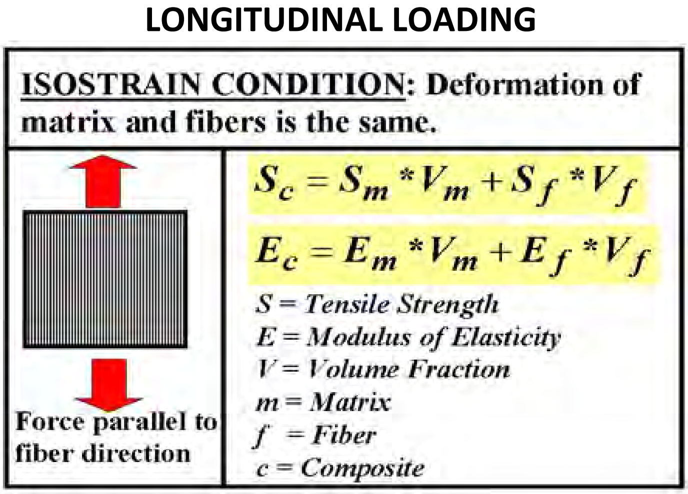
E_f is the modulus of the fiber along the length of the fiber.

V_f is the volume percent occupied by the fibers.

E_m is the modulus of the matrix (usually not dependent upon direction)

V_m is the volume percent occupied by the matrix (equal to $(1-V_f)$).

Fiber composite manufacturers often rotate layers of fibers to avoid directional variations in the modulus.



$$S_c = S_m * V_m + S_f * V_f$$

$$E_c = E_m * V_m + E_f * V_f$$

S = Tensile Strength
E = Modulus of Elasticity
V = Volume Fraction
m = Matrix
f = Fiber
c = Composite

Figure 2.4: Tensile strength and elastic modulus when fibers are parallel to the direction of stress [72].

Tensile strength and elastic modulus of composite is shown in Fig. 2.5, when fibers are perpendicular to the direction of stress.

TRANSVERSE LOADING

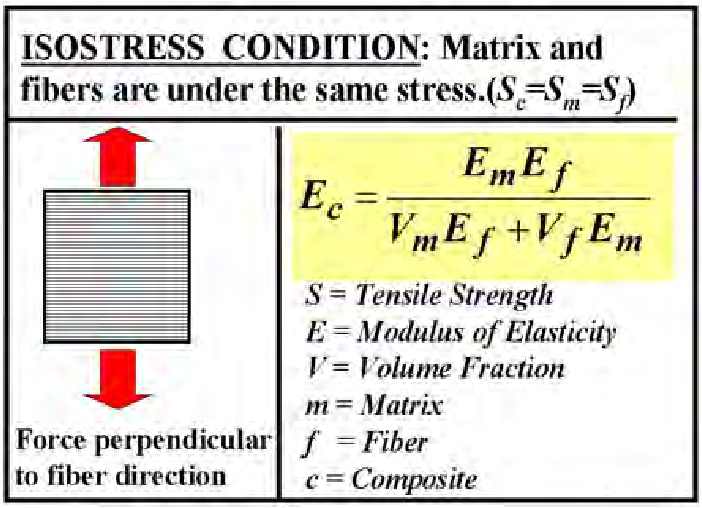


Figure 2.5: Tensile strength and elastic modulus when fibers are perpendicular to the direction of stress [72].

2.2.4 Structural Composites

Common structural composites are laminar and sandwich panel are seen in Fig. 2.6.

Laminar: It is composed of two-dimensional sheets or panels that have a preferred high strength direction such as found in wood or continuous and aligned fiber-reinforced plastics. The layers are stacked and cemented together such that the orientation of the high-strength direction varies with each successive layer. Example of a relatively complex structure is plywood [72].

Sandwich Panels: It consists of two strong outer sheets which are called face sheets and may be made of aluminum alloys, fiber reinforced plastics, titanium alloys, steel. Face sheets carry most of the loading and stresses. Core may be a honeycomb structure which has less density than the face sheets and resists perpendicular stresses and provides shear rigidity. Sandwich panels can be used in variety of applications which include roofs, floors and walls of buildings and in aircraft for wings [72].

The properties of structural composites depend on:

- ✓ Constituents
- ✓ Geometrical design

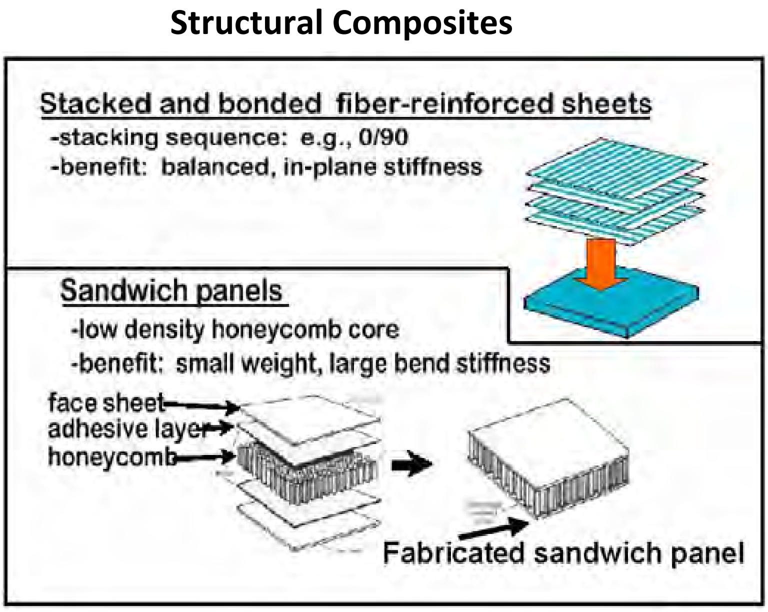


Figure 2.6: Structural composites [72].

2.2.5 Reinforcement of Fiber

When combining highly performing fibers with polymers make a composite and this composite gets improved mechanical properties. The stress applied from an external source to the composite is transferred to the fibers (which are the load-bearing element) through the fiber–matrix interface, and the mechanism of this stress transfer and the ensuing reinforcement effect may be visualized. The situation is different when fibers are long or short.

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. All of the different fibres used in composites have different properties and so affect the properties of the composite in different ways.

However, individual fiber or fiber bundles can only be used on their own in a few processes such as filament winding. For most other applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fibers orientations possible and lead to different types of fabrics, each of which has its own characteristics [72].

2.2.6 Long Fibers

The composites used in the most advanced applications (in aerospace, for example) normally include long (or continuous) fibers from which laminated structures are formed. When the fibers are more rigid than the matrix (which is the case in practically important composites), assuming a perfectly bonded interface between fiber and matrix, and loading parallel to the fiber array, the transfer of stress from the applied load to the fibers is achieved from the ends of the specimen through the loading mechanism, resulting in a fairly uniform stress field in the composite [72].

$$E_{\mathrm{T},} = E_{\mathrm{f}} V_{\mathrm{f}} + E_{\mathrm{m}} V_{\mathrm{m}} \qquad (1)$$

Thus, a simple rule of mixtures emerges for the longitudinal Young's modulus of the composite, where the subscripts f and m designate the fiber and the matrix, and *E* and *V* are the Young's modulus and fiber volume fraction respectively.

Equation **(1)** usually provides a good fit to experiment. For the transverse Young's modulus of the composites, the often quoted inverted rule of mixtures

$$1/E_{\mathrm{T}} = (V_{\mathrm{f}}/E_{\mathrm{f}}) + (V_{\mathrm{m}}/E_{\mathrm{m}}) \qquad (2)$$

2.2.7 Short Fibers

Stress transfer in discontinuous fiber composites which include the cases of either short or broken fibers and this is more complex than in continuous fiber composites. In this case, the shear and tensile stress and strain fields will not be uniform. If both the matrix and the fiber are elastic, the tensile stress profile in the fiber is uniform.

In fact, the measured shear stresses in the matrix at the ends of fibers are much larger than those given by a simple force balance model because of stress concentration effects. Therefore, the maximum shear stress will be the smaller of the following two stresses, the shear yield stress of the matrix or the shear strength of the fiber–matrix interface. Whichever of these two stresses is attained first will control the load transfer phenomenon [30, 72].

2.3 TYPES OF WOOD

Wood is an organic material and a natural composite of cellulose fibers (which are strong in tension) embedded in a matrix of lignin which resists compression [31, 32]. It is a heterogeneous, hygroscopic, cellular and anisotropic material. The walls of wood cells consist of various layers, which differ in structure and chemical composition [31]. The individual cells of wood tissue are glued together in the middle lamella, which consists mainly of cellulose (40% – 50%) and hemicelluloses (15% – 25%) impregnated with lignin (15% – 30%).

Figure 2.7: Transmission electron micrograph of a vascular cell (softwood tracheids, hardwood libriform fibers). Middle lamella (ML), primary wall (P), S1, secondary wall 1 (S1), secondary wall 2 (S2), tertiary wall (T) and warty layer (W) [73].

The middle lamella and primary wall are often called the ‘compound middle lamella (Fig. 2.7). The next layer is the secondary wall, which is subdivided into secondary walls 1 (S1) and 2 (S2). S1 and S2 contain densely packed cellulose fibrils arranged in parallel, which differ in the angle at which the fibrils run in the S1, the fibrils run at a wide angle in relation to the fiber axis and in the S2, at a small angle. The S2 is the thickest wall layer and accounts for 50% (vessels, parenchyma cells) to 90% (tracheids, libriform fibres) of the whole cell wall. At the inner border of the cell wall, there is a final thin layer called the tertiary wall (T), in which the cellulose fibrils run at an angle similar to that of the S1. In some species, the tertiary walls of tracheids, fibres and vessels are covered with a wart-bearing amorphous layer (Fig.2.7). The lignin content decreases from the compound middle lamella through the S2, while the cellulose content increases in the same direction. The hemicelluloses content is highest in the S1, but because of the thickness of the S2, most lignin and celluloses are deposited in this layer [33]. The terms ‘hardwood’ and ‘softwood’ refer to the species of tree and not necessarily to the hardness of the wood. Hardwoods are generally denser than softwoods [73].

2.3.1 Softwood

The term softwood is used to describe wood from conifers in Fig. 2.8. It may also be used to describe these trees, which tend to be evergreen, notable exceptions being bald cypress and the larches [73].

Figure 2.8: A typical and well-known softwood (Pinus sylvestris) [74].

2.3.2 Hardwood

Hardwood is wood from angiosperm tree (more strictly speaking non-monocot angiosperm trees) is shown in Fig. 2.9. It may also be used for those trees themselves, these are usually broad-leaved; in temperate and boreal latitudes they are mostly deciduous, but in tropics and subtropics mostly evergreen [75].

Figure 2.9: A typical hardwood (angiosperm trees) [74].

Table 2.1: Comparison of softwood and hardwood characteristics [73]

Characteristics	Softwood	Hardwood
Cellulose	~40% to 50%	~40% to 50%
Hemicellulose	~15% to 30%	~25% to 35%
Lignin	~25% to 35%	~20% to 30%
Fibers	long (1.4–4.4 mm)	short (0.2–2.4 mm)
Polar	low	high

2.4 MATERIALS USED IN PRESENT STUDY

In this research work, polyester and wood dust have been used to fabricate composites. Polyester has been used as a matrix that surrounds the reinforcers and wood dust has been used as reinforcers that embedded into matrix.

2.4.1 Polyester

Polyester resins are thermosetting plastic. It is cured by hardener exothermically. The use of excessive catalyst can cause charring or even ignition during the curing process. Excessive catalyst may also cause the product to fracture or form a rubbery material. Polyesters are condensation polymers formed by the reaction of polyols (also known as polyhydric alcohols), organic compounds with multiple alcohol or hydroxy functional groups, with saturated or unsaturated dibasic acids. Typical polyols used are glycols such as ethylene glycol, acids used are phthalic acid and maleic acid. Water that is a by-product of esterification reactions is continuously removed, driving the reaction to completion. Depending on the chemical structure polyester can be a thermoplastic or thermoset, however the most common polyester synthesis of polyesters is generally achieved by a polycondensation reaction [71].

Polyesters are made from two types of monomers-

a. Di-acids
b. Glycols

The reaction between di-acids & glycols is given below-

$(n+1)\ R(OH)_2 + n\ R'(COOH)_2 \text{ ---> } HO[ROOCR'COO]_n ROH + 2n\ H_2O$

Curing Process of composites is considered following things-

The initially liquid resin is converted to a solid by cross-linking chains. This is done by creating free radicals at unsaturated bonds, which propagate in a chain reaction to other unsaturated bonds in adjacent molecules, linking them in the process. The initial free radicals are induced by adding a compound that easily decomposes into free radicals. This compound is usually and incorrectly known as the catalyst. Substances used are generally organic peroxides such as benzoyl peroxide, methyl ethyl peroxide ketone.

Properties of Polyester are given below-

This is most common type of resin for composites. And also least expensive and the easiest-to-cure composite resin and also time saving.

2.4.2 Wood Dust

Wood dust is a complex mixture. Its chemical composition depends on the species of tree and consists mainly of cellulose, polyoses, and lignin, with a large and variable number of substances with lower relative molecular mass. Cellulose is the major component of both softwood and hardwood. Polyoses (hemicelluloses) are present in larger amounts in hardwood than in softwood. They contain five neutral sugar units: hexoses, glucose, mannose, galactose, and the pentoses (xylose and arabinose). The lignin content of softwood is higher than that of hardwood. The monomers of lignin are phenylpropane units joined by various linkages. The lower-molecular-mass substances significantly affect the properties of wood; these include substances extracted with nonpolar organic solvents (fatty acids, resin acids, waxes, alcohols, terpenes, sterols, steryl esters, and glycerols), substances extracted with polar organic solvents (tannins, flavonoids, quinones, and lignans), and water-soluble substances (carbohydrates, alkaloids, proteins, and inorganic material). Hardwood tends to have a higher percentage of polar-soluble substances than softwood [34-37].

2.4.3 Physico-Chemical Properties of Wood Dust

Wood dust (as Fig. 2.10) is a light brown or tan fibrous powder. The morphology of softwood tissue is simpler than that of hardwood. The bulk of softwood consists of just one type of cell, tracheids. Tracheids are elongated fiber-like cells with a square or polygonal cross-section. Less than 10% of the wood consists of short, brick-like parenchymal cells arranged radially. Softwoods contain epithelial cells that secrete resin into canals, which run horizontally and radially through the wood. In hardwoods, there is more detailed differentiation between stabilizing, conducting, and storage tissue. Stabilizing tissues contain libriform fibers and fiber tracheids, which are elongated cells with thick polygonal walls and small lumina. The conducting system is composed of vessel elements fitted together to form long tubes of up to several meters. The vessels have thin walls and large diameters. Hardwoods that contain resin canals also have a secretory system of epithelial cells. Certain characteristics of softwood and hardwood are compared. The walls of wood cells consist of various layers, which differ in structure and chemical composition. The individual cells of wood tissue are glued together in the middle lamella (which consists mainly of lignin, polyoses, and pectins). Often, there is no precise visible border between the pure middle lamella and the outer cell wall layer (the primary wall), which is formed by a net-like arrangement of cellulose fibrils embedded in a matrix of lignin and polyoses. The middle lamella and primary walls are often called the “compounded middle lamella” [38-42].

Figure 2.10: Wood dust [75].

2.4.4 Application of Wood Dust

In our country, in general, wood saw dust is used as a source of fuel or, it is dumped as unwanted materials. Wood dust has limited commercial uses, but wood is one of the world’s most important renewable resources. It is estimated that forests cover more than one-third of the world’s total land area, with a total biomass of one trillion cubic meters, of which around 3.5 billion cubic meters are harvested annually. “Industrial round wood” refers to categories of wood not used for fuel, which include sawn wood (54%), pulpwood (21%), poles, pit props (14%), and wood used for other purposes, such as particle board and fiberboard (11%). Wood dust is used to prepare charcoal, as an absorbent for nitroglycerin, as filler in plastics, and in linoleum and paperboard. Another commercial use for wood dust is in wood composts [43, 44].

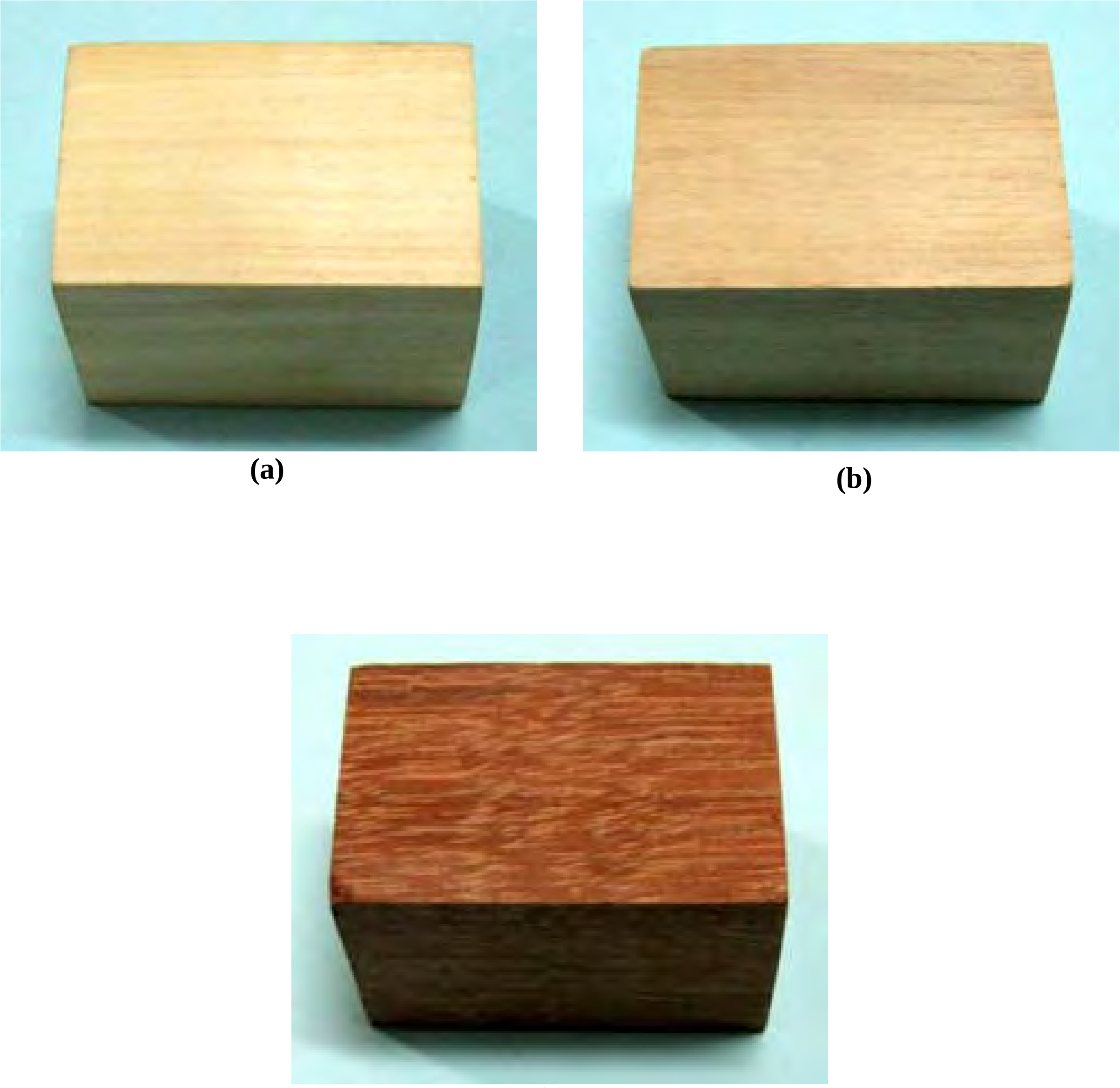
Chapter 3

EXPERIMENTAL PROCEDURE

3.1 MATERIALS PREPERATION

3.1.1 Natural Filler Preparation

Three different types of woods of various tensile strengths i.e. Gamari, Garjon and Kerosene were purchased from the local market. Several pieces of rectangular shaped test pieces (Fig. 3.1) were cut from each type of wood stock for apparent density measurement.



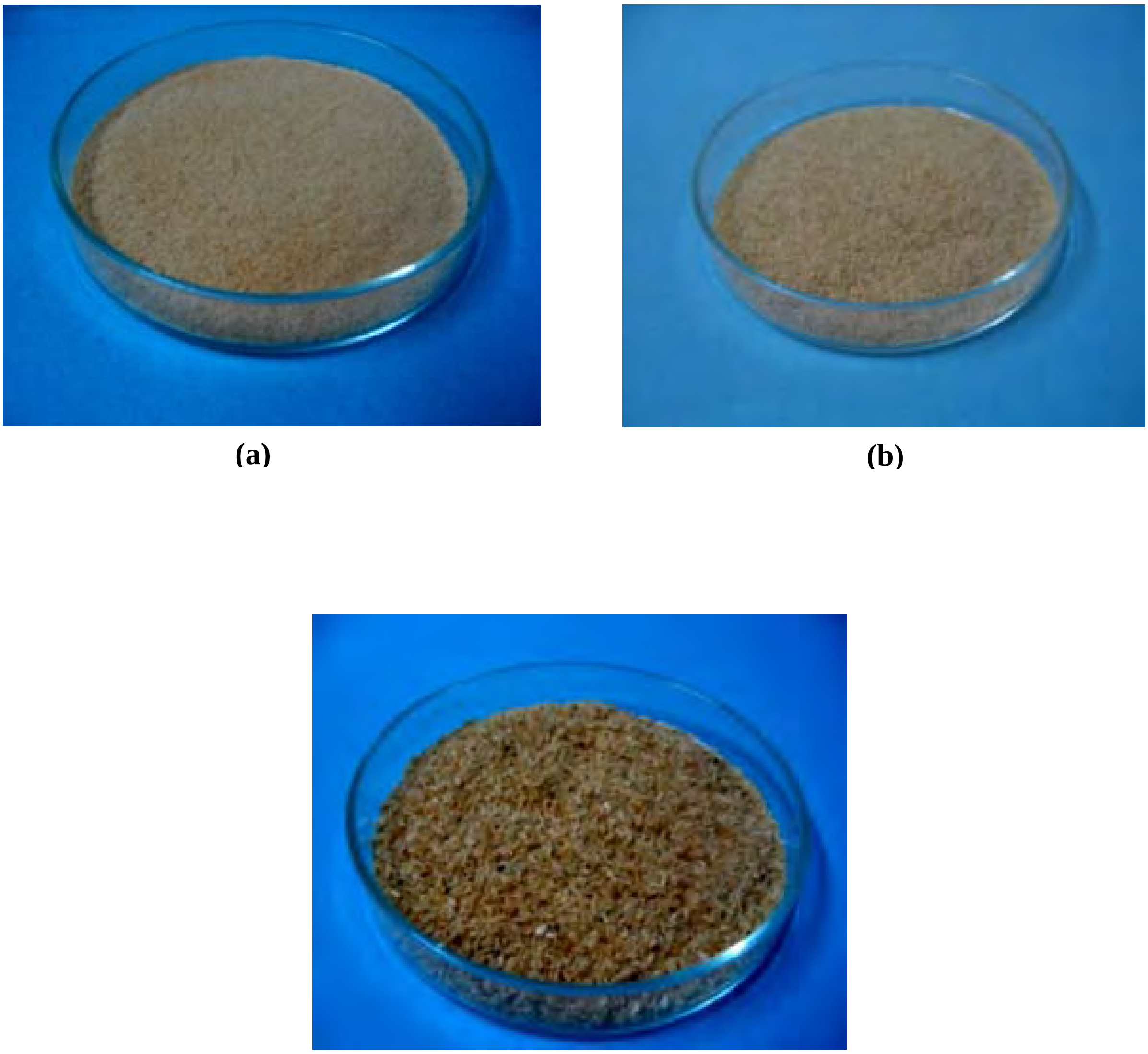
(a) (b) (c)

Figure 3.1: Rectangular blocks made from three different woods (a) Kerosene, (b) Gamari and (c) Garjan.

For getting sufficient amount of wood saw dust, part of the stock wood was also randomly sliced by power saw. The saw dust thus produced was screened to get particles of various finenesses, e.g. fine, medium and coarse sized particles by sieve. In Table 3.1, the sizes of wood saw dust particles have been shown and their photographs are shown in Figs. 3.2-3.4.

Table 3.1: Sizes of wood dust particles of various finenesses.

No. of Particles	**Name of the Particles**	**Particles Size (μm)**
1	Fine particles	0 - 250
2	Medium particles	250 - 500
3	Coarse particles	> 500

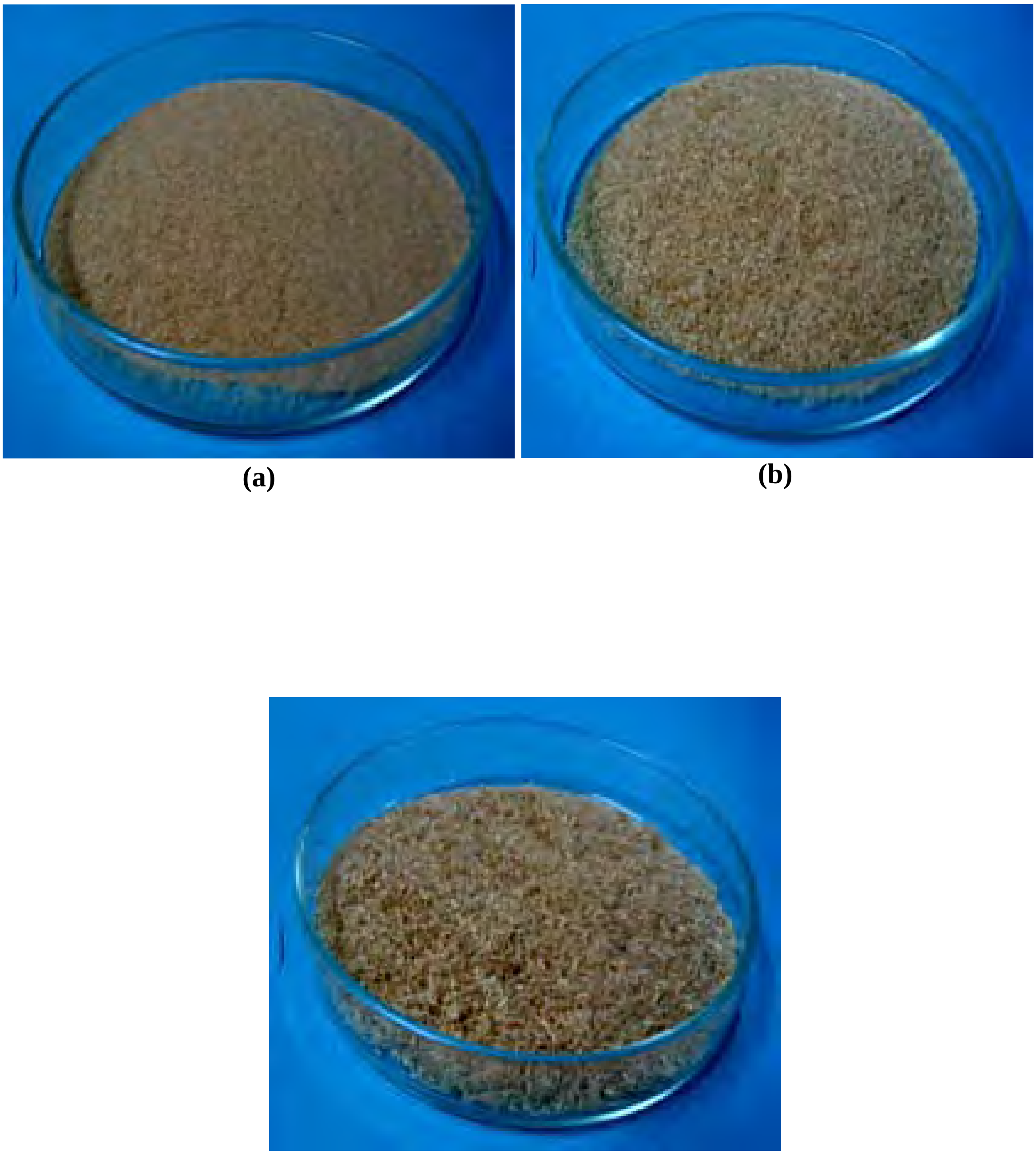


(a)

(b)

(c)

Figure 3.2: (a) Fine, (b) medium and (c) coarse particle of Kerosene wood before treatment with NaOH solution.

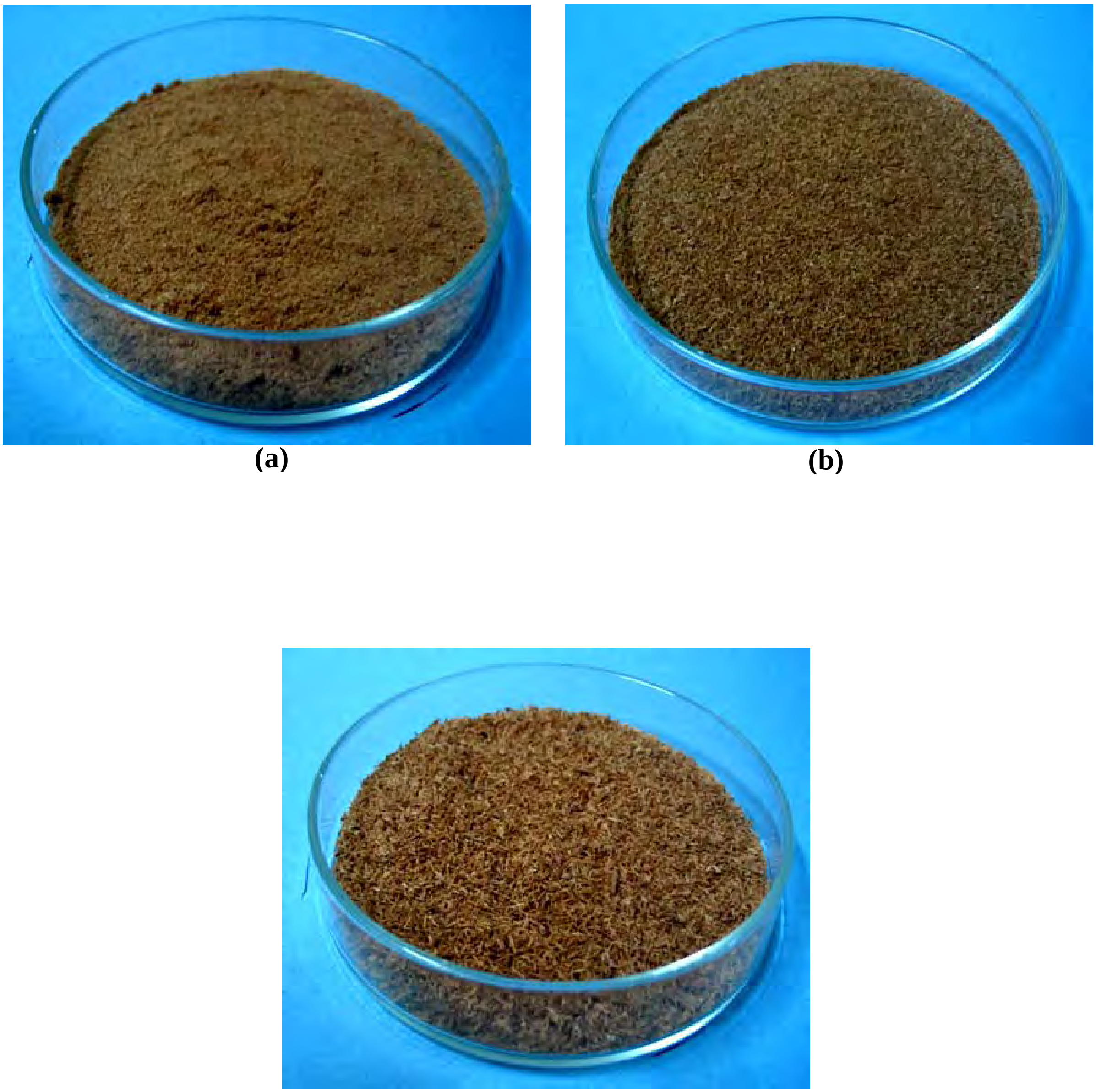


(a)

(b)

(c)

Figure 3.3: (a) Fine, (b) medium and (c) coarse particle of Gamari wood before treatment with NaOH solution.



(a) **(b)**

(c)

Figure 3.4: (a) Fine, (b) medium and (c) coarse particle of Garjan wood before treatment with NaOH solution.

It is important to mention that the amount of fine size wood particle was not sufficient for making composite. As a result, only medium and coarse particles were used for making composites. Fine dust particle was used to measure the true densities of the used wood. In order to know the effect of surface treatment on the composite properties, both the medium and coarse particles of the different woods were chemically treated separately with 10%

NaOH solution. Initially NaOH was taken into a conical flask and then distilled water added so that total weight of water was equivalent to 90% of the total solution. For proper chemical treatment, particle equivalent to only 5% (by wt) of the total volume of solution was added. The wood particle was kept into the solution for about one and half hour at room temperature. Afterwards the wood particle was separated from the NaOH solution, washed with sufficient amount tap water several times and then finally, it was washed with distilled water. The treated and washed wood particle was dried for five hours in an oven at 110 ^{o}C.

3.2 MATERIAL PREPERATION FOR COMPOUNDING AND MOLDING

Matrix material i.e. polyester was purchased from market. Liquid thermoset type polyester was selected as matrix material for this study. In order to know the effect of saw dust addition into the polyester, all types of test samples were prepared from pure polyester and wood saw dust particles reinforced composite materials. Usually air/gas remains dissolved into the liquid polyester. If this air/gas remains in the solid polyester, it makes various types of defects such as gas pockets, bubbles, microcraks etc. These defects deteriorate the properties of the cast samples. So, before casting the liquid polyester into a dies, it was made gas/air free. For doing this, required amount of polyester was taken into a beaker. The beaker with liquid polyester was put in a vacuum chamber. During vacuum period dissolved gases came on the top of the liquid polyester in the form of lots of bubbles and vanished. When it seemed nearly bubbles free then the beaker was taken out from the vacuum chamber. For each time of vacuum process, it took about 10 minutes.

Then hardener (methylethyle keton peroxide) of required proportion was also added inside the gas free liquid polyester and stirred properly for a uniform mixture of liquid polyester and the hardener. During this stirring air also dissolved inside the mixture. So, the mixture was again evacuated putting the beaker inside the vacuum chamber. Due to addition of hardener, the mixture formed pasty mass within 2-3 minutes. At this moment, the beaker was taken from the chamber and the pasty mass was cast into die of required shape.

In the case of wood dust particle reinforced composite casting, wood dust particle and liquid polyester of required proportion was taken into a beaker and the mixture was stirred properly to get uniform distribution of the reinforcing particle. The well-mixed polyester with wood particle was taken into the vacuum chamber and made gas/air free following procedure that mentioned earlier. After making gas free the beaker was taken out of vacuum chamber, required proportion of hardener was added into the wood particle and polyester mixture. This mixture was again evacuated following the same procedure. After full vacuum process, the pasty mass was cast into the die to get specimens for various tests.

In this research work, test samples were made from pure polyester and polyester based wood particle reinforced composites containing 5 and 10 wt% of wood particle. So, the main variables of the test samples were wood of various strengths, proportion of wood particle (5 and 10 wt %), size of the wood particle (medium and coarse sizes), etc. Sample codes and related variables are shown in the Table 3.2.

Table 3.2: Compositions of polyester-untreated and treated wood saw dust composites

Sample Code	**Matrix**	**Filler Particles**	**Filler (wt%)**
P(0)	Polyester	Nil	0
GMM	Polyester	Gamari Medium	5 & 10
GMC	Polyester	Gamari Coarse	5 & 10
GRM	Polyester	Garjan Medium	5 & 10
GRC	Polyester	Garjan Coarse	5 & 10
KRM	Polyester	Kerosene Medium	5 & 10
KRC	Polyester	Kerosene Coarse	5 & 10
TGMM	Polyester	Treated Gamari Medium	5 & 10
TGMC	Polyester	Treated Gamari Coarse	5 & 10
TGRM	Polyester	Treated Garjan Medium	5 & 10
TGRC	Polyester	Treated Garjan coarse	5 & 10
TKRM	Polyester	Treated Kerosene medium	5 & 10
TKRC	Polyester	Treated Kerosene coarse	5 & 10

3.2.1 Casting of Samples for Breakdown Voltage Measurement

More than eight samples of pure polyester and each group of composite made through die cast method. For case, circular die as shown in Fig. 3.5 (a) was used. Before casting, materials were prepared and made them dissolved gas free following procedure described in section 3.2. The dimensions of the finished cast samples were 50 mm diameter and 3 mm thickness, Fig. 3.5 (b).


(a) **(b)**

Figure 3.5: (a) Picture of casting mould and (b) test sample for breakdown

3.2.2 Casting of Samples for Tensile Tests

More than eight samples of pure polyester and each group of composite made through die cast method. For casting tensile test samples the mould shown in Fig. 3.6 (a) was used. Properly prepared liquid polyester and mixture of liquid polyester and wood particles were cast in the mould and rectangular samples shown in Fig. 3.6 (b) were made. The rectangular samples were then machined to get standard ASTM tensile test samples, Fig. 3.6 (c). The final dimensions of the tensile test samples were 114mm×7mm×6mm.

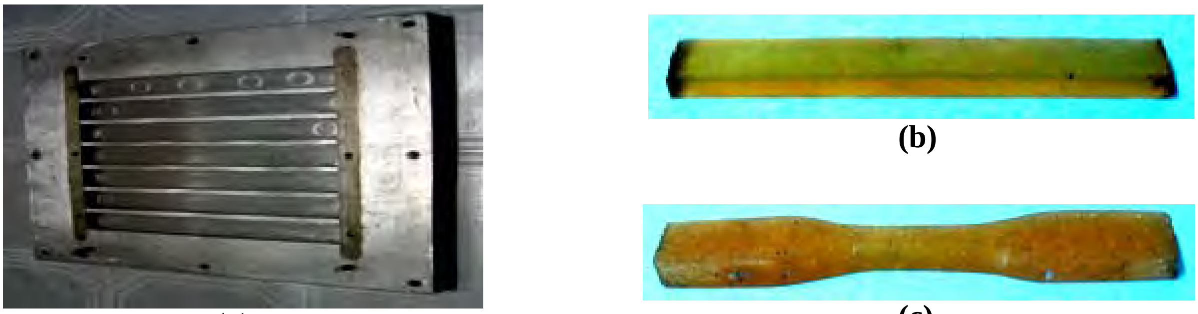

(a) **(b)** **(c)**

Figure 3.6: Photographs showing the (a) casting mould, (b) as cast sample and (c) final ASTM standard tensile test sample after machining.

3.2.3 Casting of Samples for Thermal Conductivity Tests

Five samples of each group were made by following materials and casting procedure mentioned in the earlier section. The casting used for this purpose and the as cast sample are shown in Fig. 3.7. The test samples were circular shaped with 100 mm diameter and 10 mm thickness. Among five samples, only two were taken for thermal conductivity tests. Before this of all test samples were polished by emery papers for plane surfaces.

(a) **(b)**

Figure 3.7: Photographs showing the (a) casting mould, (b) as cast sample

3.3 CHARACTERIZATION OF WOOD, POLYESTER AND WOOD PARTICLE REINFORCED POLYESTER COMPOSITES

Wood was characterized by density measurement, macrostructural analysis, tensile tests, FTIR spectroscopy, TGA, thermal conductivity tests and breakdown voltage tests. After casting of polyester and wood particle reinforced composites into required shapes and sizes, those were also characterized by various techniques used for woods except density measurement. Additionally, tensile fracture surfaces of polyester and developed composites were also observed under SEM to understand their fracture behaviours.

3.3.1 Density Measurement of Different Types of Woods

For measuring densities of different woods, the volumes of the rectangular solid wood block as shown in Fig. 3.1 were measured. Then weight of individual test block was also measured. Apparent and true densities of different types of woods were measured by appropriate method. Apparent density was measured by using equation (1), whereas true density was measured by using pycnometer, Fig. 3.8. First of all, apparent densities of Kerosene, Gamari and Garjan woods were measured. All types of wood blocks were made in cubic shapes (Fig. 3.1) so that volume of these types of woods is easy to measure. Mass of wood blocks was measured by digital balance. By measuring volume and mass of wood, the following formula was used to calculate the apparent densities.

$$\rho_A = \frac{M}{V} \tag{1}$$

Where ρ_A is apparent density, M is the mass of wood block and V is the volume of wood block.

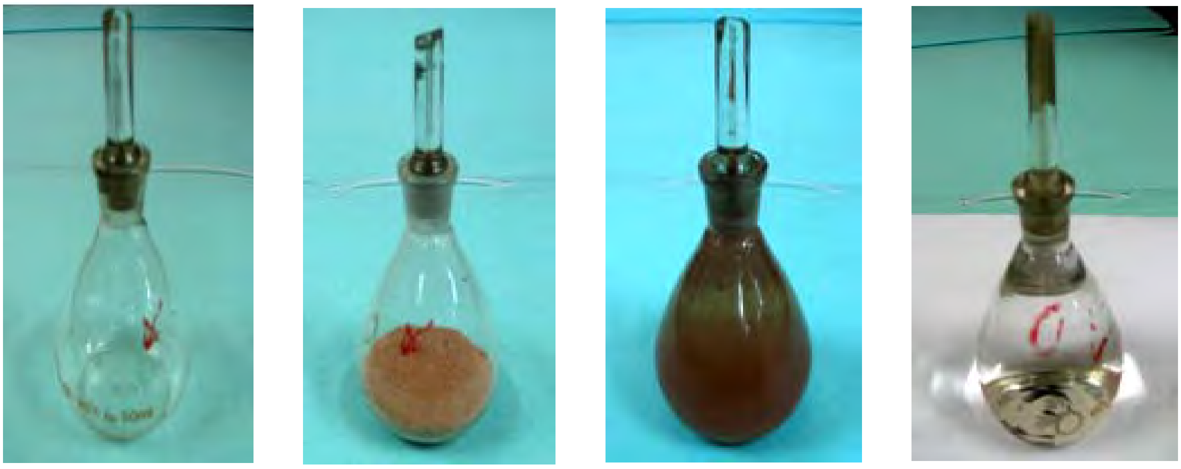

(a) (b) (c) (d)

Figure 3.8: Photographs showing (a) empty pycnometer, (b) pycnometer with fine wood particles, (c) pycnometer with fine wood particles and water and (d) water filled pycnometer.

True density was measured by using pycnometer. Here very fine particles of different woods rather than solid wood blocks were used for density measurement. Initially weight of empty pycnometer was measured, and then fine particles of wood were taken into pycnometer as shown in Fig. 3.8 (a) and (b). Weight of pycnometer with the wood particle was then measured. The pycnometer was filled with distilled water and then combined weight of pycnometer, wood particle and water was measured. Finally, the pycnometer was cleaned and dried properly. Distilled water was filled into the pycnometer (Fig. 3.7d) and total weight was again measured. After finishing measurement true density was measured by using equation (2).

$$\rho_T = \frac{W - P}{(W_1 - P) - (W_2 - W)} \times \rho_w \tag{2}$$

Where, P is the mass of empty pycnometer, W, W_1 and W_2 are mass of pycnometer with fine particle, mass of pycnometer with water and mass of pycnometer containing both fine particle and water respectively. ρ_w is the density of water. Apparent and true densities thus obtained are presented in Table 3.3.

Table 3.3: Densities of the wood saw dust

Name of the Wood	Apparent Density g/cc	True Density g/cc
Garjan	0.74	1.30
Kerosene	0.46	1.12
Gamari	0.38	0.86

3.3.2 Tensile Test

Any defect caused during casting or machining of the cast samples might influence the experimental results. So, cast samples were machined carefully to prepare tensile test samples as per ASTM D3039 standard. Among 5 to 8 machined samples, three samples were selected for test. Before tensile test, the width and thickness of the test specimens were measured. For all cases, cross-head speed of 2mm/minute was used. The load–extension curve of the specimen was recorded. In this way another two specimens were measured. These three results were averaged. Following identical test steps, tensile tests were repeated for all wood, polyester and composite samples.

3.3.3 Fractography

After tensile tests, fracture surfaces were cut for fractographic analysis under the SEM. The electrical conductivities of all test samples (wood and wood particles reinforced composites) used in this research project were very poor. To avoid this, the fracture surfaces of all samples were coated by gold sputtering technique. The samples were bonded with conductive carbon tape on an aluminium stub. Then the stub and fracture surface was further connected with highly conductive copper foil. For fractographic analysis, 15 kV accelerating voltage was used. Under SEM, various fracture features were observed and they were photographed.

3.3.4 Breakdown Voltage Test

Similar to tensile tests, three samples (fine surface sample) for each group were selected to determine the breakdown voltage. Breakdown voltages were measured using 100 kV capacity high voltage testing machine. Before starting the test, the specimen was tightly set between two electrodes as shown in Fig. 3.9. Then of high voltage testing machine was switched on and voltage was gradually increased. At some stage, the indicator of potentiometer returned to zero making a significant noise. The breakdown voltage thus obtained was converted for per cm thickness of sample. Following the same procedure, all samples were tested.

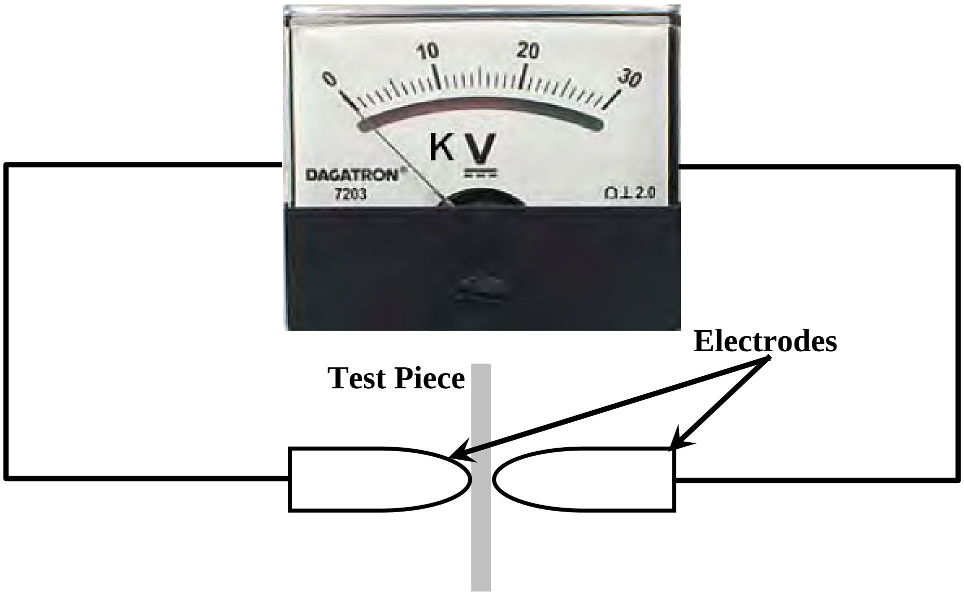


Figure 3.9: Breakdown voltage test arrangement.

3.3.5 Thermal conductivity test

Among 5 samples, only two specimens for each group were tested. Thermal conductivity was measured using Ritz apparatus. The test arrangement is shown in Fig. 3.10.

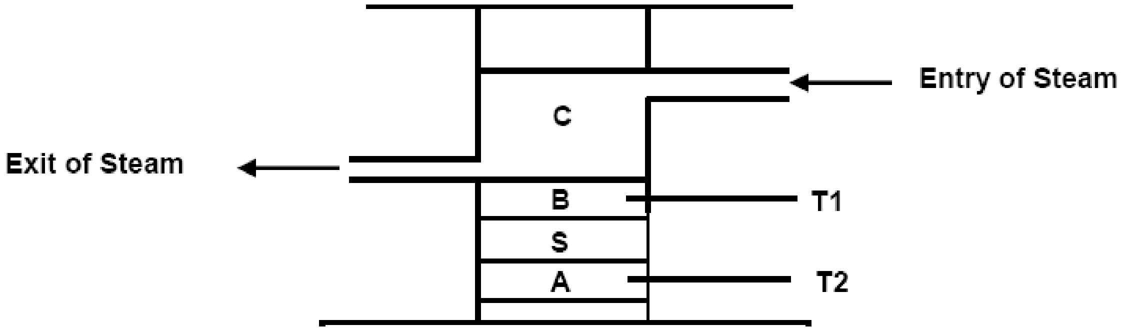


Figure 3.10: Thermal conductivity measurement setup.

In this process steam is passed into a brass chamber C has two parts consisting of hollow brass cylinder. At the bottom of steam chamber there is a copper plate B, which quickly absorbs heat from steam and transfers to the sample S placed in contact with the copper plate. Below the test sample, there is another copper plate A. Heat flows from the upper surface to the lower surface of the test piece. T1 and T2 are two thermometers to measure the temperatures of the opposite of the test specimen S. When upper temperature and lower temperature became steady position then heat supply was stopped. Cooling temperature and time were recorded until cooling temperature reached to room temperature. Cooling temperature and time were plotted in the excel sheet and obtained a cooling curve. From this cooling curve thermal conductivities were measured.

3.3.6 Fourier Transformed Infrared Radiation (FTIR) Spectroscopy

Fine powder was made from woods and composites by cutting them by a clean hacksaw. Each of them was collected on a clean piece of paper. This powder was mixed with adequate potassium bromide powder and uniformly distributed on the dies. Another dies was covered on this powder and pressed by a hand press machine to produce transparent discs which were used as test samples for FTIR machine. FTIR measurements were carried out on a SPECTRUM 100 Fourier-transform infrared spectrophotometer at a resolution of 4 cm^{-1} with an accumulation of 100 scans for each spectrum. The infrared spectra were recorded in absorbance units in the 4000–400 cm^{-1} range. FTIR spectra were measured in KBr pellets containing 1% finely powder samples. The machine was turned on and the transparent disc was placed into the slot in the machine. Automatic analysis was turned on and graph was saved. All samples of the wood and composites were tested in the same way.

3.3.7 Thermo-Gravimetric Analysis (TGA)

Small solid particle was collected from the composites and woods. These samples were measured by digital weighing machine. For thermal stability analysis, the decomposition rates were measured by a SETARAM TGT DTA 92 thermo-gravimetric apparatus. A sample of initial mass of 15 to 20 mg was introduced into a platinum crucible. The mass variation of the sample (TG) was then measured as a function of temperature (or time), and the rates of mass loss (DTG) were determined by using a thermo-balance under an inert gas nitrogen atmosphere up to 500°C, at a heating rate of 20°C min^{-1}. Finally, the data were saved. All samples of the woods and composites were tested in the same way.

3.3.8 Water Absorption Test

Since the composites absorb moisture, the mechanical property of composites must degrade. So, water absorption test of the developed composites was carried out at ambient temperature. For doing this, specimens were immersed in water bath during four weeks at ambient temperature. It might be mentioned that before immersing into all test samples were weighed out properly. After a week, these immersed samples were taken out from the water bath and wiped off water by tissue paper on both sides of the surfaces and reweighed. Again these test samples were reimmersed in the water bath. In this way, next three weeks measurements were carried out after every week. The percentage of the water absorption (A) was calculated from the following relationship-

$$A(\%) = \frac{M_w - M_d}{M_d} \times 100 \qquad (3)$$

Where, M_w = Mass of sample at wet condition

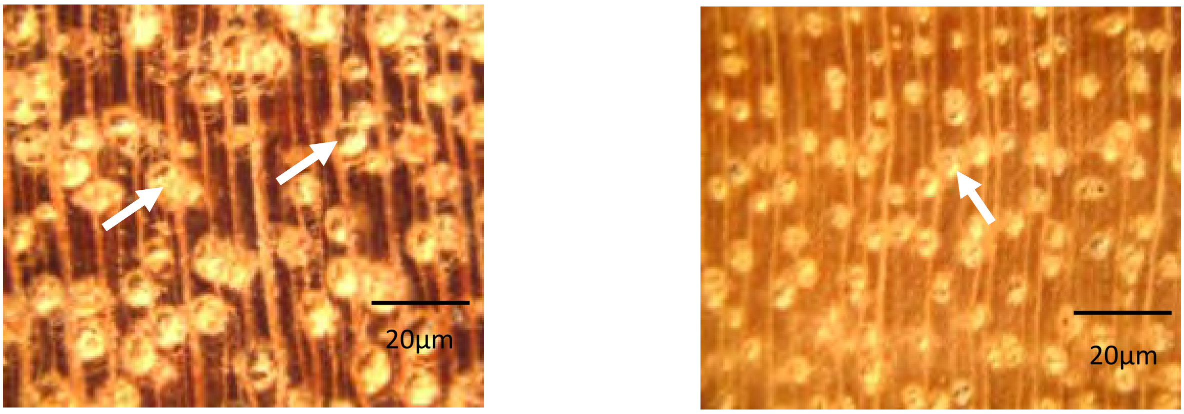
M_d = Mass of sample at dry condition

Chapter 4

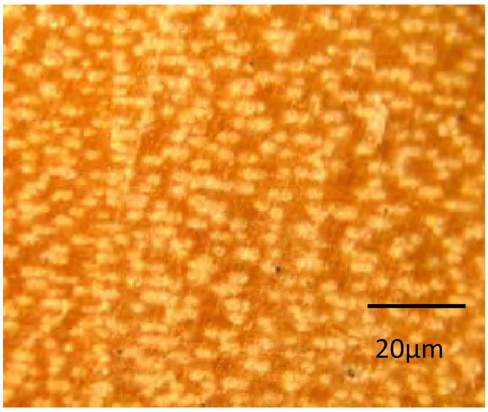
RESULTS AND DISCUSSION

4.1 MACROSTRUCTURE OF WOOD

The macrostructures of the woods (Garjan, Kerosene and Gamari) are shown in Fig. 4.1. From these macrographs it is clear that, all woods contain pores of various sizes and distributions. The pores contain dry lignin (marked by arrows).



Garjan Kerosene



Gamari

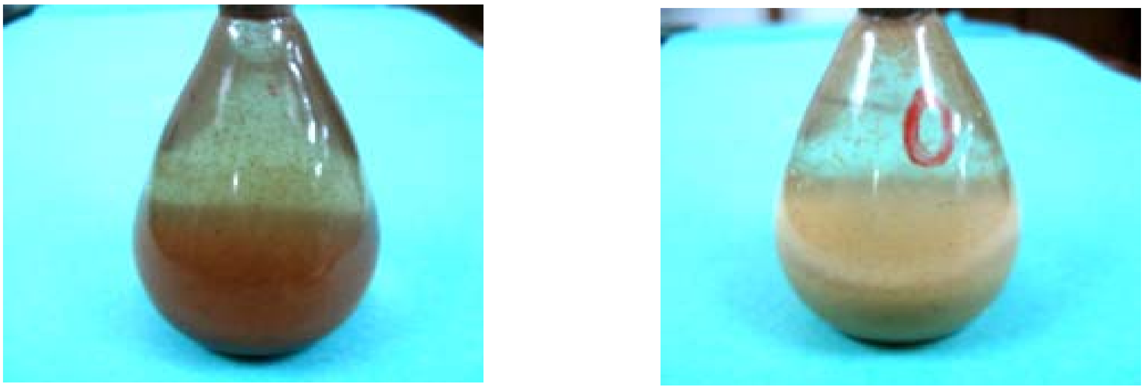
Figure 4.1: Macrostructures of various woods.

Close observation the macrostructures of woods reveals that sizes of pores in Garjan wood is much coarser than other two woods. In this case, Gamari wood shows lignin containing pores of the smallest sizes. These variations in lignin containing pores influenced their properties, which will be discussed in the subsequent sections.

4.2 DENSITY OF WOOD

Both the apparent and true densities of all woods are presented in Table 3.3. From this table it is clear that apparent density of Garjan is the highest and that of Gamari is the lowest. This table also reveals that true densities of all woods are higher compared to the corresponding apparent densities. The reason is that apparent density was measured using rectangular blocks of wood, which contained a lot of lignin associated pores. The presence of pores of various sizes and distributions in the wood block is very clear in Fig. 4.1. On the other hand, very fine machined particle was used for true density measurement. In this case, influence of pores on density became negligible. So, the true densities of all woods increased significantly.

From Table 3.3, it is also clear that similar to apparent density, Garjan wood also showed the highest density and the Gamari showed the lowest. The true densities of Garjan and Kerosene are 1.3 and 1.12 g/cc respectively, which are higher than the density of water (1 g/cc). Because of this, Garjan and Kerosene wood particles settled down at the bottom level of the pycnometer during the time of density measurement, Fig. 4.2. On the other hand, the true density of Gamari wood is 0.86 g/cc. As the true density of Gamari wood is lower than unity i.e. lower than the density of water, the wood particles floated above the water level in the pycnometer, Fig. 4.2.



Garjan Kerosene

Gamari

Figure 4.2: Settled and floated wood particles during true density measurement of various woods.

Several workers conducted research on these three woods and they also mentioned higher density of Garjan compared to that of other two woods [45, 46].

4.3 TENSILE PROPERTIES

4.3.1 Tensile Properties of Pure Wood

The tensile strength of as received wood was measured using Instron Universal Testing Machine at a cross-head speed of 2 mm/min according to ASTM D3039 standard (rectangular specimens in Fig. 3.6). The tensile properties of wood samples are presented in Table 4.1.

Table 4.1: Tensile properties of various woods.

Wood	Serial No.	Max. Load (KN)	Max. Stress (MPa)	Avg. Stress (MPa)	Break Stress (MPa)	Max. Strain (mm/mm)	Young's Modulus (MPa)
Garjon	1	4.616	128.20	125.00	128.20	0.2963	1473
	2	4.467	127.5		127.5	0.2885	1450
	3	4.140	123.80		123.80	0.2707	1393
	4	4.998	118.80		118.80	0.2923	1226
Kerosene	1	3.335	92.24	85.00	92.24	0.5443	4148
	2	3.257	85.27		85.27	0.4907	4395
	3	3.103	84.36		84.36	0.5291	3988
	4	3.103	78.60		78.60	0.4878	3622
Gamari	1	2.267	63.39	64.00	63.39	0.3272	4222
	2	2.336	65.12		65.12	0.2192	3719
	3	2.184	66.45		66.45	0.1160	3966
	4	2.109	61.00		61.00	0.1165	3485

From Table 4.1, it is clear that Garjan wood resulted the highest strength and Gamari showed the lowest. In this respect, there is relationship between densities of woods. As the density increased, the strength of the wood also increased (Tables 3.3 and 4.1).

4.3.2 Tensile Properties of Pure Polyester and Composites

4.3.2.1 Effect of Wood Strength, Proportion and Sizes of Wood Particle on Composite Strength

The tensile strength for polyester based composites reinforced with different concentrations of as-received wood saw dust particle was measured using Instron Universal Testing Machine at a cross-head speed of 2 mm/min according to ASTM D3039 standard (rectangular specimens in Fig. 3.6). The tensile properties of the untreated and treated composites are presented graphically in Fig. 4.3 - 4.6.

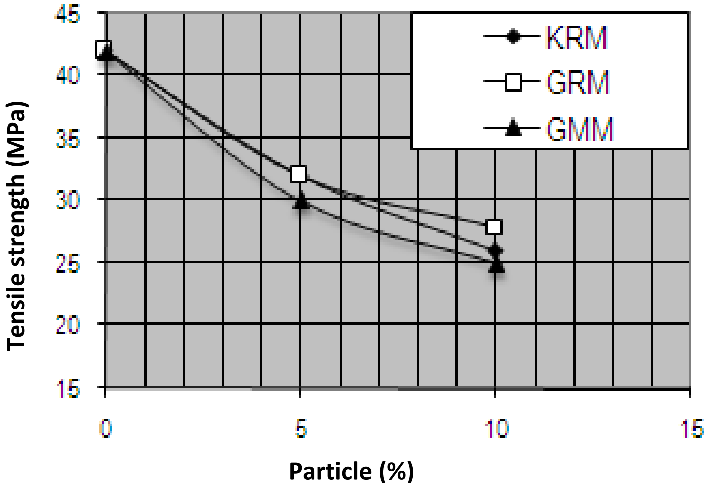


Figure 4.3: Tensile strength versus filler particle wt% curves of untreated medium size wood particle reinforced polyester matrix composites.

From Fig. 4.3, it is revealed that Garjan wood particle reinforced composites showed the highest strength and the Gamari wood particle reinforced composites showed the lowest strength. In the previous section, the strength of Garjan wood has been found to be the highest and strength of Gamari has been found to be the lowest. This diagram clearly indicates that wood strength has a significant effect on the composite strength. Wood particle of higher strength wood resulted higher strength. The possible reason behind the higher strength of higher strength wood reinforced wood particle is that during tensile loading, higher strength wood particle showed more rigidity and acted as barrier for easy crack formation. On the other hand, inter particle separation between soft wood particle is easier, i.e. crack formation between wood particle/polyester matrix interfaces or inside wood particle is easier in the case of soft wood particle reinforced composites. Once crack is formed, the composites become unstable and fail catastrophically. So, soft wood particle reinforced composites showed relatively lower strength. This type of results has also been mentioned by other investigator [64]. From Fig. 4.3, it is also clear that with increase in the proportion of wood particle, the strengths of all composites also decreased. With the increase in the proportion of wood particle, their uniform mixing and distribution inside the matrix becomes difficult and clusters (stacking) of wood particle are formed at various locations of the developed composites. As cluster of wood particle has no or little bonding strength within itself, it behaves a weak point in the composites and cracks are formed easily during tensile test. So, the composite strength gradually decreases with increase in the proportions of the wood particle.

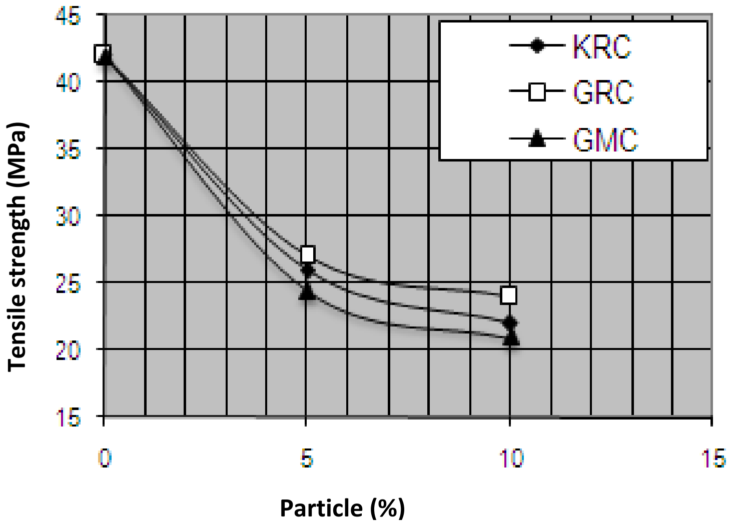


Figure 4.4: Tensile strength versus filler particle wt% curves of untreated coarse size wood particle reinforced polyester matrix composites.

Size of the wood particle has also significant effect on the tensile strength of the composites. Similar to the increase in the proportion of wood particle, increase in size of the wood particle also decreased the composite strengths, which is clear from Figs. 4.3 and 4.4. For all woods and proportions of wood particle, coarse size wood particle resulted inferior tensile strengths. It has been mentioned that the cluster of wood dust practically has a very little tensile strength. So, as the size of wood particle increases, local weakness inside the composites also increases. This ultimately decreased the composite strength, when coarse wood particle was used for making composites. The tensile strengths of this type of composites have been investigated and similar results have been found by the team of Raj et al and Mosadeghzad et al [47, 48].

4.3.2.2 Effect of Chemical Treatment of Wood Particle on Composite Strength

Figs. 4.5 and 4.6, respectively, show tensile strength versus filler particle wt% curves of treated (with 10% NaOH) medium and coarse size wood saw dust particle reinforced polyester matrix composites. From these figures, it is clear that chemical treatment slightly improved the tensile strengths of all types of composites. However, the general features such as the effects of wood strength, proportions of wood particle and their sizes were found to dominate the tensile strength of all composites reinforced with untreated wood particle.

It has been found that composites with alkali treated fillers (wood particles) show higher tensile strength. The increase of tensile properties is possibly due to the fact that the alkali treatment improves the filler surface adhesive characteristics and partly removes the relatively weak lignin portion of the wood particle. In other words, alkali treatment reduces filler size and thereby increases the aspect ratio, therefore, leads to offer better filler-matrix interface adhesion and development in mechanical properties. So, tensile strength of treated wood dust composites resulted higher tensile strength compared to that of composites reinforced with untreated wood particle. The tensile strengths of this type of composites with chemically treated wood dust have been investigated and similar results have been found by the teams of Mosadeghzad et al, Prasad et al, and Kuruvilla et al [48-50].

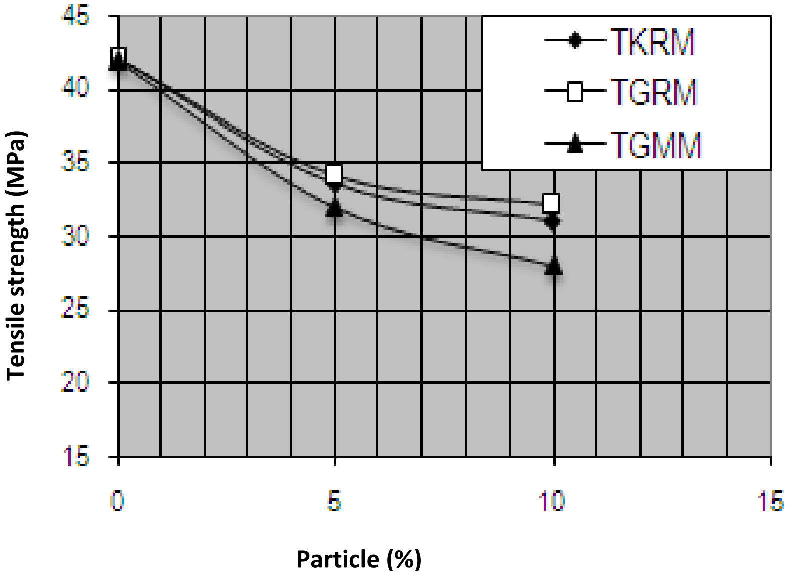


Figure 4.5: Tensile strength versus filler particle wt% curves of treated medium size wood particle reinforced polyester matrix composites.

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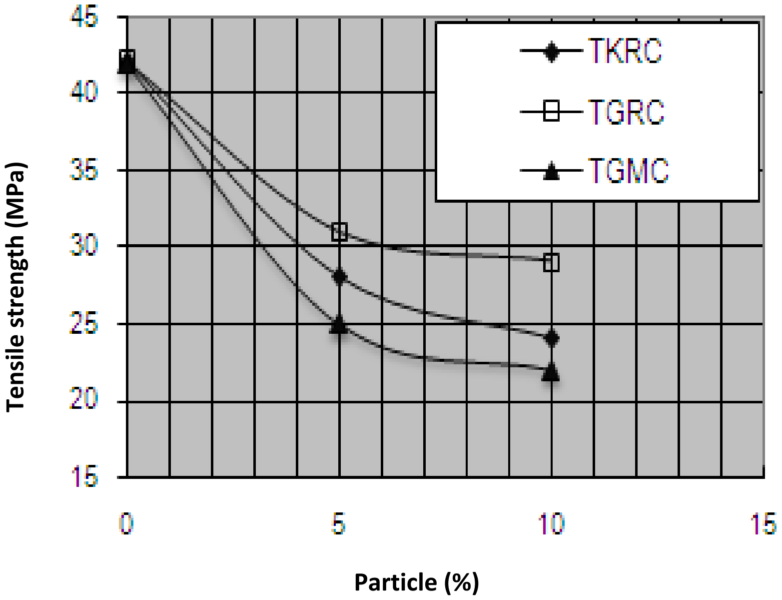


Figure 4.6: Tensile strength versus wt% particle curve of treated coarse size wood particle reinforced polyester matrix composites.

4.3.2.3 Stress- Strain Behaviors of Composites

The Stress–Strain curves of pure polyester and Garjan wood dust filled polyester composites were plotted by using tensile test data. From the curves it has been revealed that that pure polyester showed brittle type tensile behaviour, Figs. 4.7- 4.8. These results are due to fact that polyester and wood particle are brittle type materials. So, product formed after mixing of these materials arguably will also be brittle in nature. Interesting observation is that increase in proportion of wood particle or sizes of wood particle embrittled the polyester more as the ductility of the composites were found to decrease. This observation further supported either increase in the proportion of reinforcing wood particle or their sizes caused poor wood particle/polyester matrix interface bonding and easy crack formation. So, wood particle reinforced composites faced early failures and resulted poor ductility. Similar observation was found for other types of woods (Gamari and Kerosene).

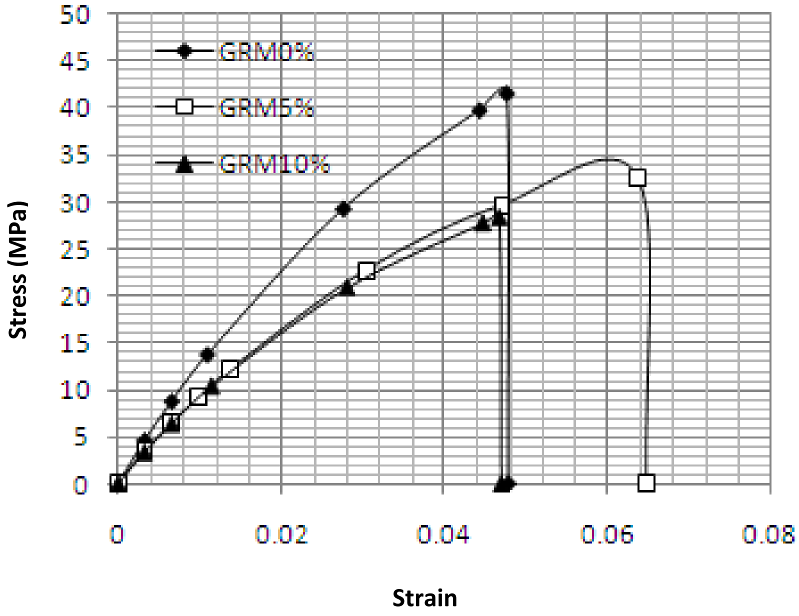


Figure 4.7: Stress vs. strain curves for pure polyester and Garjan untreated medium size wood particle reinforced composites.

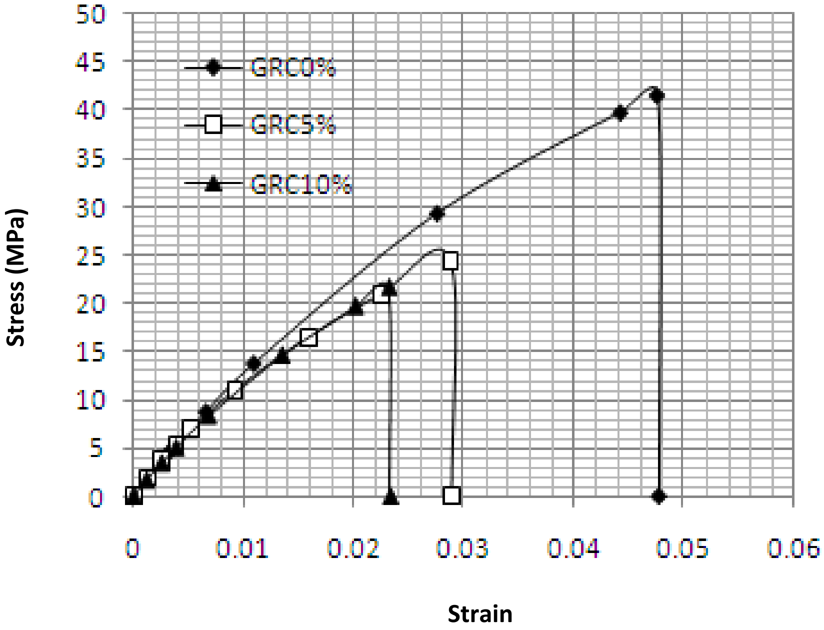


Figure 4.8: Stress vs. strain curves for pure polyester and Garjan untreated coarse size wood particle reinforced composites.

4.4 FRACTOGRAPHY OBSERVATION ON TENSILE FRACTURE SURFACES

After tensile tests of pure polyester and wood particle reinforced composites, the fracture surfaces were cut carefully to observe various fracture features under scanning electron microscope (SEM). In the case of pure moulded polyester, brittle type fracture mode was observed, Fig. 4.9. On the fracture surfaces, some micron size particles (marked by circles) were observed. These particles possibly were foreign dust particle picked up during moulding of the polyester or its production period. For some tensile samples of pure polyester, fracture was found to initiate from these foreign dust particle, Fig. 4.10. After nucleation of the crack around several dust particle (marked by circle) existing voids created due to dissolved air, it was propagated towards outer surfaces (marked by arrows).

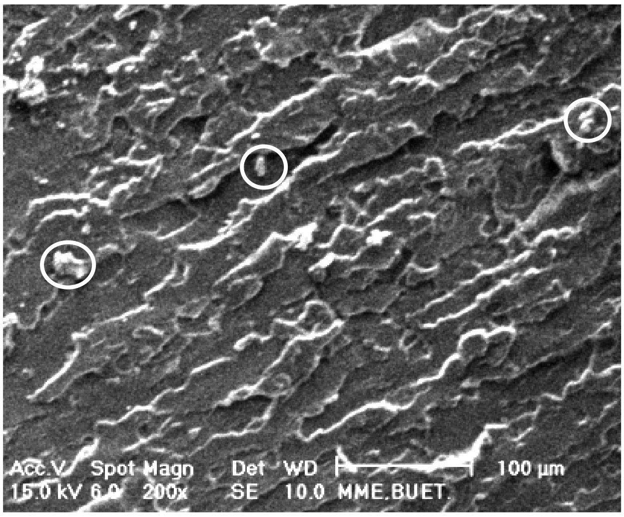


Figure 4.9: SEM micrograph showing brittle type fracture surface of pure polyester.

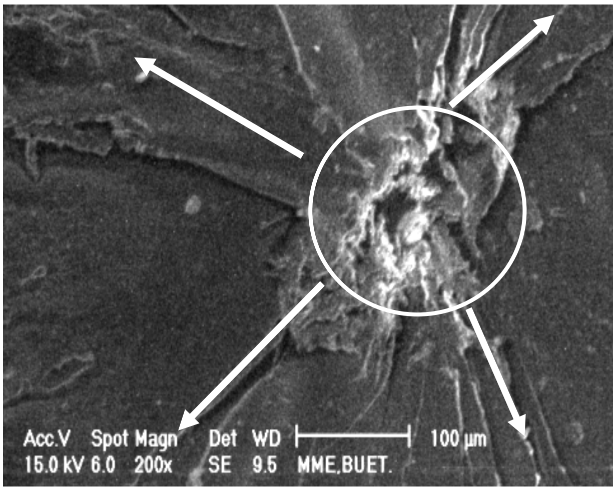


Figure 4.10: SEM micrograph showing the dust particle nucleated cracks in the pure polyester.

For all moulded composites, addition of 5% wood particle resulted better distribution of the reinforcing particle. However, bubbles created by dissolved air became very well shaped, Figs. 4.11 and 4.12. With increase in the proportion of wood particle and their sizes, tendency of wood stacking became very severe, Fig. 4.13. This type of wood stacking has been thought to be responsible to reduce the strength of the wood particle reinforced composites with increase either in the proportion of wood particle or their sizes.

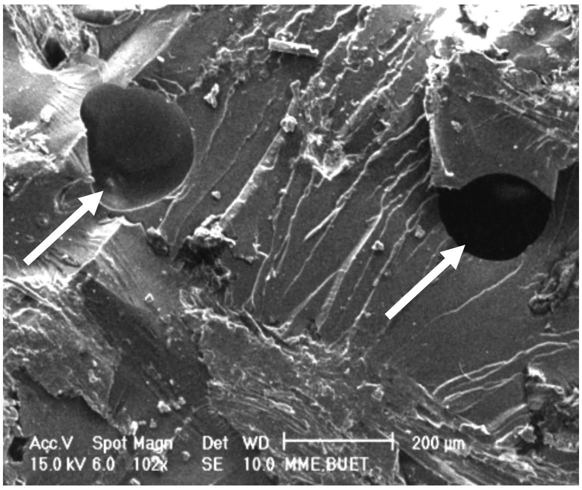


Figure 4.11: SEM micrograph showing the distribution of wood particle and air bubbles (marked by arrows) in untreated 5% wood particle reinforced polyester composites.

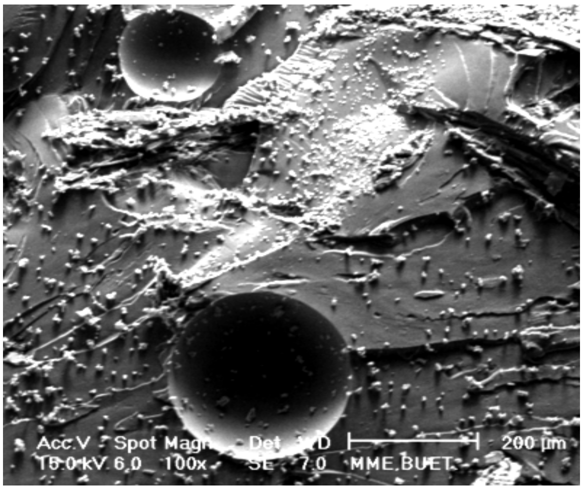


Figure 4.12: SEM micrograph showing the distribution of wood particle and air bubbles in treated 5% wood particle reinforced polyester composites.

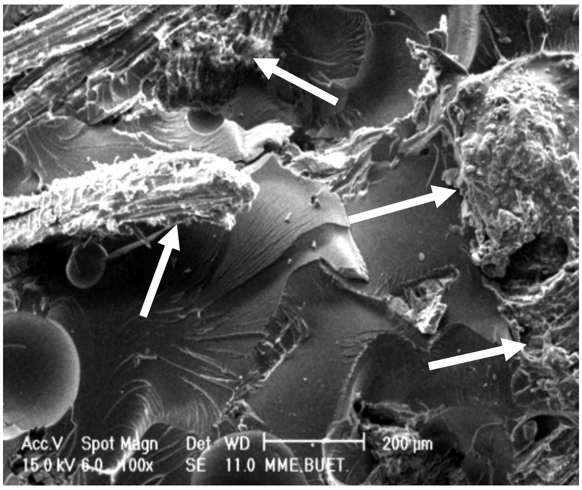


Figure 4.13: SEM micrograph showing the typical wood particle stacking in the (marked by arrows) in 10% wood particle reinforced polyester composites.

It has been mentioned that addition of wood particle caused more air bubbles in the composite and the air bubbles played important role to nucleate early crack during tensile test. This behaviour subsequently reduced the composite strength. The dominating behaviour of air bubbles in crack formation is very clear from the presence of secondary internal cracks around the air bubble, Fig. 4.14. This observation suggests that the level of vacuum treatment used the moulding of the composites was not sufficient and that adequate vacuum might improve the strength of the developed composites.

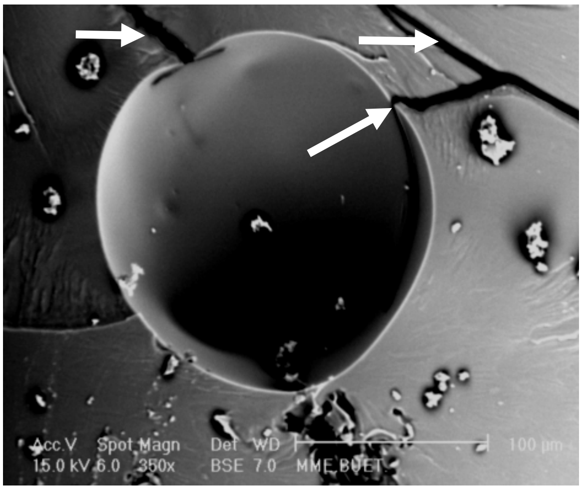


Figure 4.14: SEM micrograph showing the secondary cracks around air bubble present in the wood particle reinforced composites.

Here it is important to mention that the main purpose of this research project was to investigate the effect of wood strength on the properties of wood particle reinforced composites. Concerning the fracture behaviour, no significant effect of wood strength on the tensile fracture modes of the developed composites was observed. Similarly, chemical treatment of the wood particle did not change the fracture behaviour significantly. The possible reasons behind are that all woods were in brittle nature and that chemical treatment did not change the brittle behaviour of the wood particle.

4.5 BREAKDOWN VOLTAGE OF WOOD, POLYESTER AND COMPOSITES

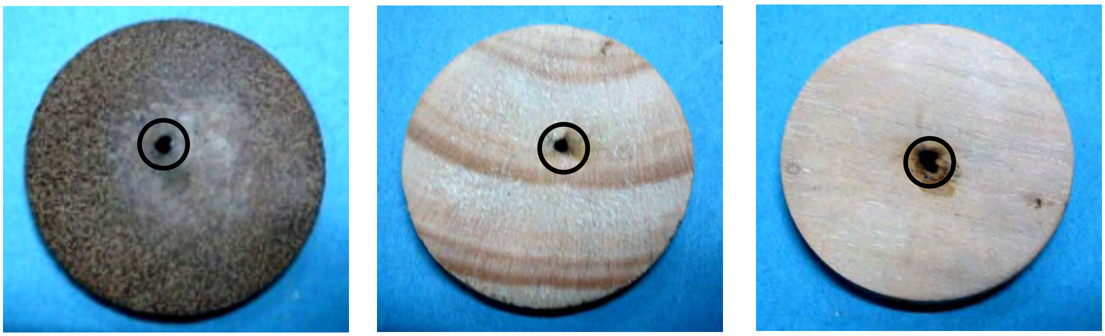
4.5.1 Breakdown Voltage of As-received Wood and Polyester

The breakdown voltages of various wood samples were measured at fixed frequency (50 Hz) in high voltage testing machine (100 KV). The average breakdown voltages of Garjan, Kerosene and Gamari woods of 1cm thick samples were 7.5, 11 and 12 KV respectively. Although, Garjan wood resulted the highest level tensile strength or density, but it showed the lowest average breakdown voltage. From these experimental results, it is clear that wood strength did not influence the breakdown voltages of the wood samples. However, size of porosity has been found to influence the breakdown voltage of wood. From Fig. 4.1, it is clearly revealed that Garjan wood has the coarsest lignin related porosity, whereas, Gamari wood showed the smallest size porosity. As a result, Gamari wood showed the highest level of breakdown voltage. It has been found that entrapped air results lower level of breakdown voltage compared to solid wood [65]. Thus the probable reason behind the lowest level of breakdown voltage of Garjan wood is due to its coarse lignin related porosity.

Various woods were used to measure breakdown voltage as shown in Fig. 4.15. It is important to mention that during breakdown voltage test burning caused large hole in the wood, Fig. 4.16. Pure moulded polyester showed air pockets of microscopic level and the proportion of air pockets was also low. So, moulded pure polyester showed better breakdown voltage. Compared to wood samples, the size of pore created after breakdown voltage test in polyester sample was also very small, Fig. 4.17. Similar to pure wood and polyester samples, pores of various sizes were also created due to burning action during breakdown voltage test. A composite sample with a typical burning related pore (marked by circle) is shown in Fig. 4.18.

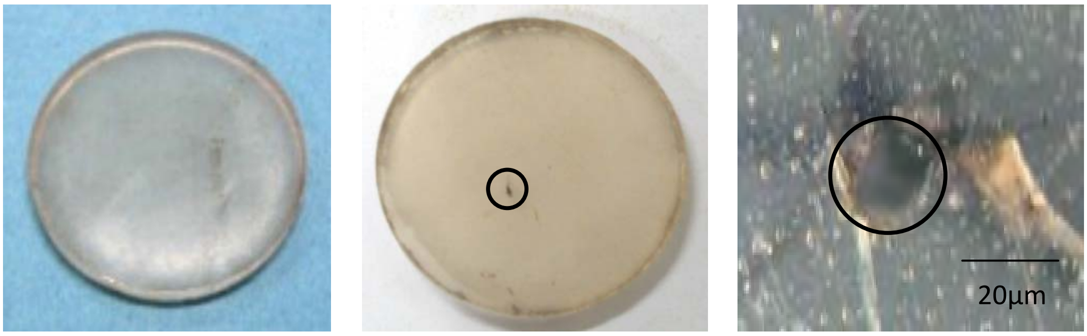
Garjan Wood Kerosene Wood Gamari Wood

Figure 4.15: Samples of various woods before breakdown voltage test.



Garjan Wood Kerosene Wood Gamari Wood

Figure 4.16: Various wood samples after breakdown voltage test. Note: During breakdown voltage test burning caused large size pores in all wood samples (marked by circles).



(a) **(b)** **(c)**

Figure 4.17: Photographs of polyester samples (a) before breakdown voltage test, (b) after breakdown voltage test and (c) close-up view of pore marked by circle on Fig. 4.17b.

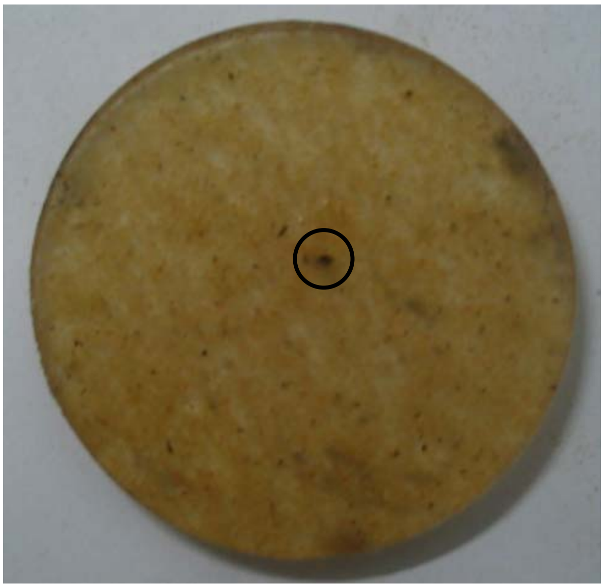


Figure 4.18: Photograph showing a typical pore created during breakdown voltage test of a composite sample.

4.5.2 Effect of Proportion and Wood Particle Size on Breakdown Voltage of Polyester

The effects of proportions and sizes of wood saw dust composites on breakdown voltage are illustrated graphically in Figs.4.19 and 4.20. From these figures it is clear that a gradual decrease in breakdown voltage were resulted for all woods. In this case, Gamari wood particle reinforced composites showed better breakdown voltages, possibly due to the smaller size of porosity in the Gamari wood. The reason of deterioration in breakdown voltage of the wood saw dust reinforced polyester based composites with wood particle content is that as the filler content increases, thereby increasing the interfacial area with worsening interfacial bonding between the filler and the matrix leads to decreasing value of the breakdown voltage and wood saw dust exhibits polar nature, whereas the nature of the polyester is non-polar [53]. Besides this, the breakdown voltage of wood is significantly lower than that of polyester. So, the breakdown voltage decreased for all untreated wood saw dust reinforced composites. From Fig. 4.20, it is clear that coarse particle further deteriorated the breakdown voltages of the composites. The reason behind is that coarse particle caused improper mixing and pre-cracks (during fabrication) in the composites, which ultimately consequence the easy passage of current in the composites.

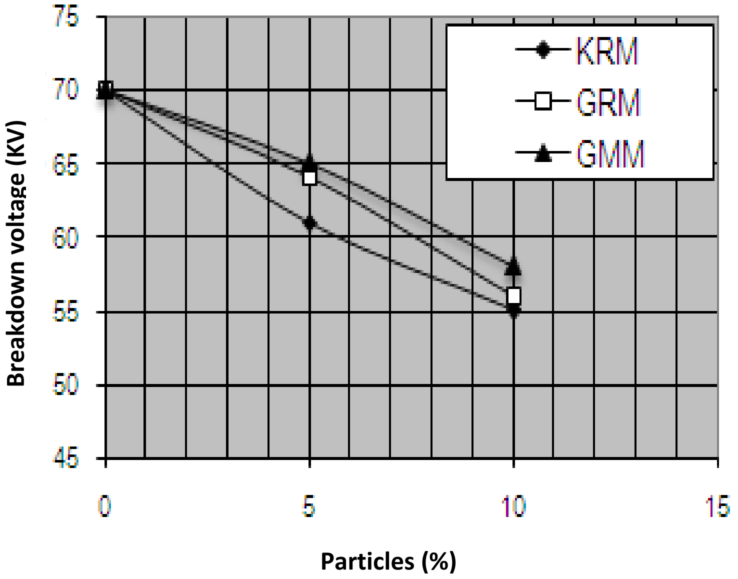


Figure 4.19: Variation in breakdown voltage of composites with medium size wood particle content.

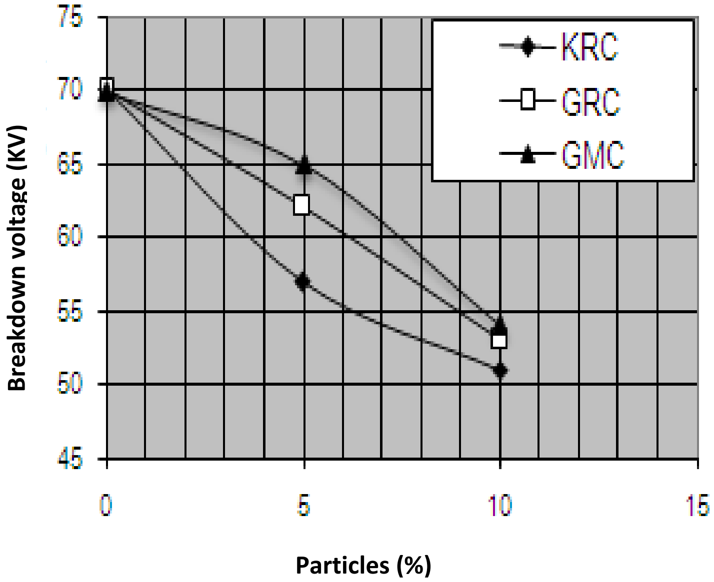


Figure 4.20: Variation in breakdown voltage of composites with coarse size wood particle content.

4.5.3 Effect of Chemical Treatment of Wood Particle on Breakdown Voltage

Figs. 4.21 and 4.22 show the effects of chemical treatment of wood particle on the breakdown voltage of wood particle reinforced composites. When wood dust was treated with alkali, then part of the lignin was removed and this treatment reduces filler size and thereby increases the aspect ratio. These overall effects caused better filler-matrix interface adhesion and improved the breakdown voltages of the composites (Figs. 4.19 to 4.22). The improvement in breakdown voltages has been observed for both (medium and coarse) size particle.

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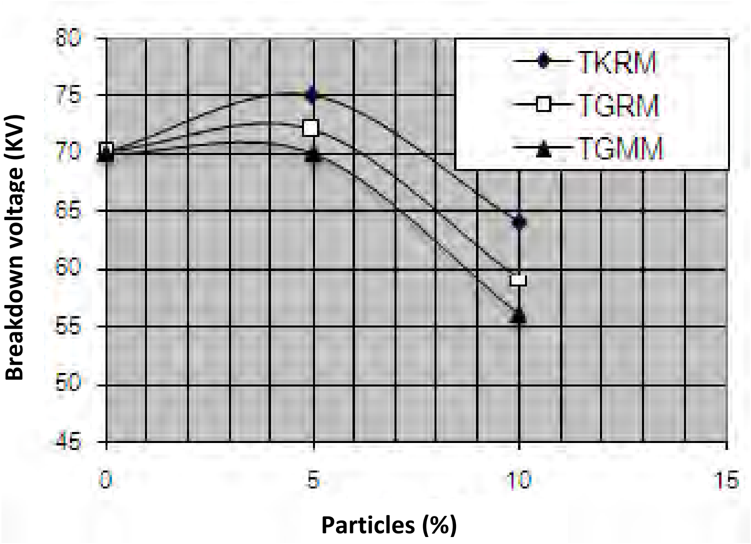

Figure 4.21: Variation in breakdown voltage of composites with treated medium size wood particle content.

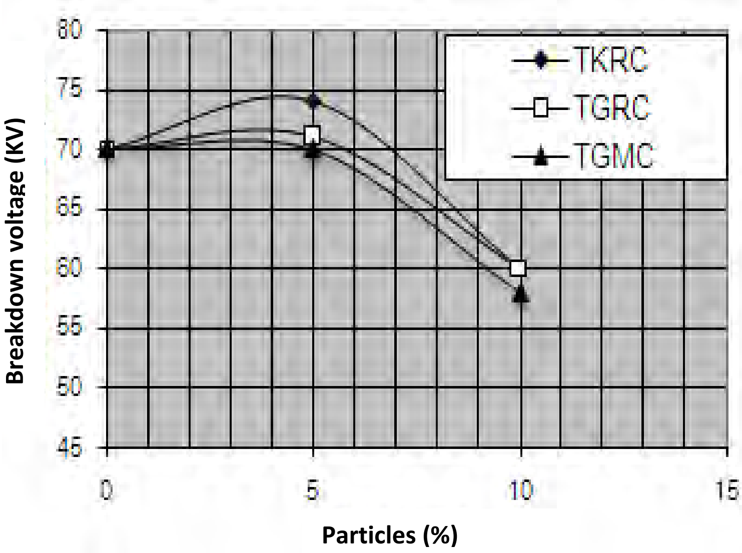

Figure 4.22: Variation in breakdown voltage of composites with treated coarse size wood particle content.

4.6 THERMAL CONDUCTIVITY OF COMPOSITES

4.6.1 Effect of Proportion and Wood Particle Size on Thermal Conductivity of Polyester

Thermal conductivities of pure wood, polyester and wood dust filler composites were measured by using Sale's method. The thermal conductivity curves of pure woods and polyester are presented in Fig. 4.23. From this figure it is clear that pure polyester has lower thermal conductivity than that any as-received wood used for this research. These types of different thermal conductivity values of pure wood and polyester have been mentioned by others [66, 67].

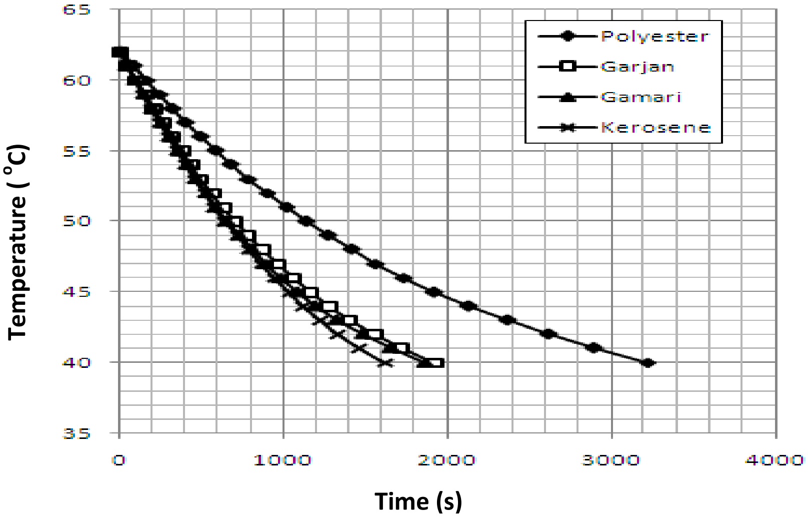

Figure 4.23: Temperature versus time curves of pure polyester and as-received wood.

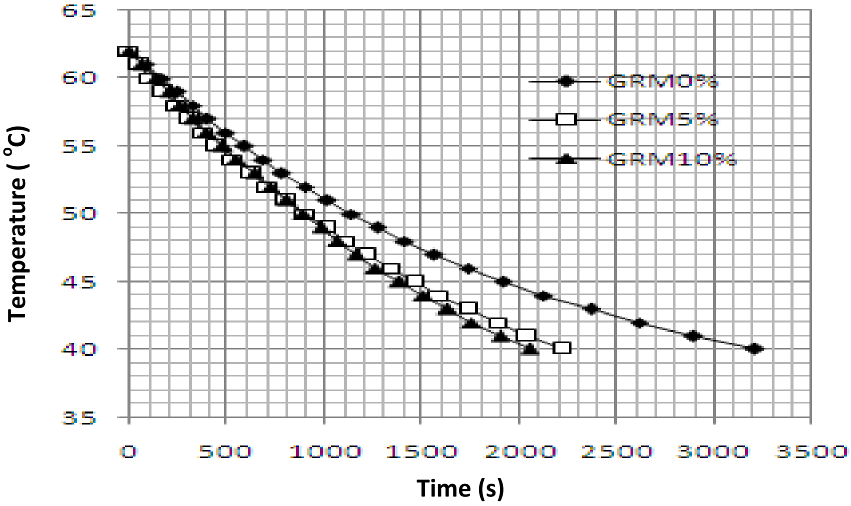

Figure 4.24: Temperature versus time curves of pure polyester and untreated medium size Garjan wood particle reinforced composites.

The effects of volume fraction of wood particle of various woods on the temperature vs. time curves for the thermal conductivity tests are presented in Figs. 4.24-4.26. From these figures it has been seen that the cooling rate of pure polyester is slower, i.e. the thermal conductivity of pure polyester is lower than that of the wood particle reinforced composites. From these figures, it is also evident that with increase in the proportion of wood particle, thermal conductivities of all woods slightly increased.

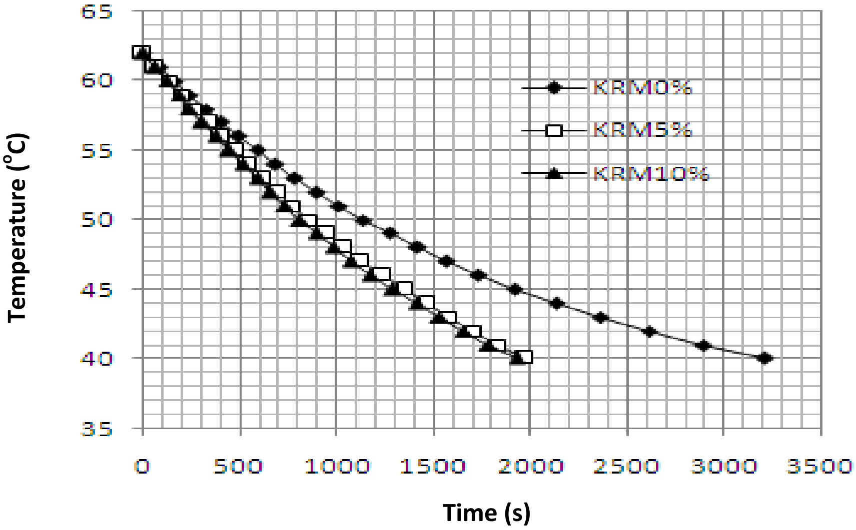

Figure 4.25: Temperature versus time curves of pure polyester and untreated medium size Kerosene wood particle reinforced composites.

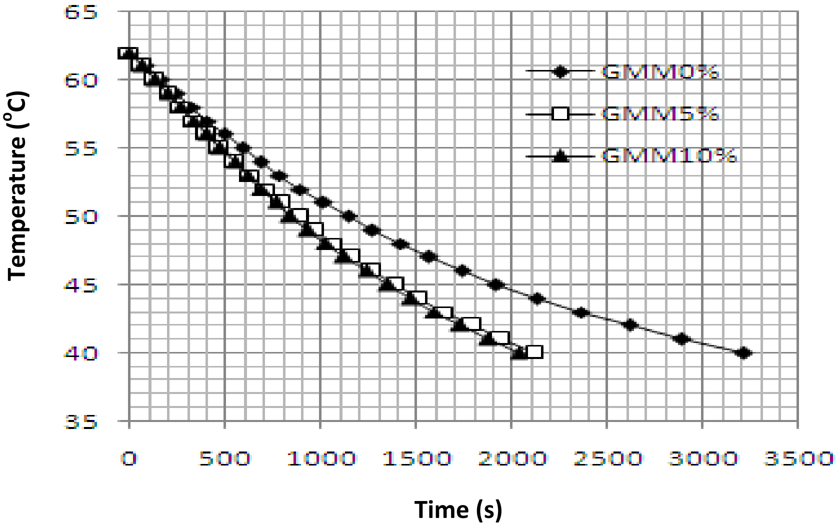

Figure 4.26: Temperature versus time curves of pure polyester and untreated medium size Gamari wood particle reinforced composites.

The reason of improvement in thermal conductivity of the wood saw dust reinforced polyester based composites is that the filler material contains thermal releasing objects which are thermally conductive whereas used wood is thermally conductive compared to polyester. The effects of coarse size wood particle on the thermal conductivities of wood particle reinforced composites are presented in Figs. 4.27-4.29. From these figures it is clear that coarse size of wood particles within the range used in this research has no significant effect on the thermal conductivities of the developed composites. These results are arguable due to the reason that all woods have similar type of cellulosic materials having similar thermal conductivity levels.

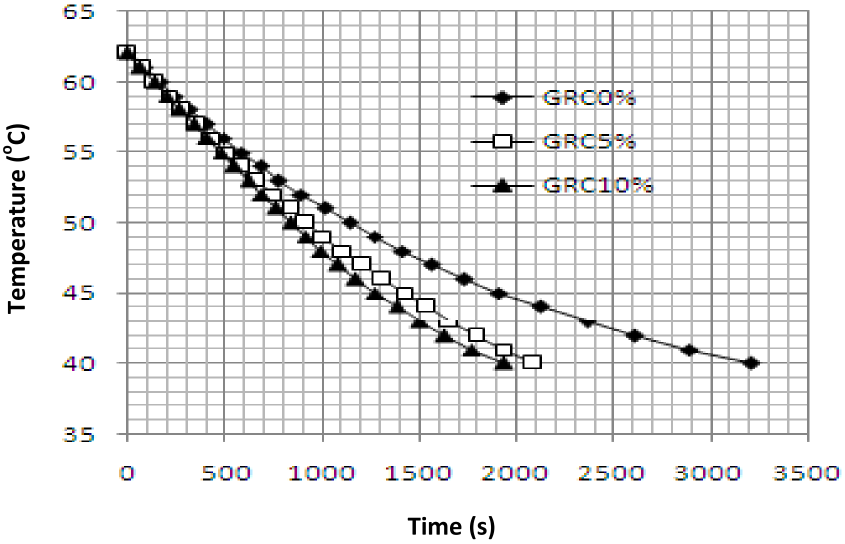

Figure 4.27: Temperature versus time curves of pure polyester and untreated coarse size Garjan wood particle reinforced composites.

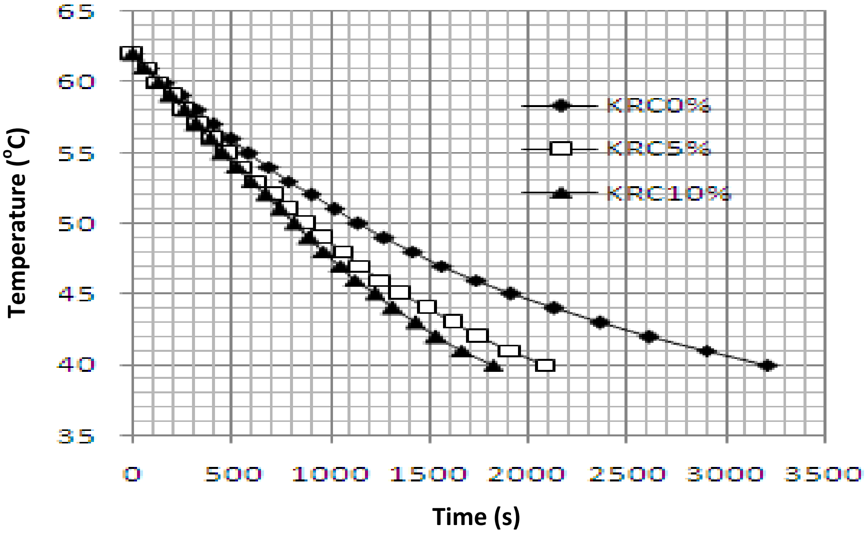

Figure 4.28: Temperature versus time curves of pure polyester and untreated coarse size Kerosene wood particle reinforced composites.

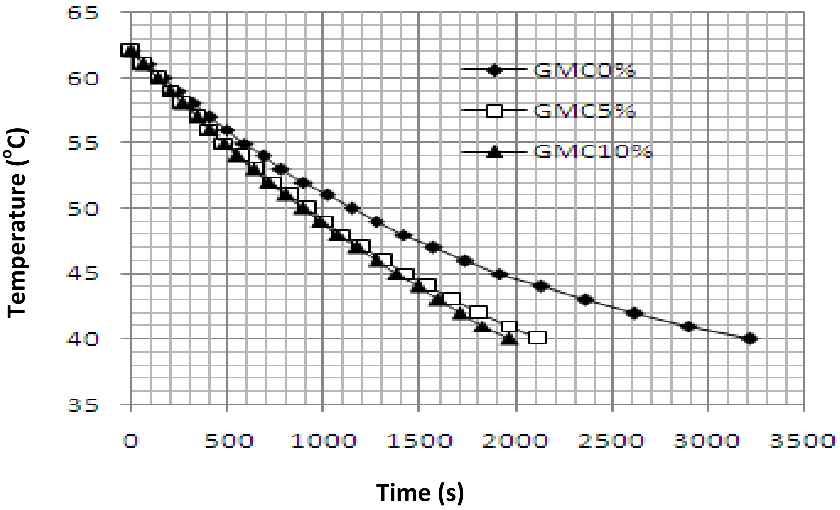

Figure 4.29: Temperature versus time curves of pure polyester and untreated coarse size Gamari wood particle reinforced composites.

4.6.2 Effect of Chemical Treatment of Wood Particle on Thermal Conductivity of Composites

The temperature versus time curves of chemically treated wood particle reinforced composites are shown in Figs. 4.30-4.32. It has been found that the composites with alkali treated fillers result lower thermal conductivity than untreated fillers. The possible reason behind is that during chemical treatment lignin portion was partially removed. Usually wood with lignin and/or moisture shows higher thermal conductivity [66, 67]. So, the experimental results found in the research project are really not exceptional.

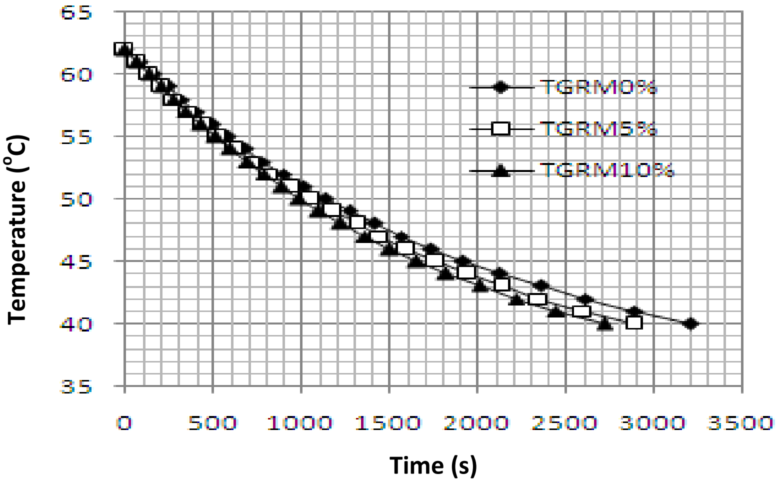

Figure 4.30: Temperature versus time curves of pure polyester and treated medium size Garjan wood particle reinforced composites.

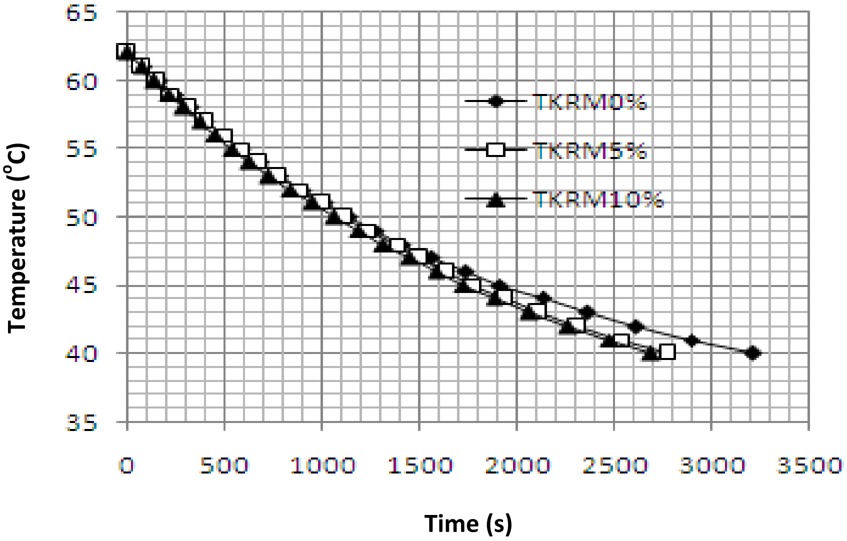

Figure 4.31: Temperature versus time curves of pure polyester and treated medium size Kerosene wood particle reinforced composites.

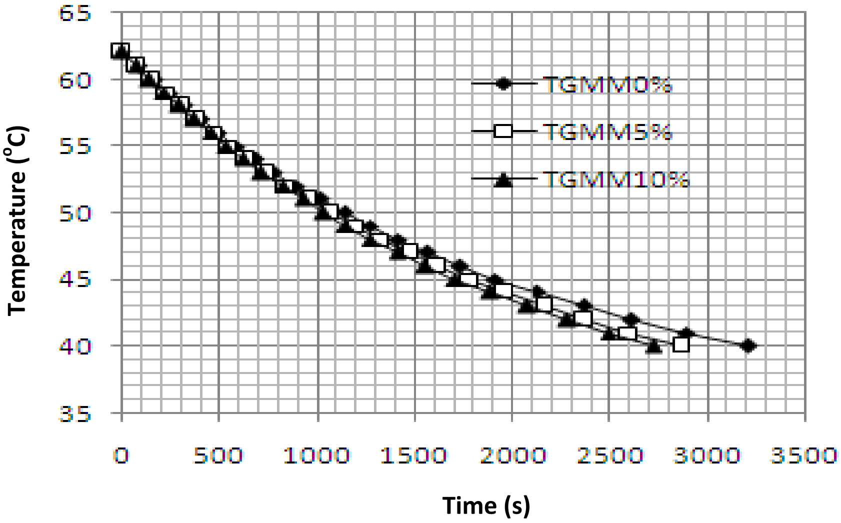

Figure 4.32: Temperature versus time curves of pure polyester and treated medium size Gamari wood particle reinforced composites.

It has been mentioned that two different sizes of wood particle (medium and coarse) were used to reinforce the composites. For coarse size particle reinforced composites changes in thermal conductivities were very similar to that found for medium size particle reinforced composites, which is clear from Figs. 4.33- 4.35. The reasons of variation in thermal conductivity changes with fiber content were also not different.

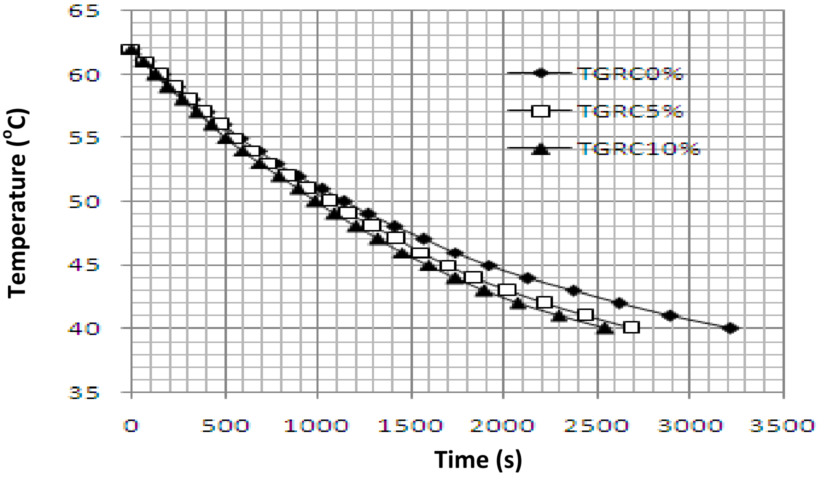

Figure 4.33: Temperature versus time curves of pure polyester and treated coarse size Garjan wood particle reinforced composites.

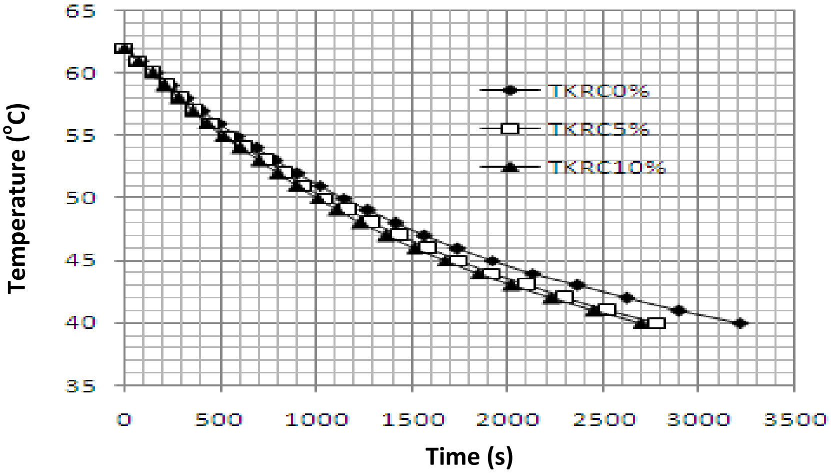

Figure 4.34: Temperature versus time curves of pure polyester and treated coarse size Kerosene wood particle reinforced composites.

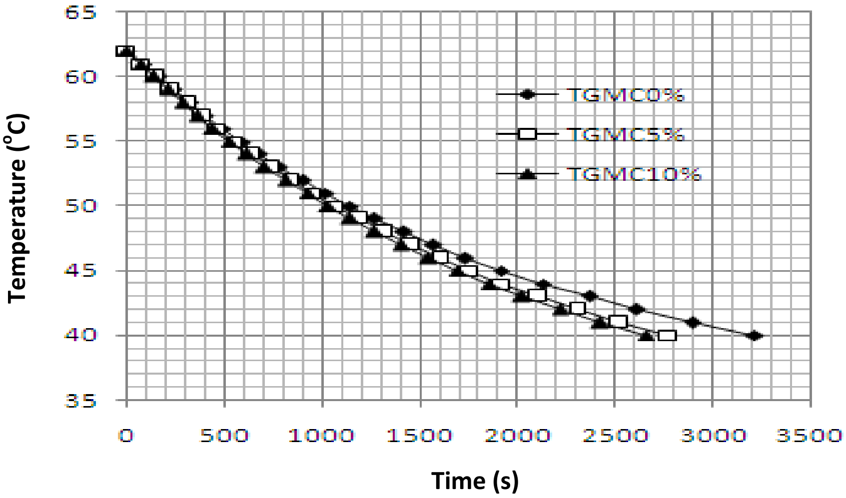

Figure 4.35: Temperature versus time curves of pure polyester and treated coarse size Gamari wood particle reinforced composites.

4.7 CHARACTERIZATION OF PURE POLYESTER, WOOD AND COMPOSITES BY FTIR

4.7.1 FTIR Results of Pure Polyester and As-received Woods

Pure polyester, woods and developed composites were characterized by FTIR. These spectra reveal the presence of various types of bonds in the materials.

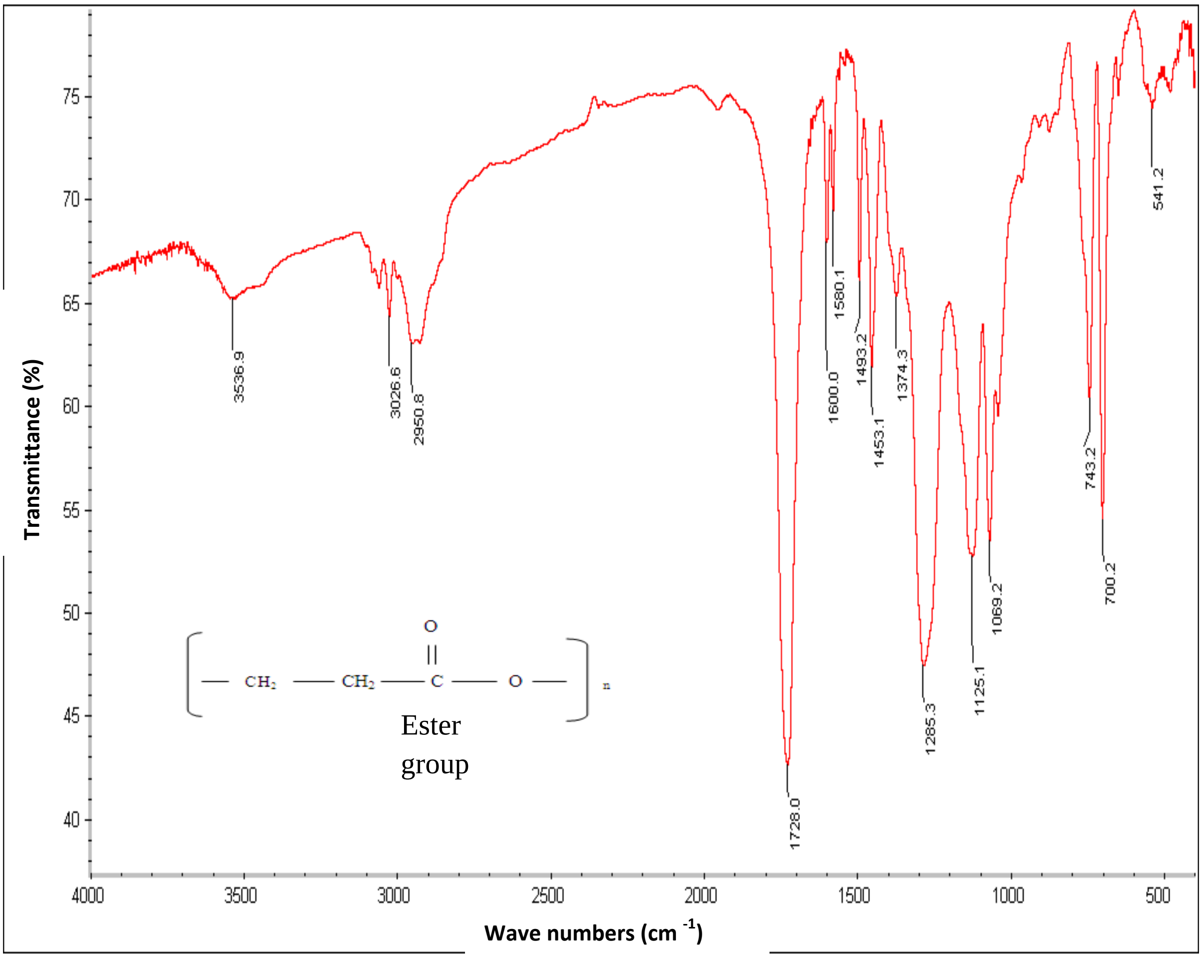

Figure 4.36: FTIR spectra of pure polyester.

The FTIR profile of polyester is shown in Fig. 4.36. From this spectrum the major peaks are found at 3536.9 cm^{-1}, 2950.8 cm^{-1}, 1728.0 cm^{-1}, (1100 - 1300) cm^{-1}, 1069 cm^{-1} and 700 cm^{-1} corresponding to the bonds –OH stretch (alcohol), C-H stretch (alkyl), C=O stretch (ester), C-O stretch (ester), C-O stretch (aliphatic amines), C─H stretch (aromatic).

The FTIR profile of untreated Garjan wood is shown in Fig. 4.37. From this spectrum the major peaks are found at 3420.8 cm^{-1}, 2921.5 cm^{-1}, 1734.3 cm^{-1}, 1684.8 cm^{-1}, 1267.2 cm^{-1}, and 1023.7 cm^{-1} corresponding to the bonds –OH stretch(alcohol), C-H stretch(alkyl), C=O stretch (esters), C-

O stretch (ester), C-O stretch (aliphatic amines). In Fig. 4.38 the FTIR spectra of as-received Gamari wood is presented. From Figs. 4.37 and 4.38, it is clear that for all as received woods FTIR absorption spectra have been found almost at similar locations, which indicate that all woods are composed of similar types of fiber.

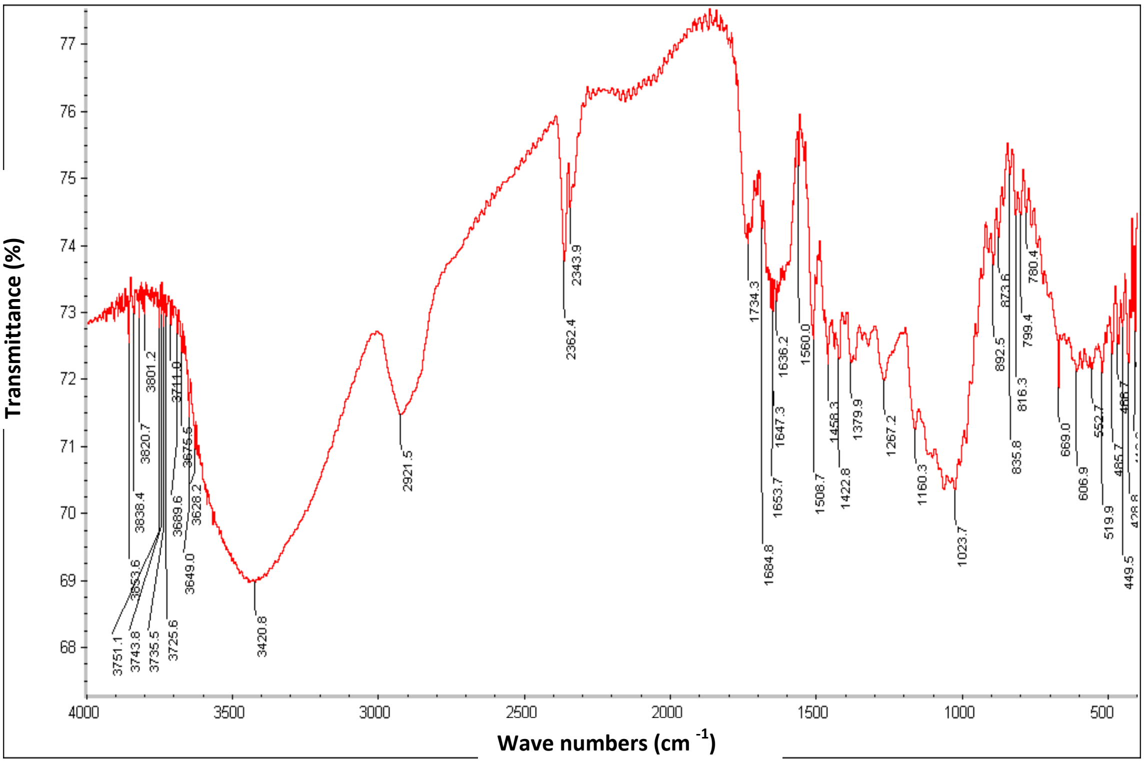

Figure 4.37: FTIR spectra of as-received Garjan wood.

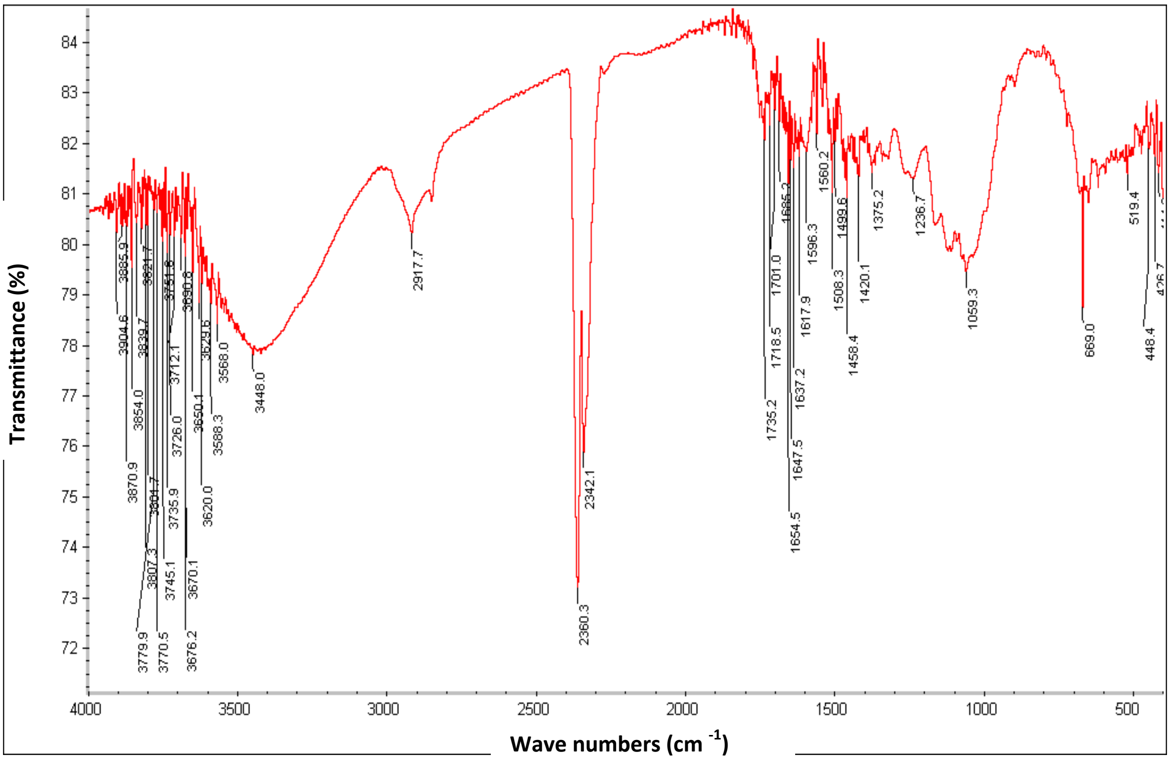

Figure 4.38: FTIR spectra of as-received Gamari wood.

4.7.2 Effects of Chemical Treatment on FTIR Results of As-received Woods

Wood dust particle was treated with 10% NaOH solution. After this chemical treatment, wood particle was investigated by FTIR. The FTIR spectra of chemically treated wood particle are shown in Figs. 4.39-4.40. After this chemical treatment, wood particle were investigated by FTIR.

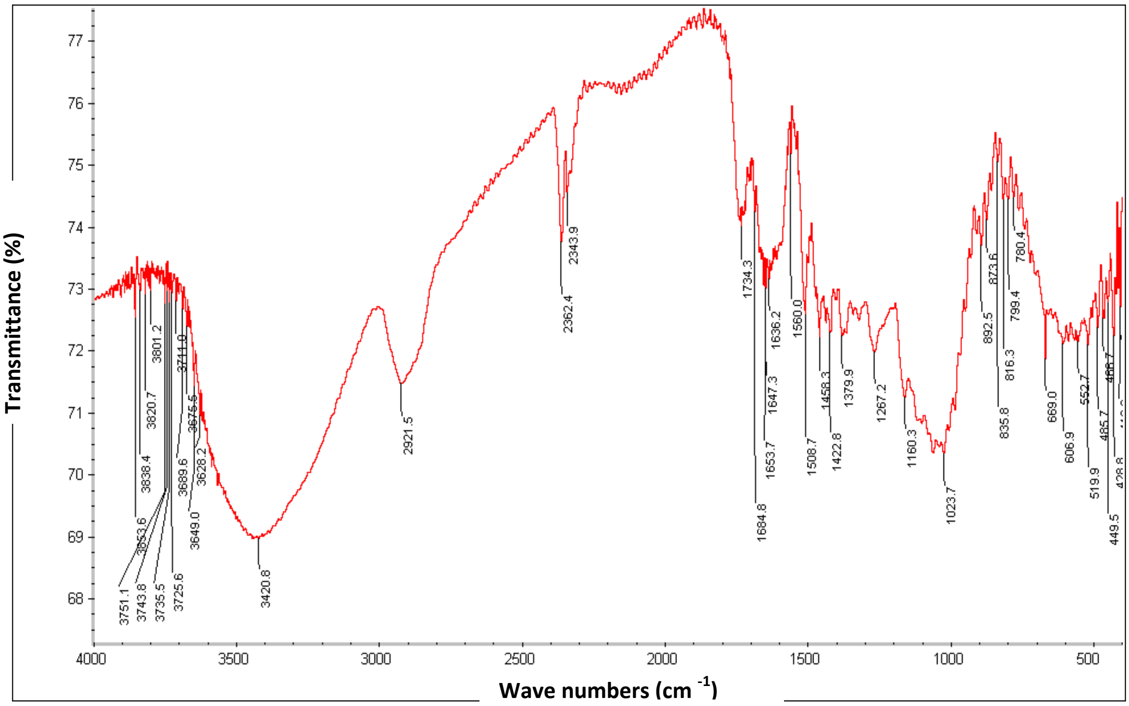

Figure 4.39: FTIR spectra of treated Garjan wood.

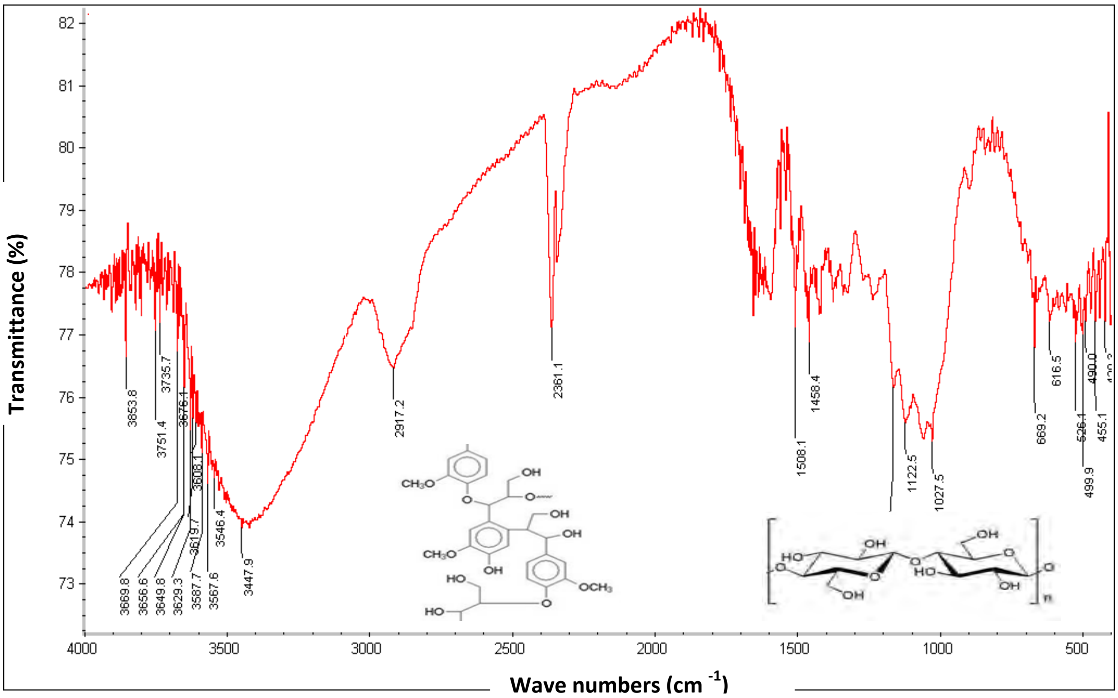

Figure 4.40: FTIR spectra of treated Gamari wood.

Comparison of FTIR spectra of un-treated (Garjan and Gamari as-received wood particle) and treated wood particle presented in Figs. 4.37-40, it is revealed that, for all cases, the positions of absorption peaks are located at similar locations. Only difference among these FTIR spectra is the degree of absorption of peaks at various locations. It is thought that chemical treatment significantly removes the lignin portion of the wood particle. The decrease in the size of peak at 2362 cm^{-1} level for all woods indicates the proportion of the characteristic fiber at this location has been decreased. As chemical treatment significantly decreases the lignin portion of the wood fiber, this results suggests that the peak position of the lignin in the wood is at 2362 cm^{-1}. Some other researchers have also been mentioned similar results [48, 68].

4.7.3 FTIR Results of Wood Particle Reinforced Polyester Composites

The FTIR profile of untreated Kerosene 10% wood dust developed composite is shown in Fig. 4.41. The major peaks are 3529.7 cm^{-1} and 2950.2 cm^{-1} and 1734.2 cm^{-1} and 1285.4 cm^{-1} corresponding to the bonds –OH stretch (alcohol), C-H stretch (alkyl), C═0 stretches (ester) C-O (ester). From above figure, it has been seen that peaks of developed composite are existed from 3500 – 4000 cm^{-1} which has both polyester and untreated Kerosene wood individually, small peaks are visible from 3000 - 2000 cm^{-1} which has in pure polyester.

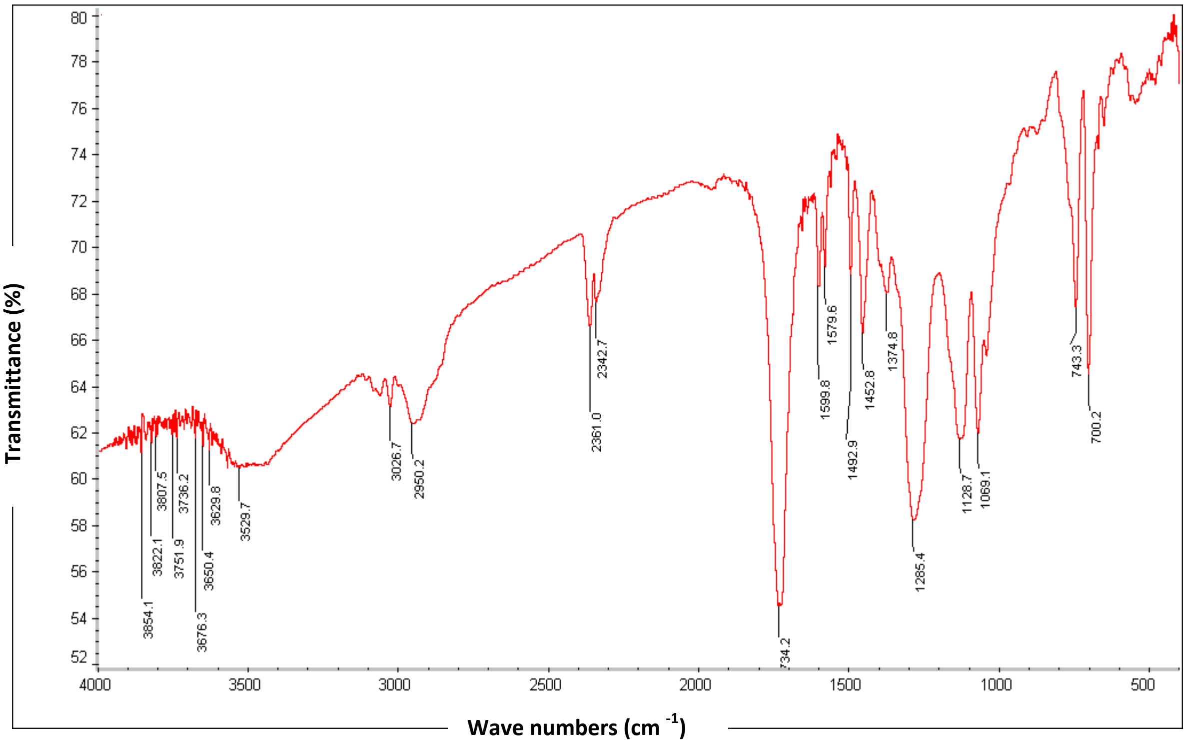

Figure 4.41: FTIR spectra of untreated 10% Kerosene wood particle reinforced composite.

Besides this many peaks of developed composite are existed from 2000 – 500 cm^{-1} which has seen in pure polyester and wood dust. Peaks of wood did not merge with pure polyester because when wood dust increased into matrix then, wood dust was dominating in the composites. From this figure it is also clear that pure polyester and wood dust mixed uniformly and maintained their individual characteristics.

The FTIR profile of treated Kerosene 10% wood dust developed composite is shown in Fig. 4.42. The major peaks are 3564.1 cm^{-1}, 2357.5 cm^{-1}, 1731.5 cm^{-1}, and 1285.5 corresponding to the bonds –OH stretch (alcohol), C═0 stretches (ester), C-O (ester). From this figure, it has been seen that all peaks of developed composite are present from 3500 – 4000 cm^{-1} which has both polyester and Kerosene treated wood, small peaks are visible from 3000 - 2500 cm^{-1} which has in pure polyester and a peak of treated Kerosene wood dust has been seen in the range of 2340-2361 cm^{-1} which has no untreated kerosene wood dust. It has also been seen that a peak of developed composite merged in the range 2900 -2950 cm^{-1}. Besides this, maximum peaks of developed composite are visible from 2000 – 500 cm $^{-1}$ which has both pure polyester and treated wood dust. From this figure it is clear that pure polyester and wood dust mixed uniformly and remained their individual characteristics.

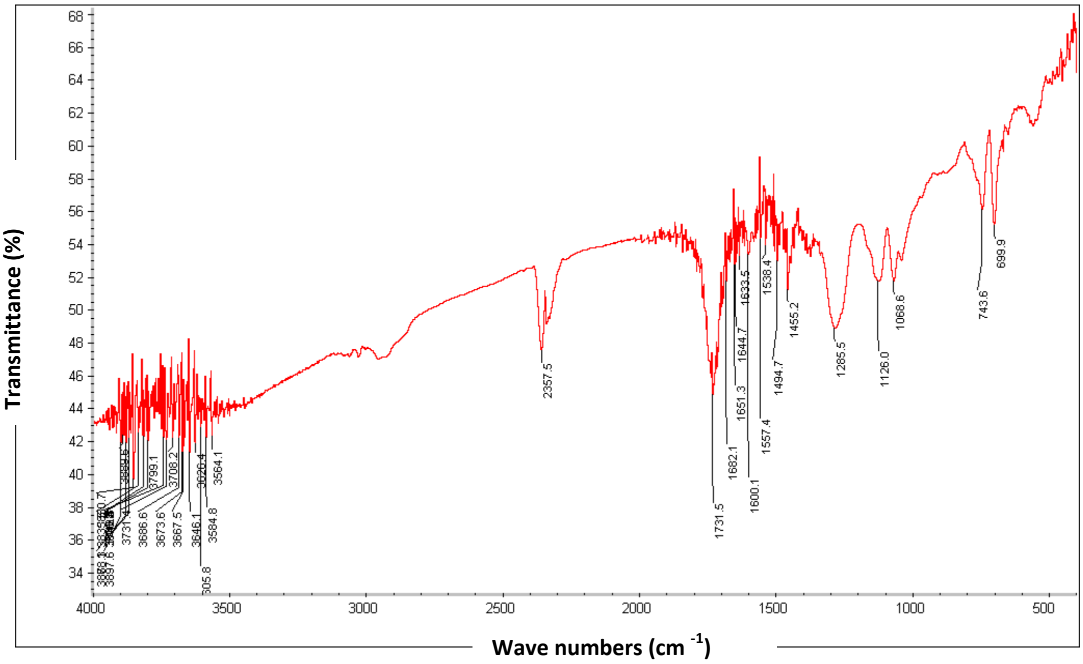

Figure 4.42: FTIR spectra of treated 10% Kerosene wood particle reinforced composite.

For other woods (Garjan and Gamari) particle reinforced polyester based composites similar spectra has been found.

4.8 THERMO-GRAVIMETRIC ANALYSIS (TGA) OF WOOD PARTICLE REINFORCED COMPOSITES

4.8.1 Effect of Wood Particle on Thermal Stability of Pure Polyester

The TGA curves of weight loss as a function of temperature for polyester and untreated Kerosene wood particle reinforced composites are shown in Fig. 4.43. From this figure it is clear that pure polyester and all composites (containing both 5 and 10 % wood particle) exhibit thermal degradation and mass loss at elevated temperature. On glass transition temperature (187.78°C) of pure polyester, up to 10% of wood particle has no significant effect. At the glass transition temperature pure polyester begins to lose their rigidity, strength and dimensional stability [54]. From the experimental results, it seemed that up to 10% wood particle did not cause further deteriorate the stability of the polyester. With increase in temperature the thermal degradation behaviours of both pure polyester and composites are very similar. However, at final temperature, pure polyester experienced more decomposition (weight loss) and that increase in wood particle content slightly decreased the decomposition. This result is due to fact that polyester contains more volatile materials than woods. So, woods contents composites might have higher residue than pure polyester.

The increase of the thermal stability is attributed to the formation filler composites shielding network. Moreover, wood particle contain different types of minerals, which might have better thermal stability compared to that of pure polyester. The Thermo-Gravimetric analyses of these types of composites without chemically treated wood dust have been investigated and similar result has been by the team of Panfang Lu et al and Tartaglione et al [55, 56].

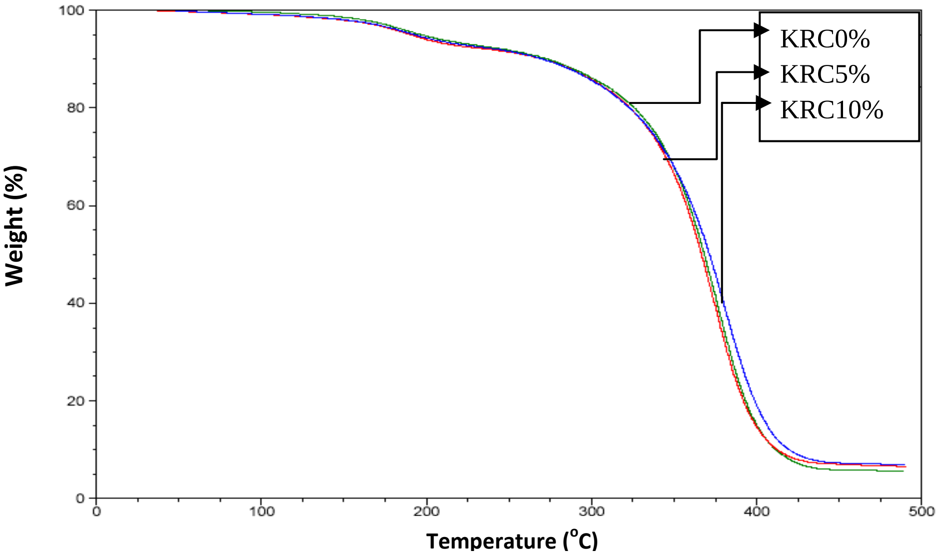

Figure 4.43: Effect of untreated Kerosene wood particle content on the thermal stability behaviour of pure polyester.

In order to investigate the effect of wood strength on the thermal stability of composites, various woods (Garjan, Kerosene and Gamari) were used. For untreated wood particle reinforced composites, effect of wood strength was found, Figs. 4.43, 4.44 and 4.45. This observation suggests that strength possibly does not control the high temperature stability of the wood particle. In the FTIR investigation, it has been found that all woods are composed of very similar type of fiber, which is responsible to control its thermal stability.

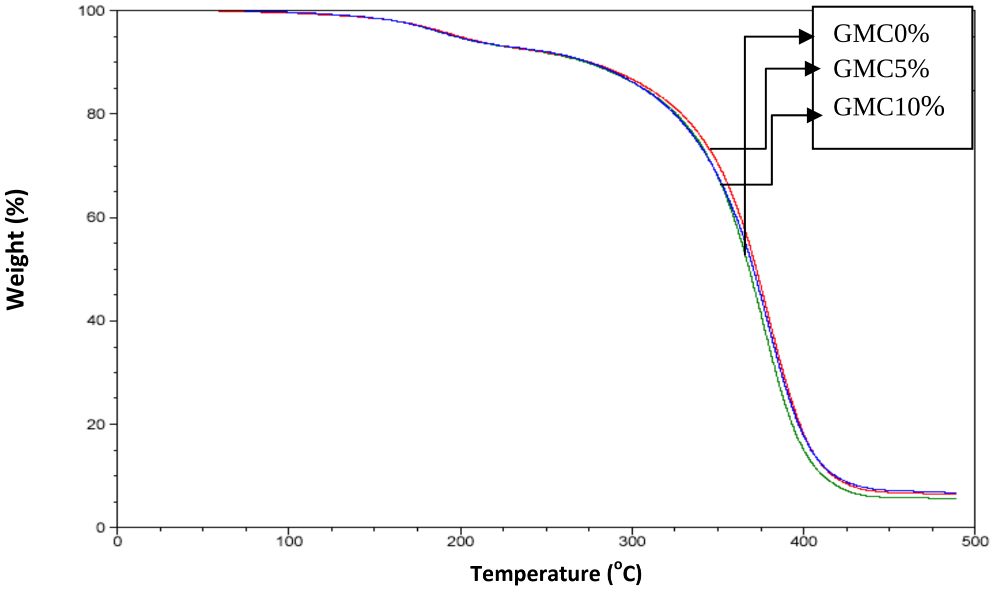

Figure 4.44: Effect of untreated Gamari wood particle content on the thermal stability behaviour of pure polyester.

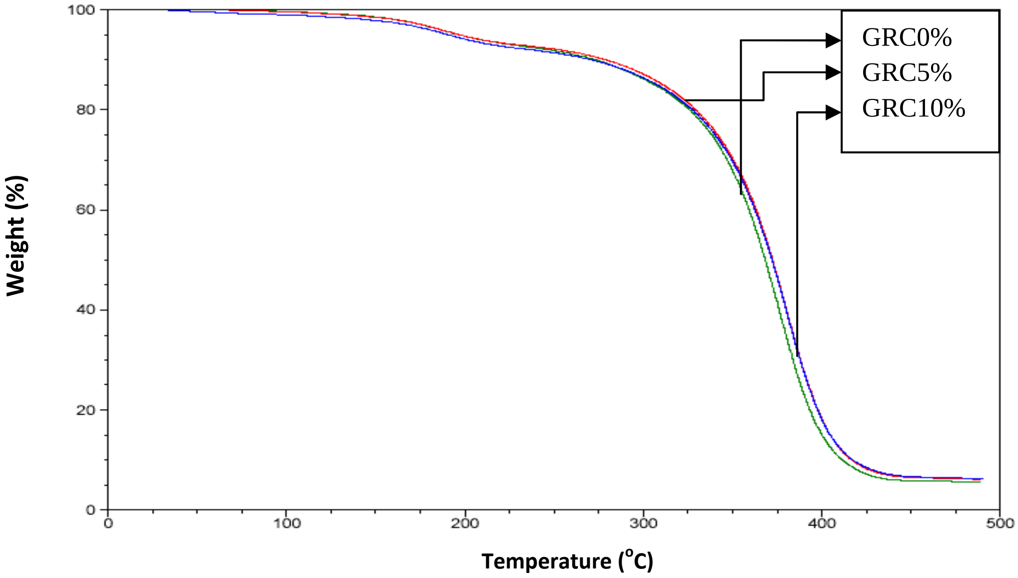

Figure 4.45: Effect of untreated Garjan wood particle content on the thermal stability behaviour of pure polyester.

4.8.2 Effect of Chemical Treatment of Wood Particle on Thermal Stability of Pure Polyester

The TGA curves of weight loss as a function of temperature for pure polyester and treated wood particle reinforced composites with varying amount of particle content are illustrated in Figs. 4.46-4.47. All composites exhibit thermal degradation and mass loss at an elevated temperature. From these figures it is revealed that chemically treated wood particle reinforced composites exhibited somewhat better thermal stability compared to that of either pure polyester or composites with untreated wood particle.

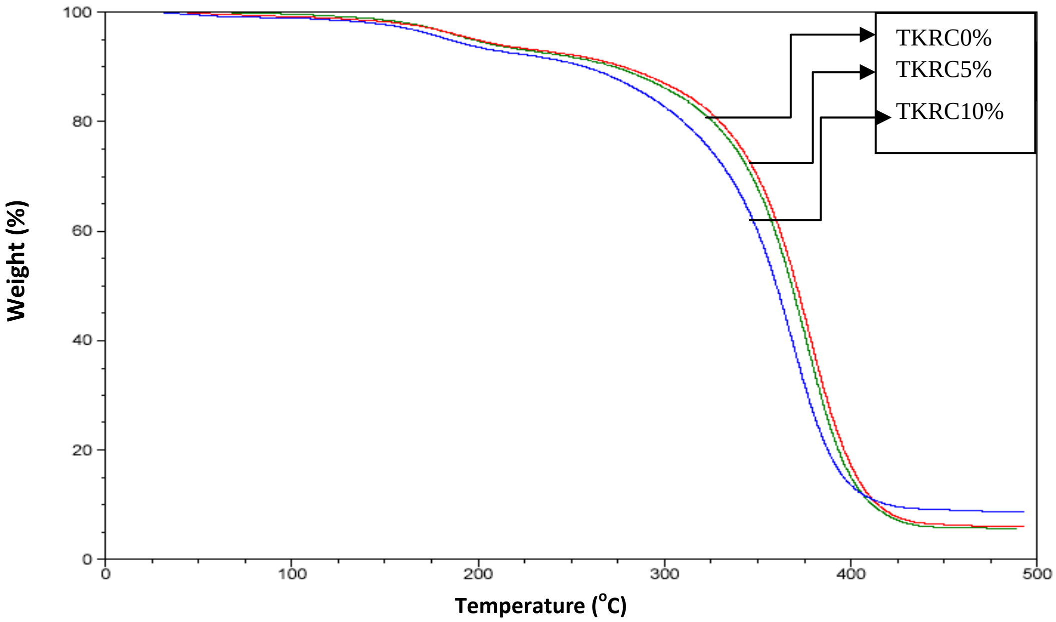

Figure 4.46: Effect of treated Kerosene wood particle content on the thermal stability behaviour of pure polyester.

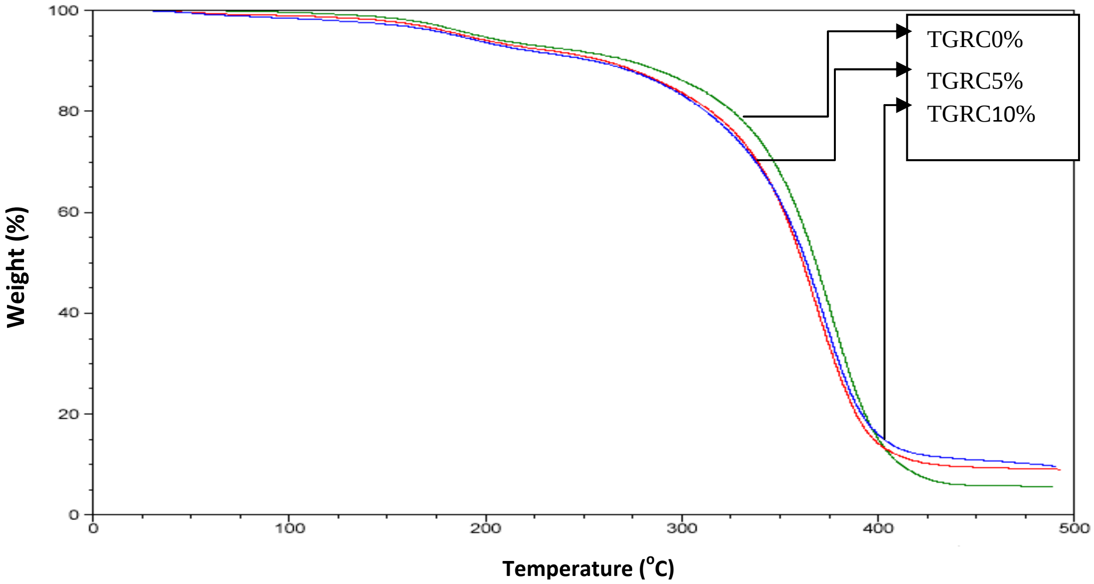

Figure 4.47: Effect of treated Garjan wood particle content on the thermal stability behaviour of pure polyester.

For all woods, chemical treatment increased the thermal stability of the composites. This observation will be very clear, when the thermal stability curves of untreated and treated wood particle reinforced composites (Figs. 4.43-4.47) are compared. The possible reason is that lignin might have higher volatilization tendency compared to that of pure wood fiber. During chemical treatment a certain portion of lignin was washed out, which caused the rest of wood to be more thermally stable.

4.9 WATER ABSORPTION TEST RESULTS

4.9.1 Effect of Wood Particle Content on Water Absorption Test Results

After calculation % of water uptake of the composites, % of water absorptions versus conditioning period were plotted and following curves have been obtained which are illustrated in Figs.4.48-4.53.

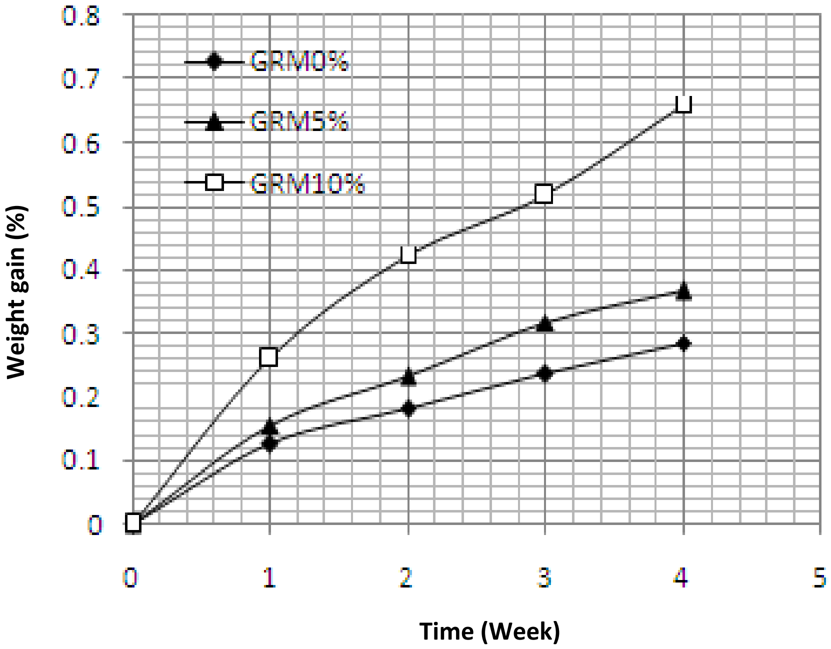

Figure 4.48: Time versus water absorption curves of untreated medium size Garjan wood particle reinforced polyester composites.

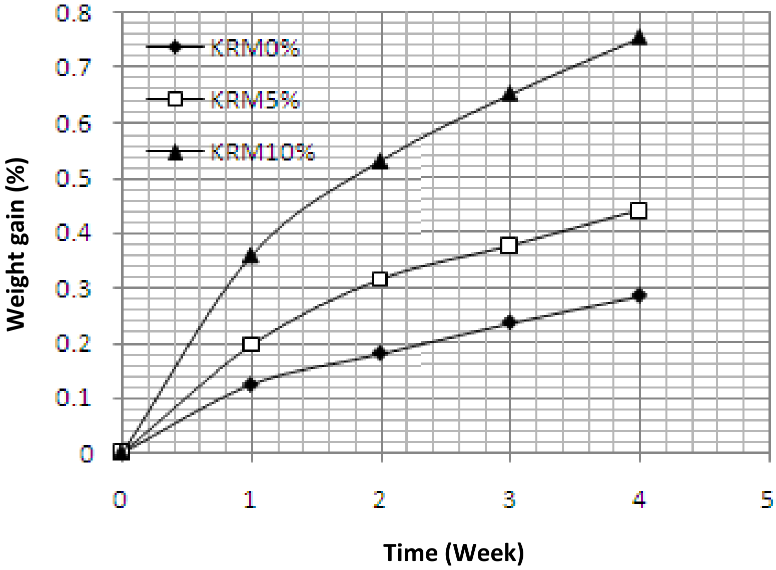

Figure 4.49: Time versus water absorption curves of untreated medium size Kerosene wood particle reinforced polyester composites.

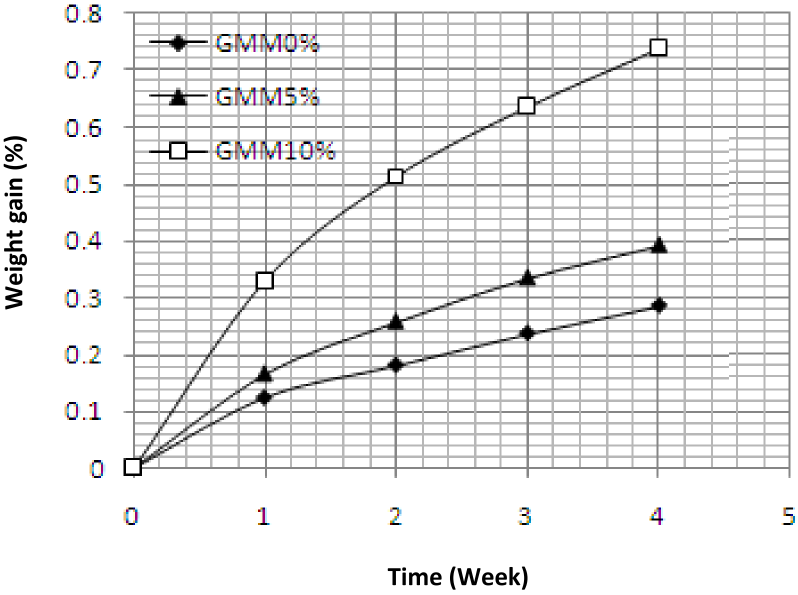

Figure 4.50: Time versus water absorption curves of untreated medium size Gamari wood particle reinforced polyester composites.

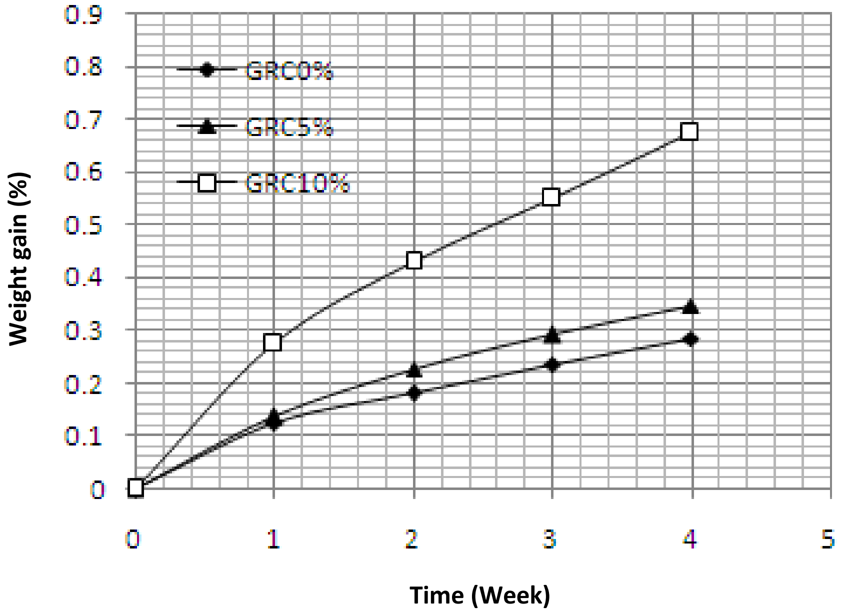

Figure 4.51: Time versus water absorption curves of untreated coarse size Garjan wood particle reinforced polyester composites.

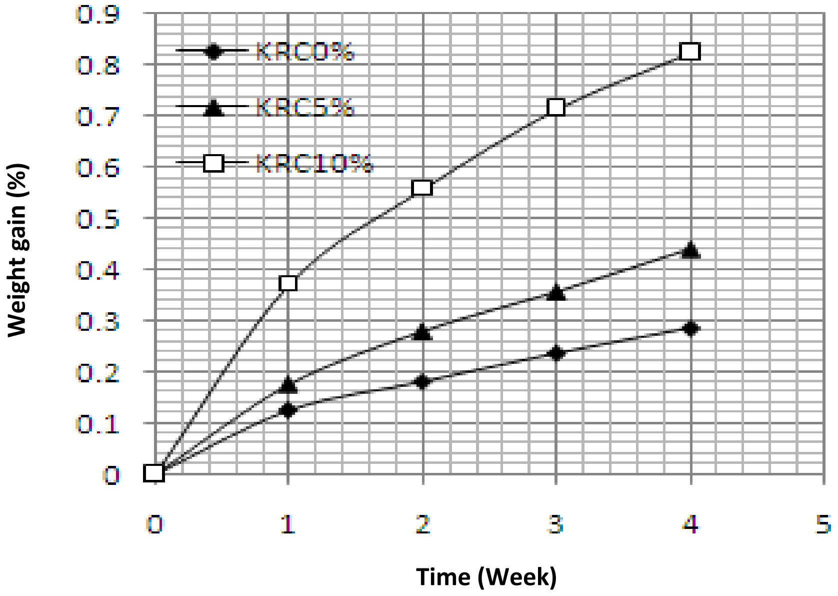

Figure 4.52: Time versus water absorption curves of untreated coarse size Kerosene wood particle reinforced polyester composites.

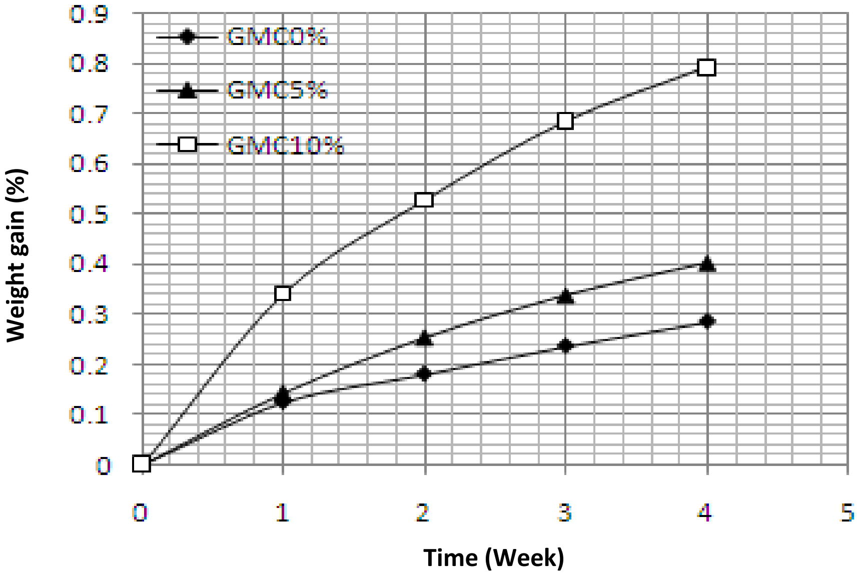

Figure 4.53: Time versus water absorption curves of untreated coarse size Gamari wood particle reinforced polyester composites.

From Figs. 4.48-4.53, it is clear that pure polyester has lower water absorption tendency and that with increase in the wood particle content increased the water absorption tendency. This observation is true for all woods and their particle size used for the present research. All natural fibers/particle have a tendency to absorb water and possess low wettability by hydrophobic resins due to presence of hydrophilic hydroxyl groups of cellulose, hemicellulose and lignin. Hemicellulose is mainly responsible for the moisture absorption in composites. Non-crystalline cellulose and lignin also play an important role in water uptake process. Water diffusion in polymeric composites has shown to be governed by the three mechanisms [57, 58]. The first involves diffusion of water molecules inside the micro gaps between polymer chains. The second involves capillary transport into gaps and finally flaws at the interfaces between fiber and matrix [59]. Absorption of water in composites causes swelling of fiber till the cell walls are saturated with water and beyond that water exists as free water in the void structure leading to composites delamination or void formation. Absorbed water causes weakening of interfacial adhesion and hydrolytic degradation of both matrix and fiber [60]. Cracking and blistering of fiber cause high water absorption, while degradation causes leaching of small molecules [61]. With increase in the wood particle content and time period, the total water intake also increased. So, the overall water absorption of the composites increased with wood particle content as well as with time period. The water absorption of this type of composites has been investigated by the team of Bismarck et al and Mosadeghzad et al and similar results have been found [62, 48].

4.9.2 Effect of Chemical Treatment of Wood Particle on Water Absorption Behaviours of Composites

In order to know the effect of chemical treatment on the water absorption of behaviours of the developed composites, chemically treated wood particle reinforced composite were also tested keeping them under water. The Time-Water Absorption curves of composites with chemically treated wood particle are shown in Figs. 4.54-4.56.

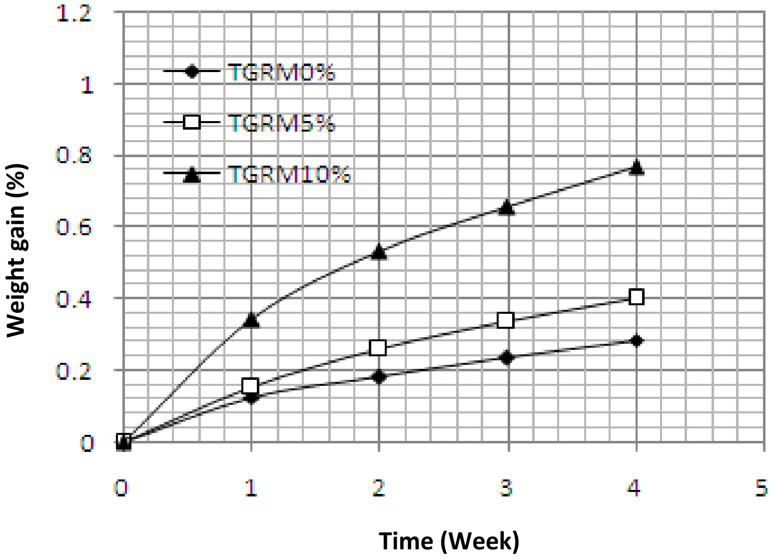

Figure 4.54: Time versus water absorption curves of treated medium size Garjan wood particle reinforced polyester composites.

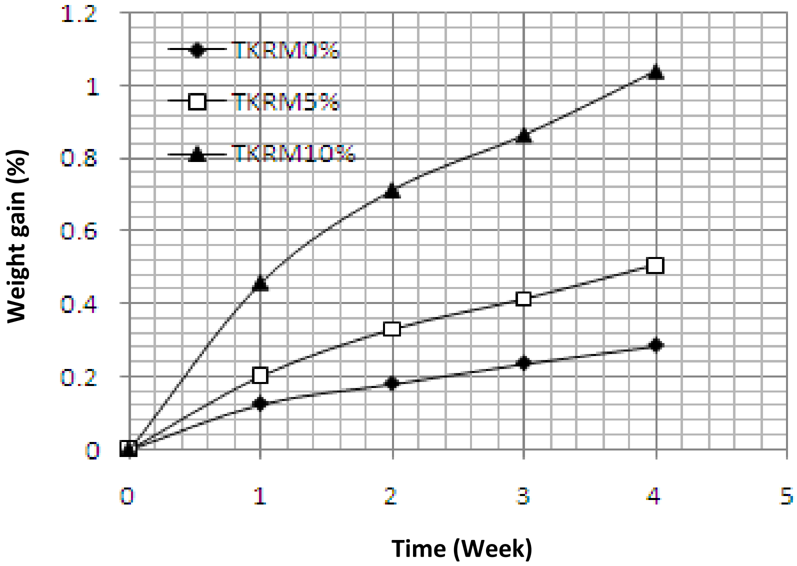

Figure 4.55: Time versus water absorption curves of treated medium size Kerosene wood particle reinforced polyester composites.

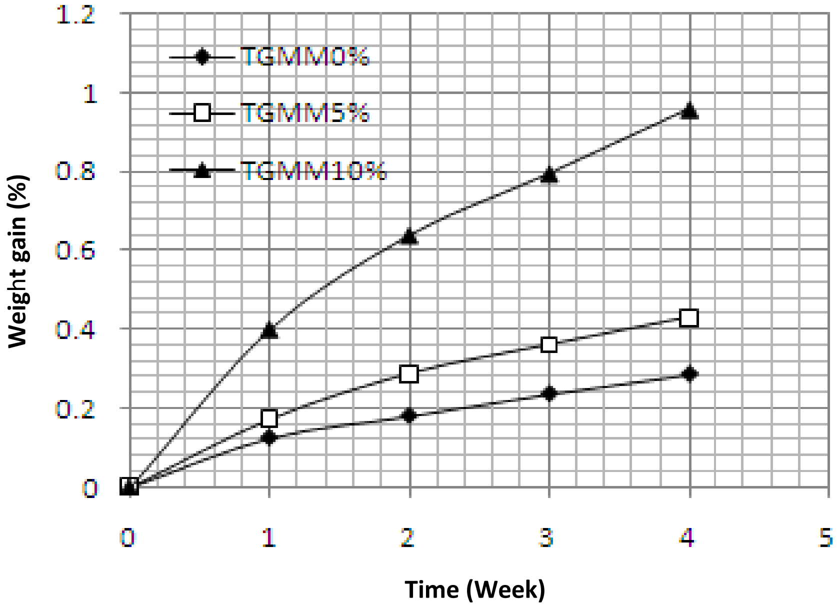

Figure 4.56: Time versus water absorption curves of treated medium size Gamari wood particle reinforced polyester composites.

From Figs. 4.54-4.56, it is clear that, for all wood particles, water absorption increased with both time period and wood particle content. The reason has already been discussed. Here initiative will be taken to discuss the effect of chemical treatment on the water absorption behaviour of the developed composites. From Figs. 4.48-4.56, it is clear that chemical treatment caused the composites to absorb more water for all wood composites. This means, chemical treatment degraded the composites in terms of water absorption. It has been mentioned that chemical treatment mainly removes the lignin portion of the as-received wood particle. In general, lignin behaves as glue in the wood fiber. During chemical treatment, the lignin portion of the wood is selectively washed out, which causes extra pores inside the wood particle. These newly created extra pores accommodate more water during the water absorption test. So, chemically treated wood particle reinforced composites showed relatively more water absorption. The water absorption of this type of composites with chemically treated wood dust has been investigated and similar results have been found by the team Mosadeghzad et al and Andrzej et al [48, 63].

4.9.3 Effect of Types of Woods Particle on Water Absorption Behaviour of Composites

In this research work three different types of woods as Garjan, Kerosene and Gamari having high, medium and low strength and density respectively were used. The experimental results revealed that high density wood exhibited higher strength. This density or strength of the woods also influenced the water absorption behaviour of the developed composites, which is clear from Figs. 4.57-4.62.

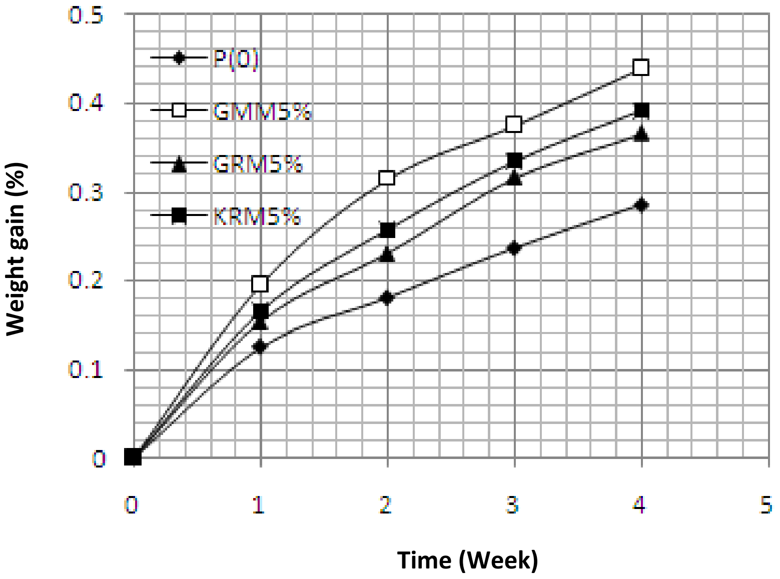

Figure 4.57: Effects of types of wood particle on water absorption behaviour of medium size untreated 5% wood polyester composites.

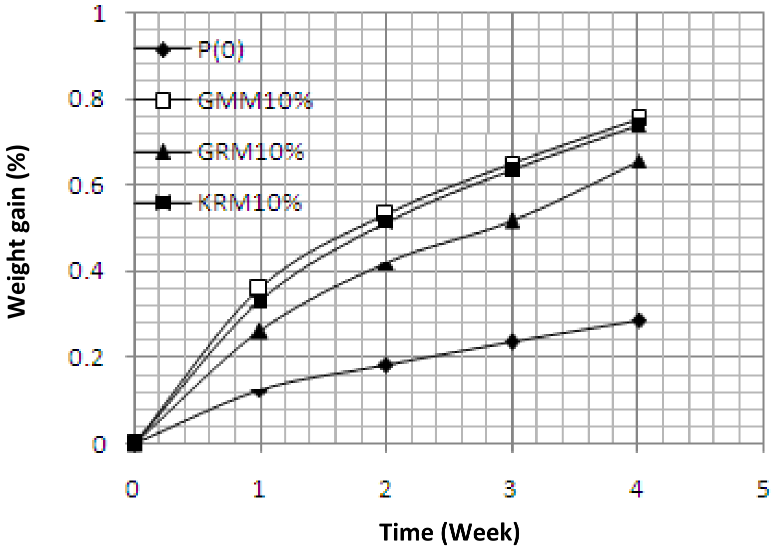

Figure 4.58: Effects of types of wood particle on water absorption behaviour of medium size untreated 10% wood polyester composites.

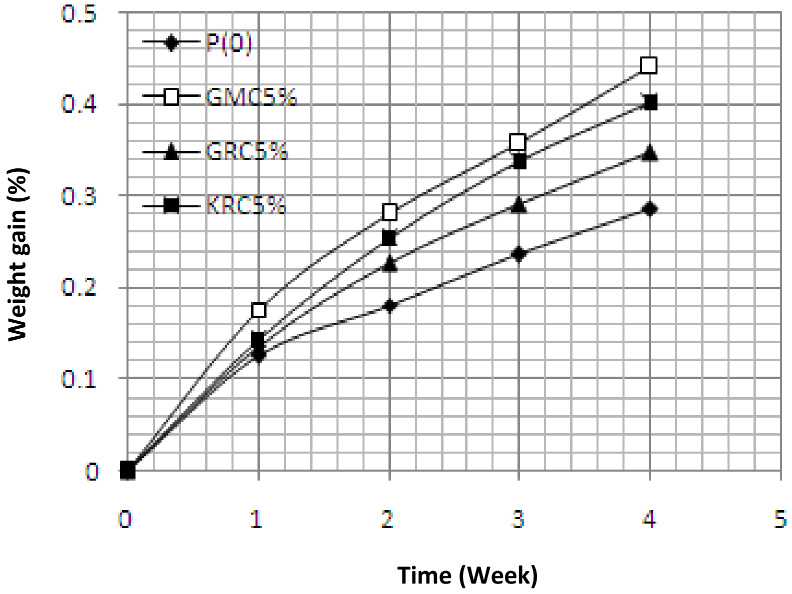

Figure 4.59: Effects of types of wood particle on water absorption behaviour of coarse size untreated 5% wood polyester composites.

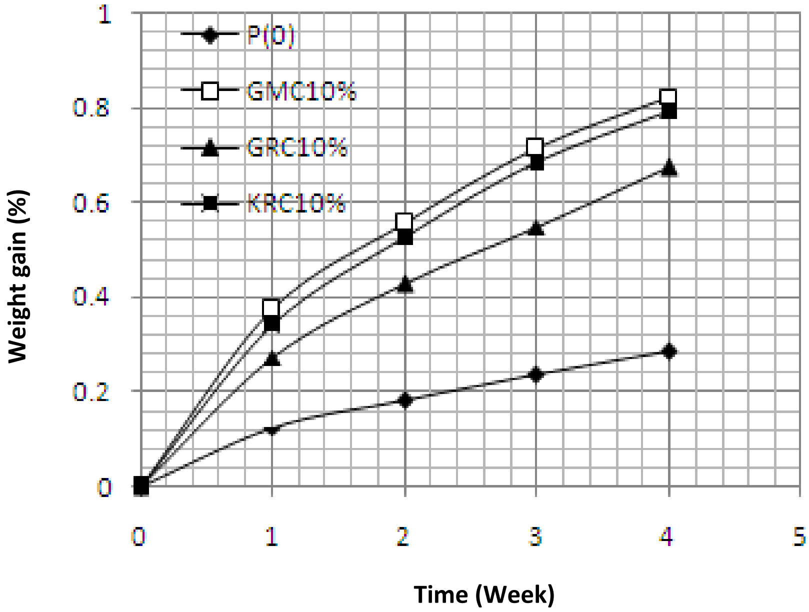

Figure 4.60: Effects of types of wood particle on water absorption behaviour of coarse size untreated 10% wood polyester composites.

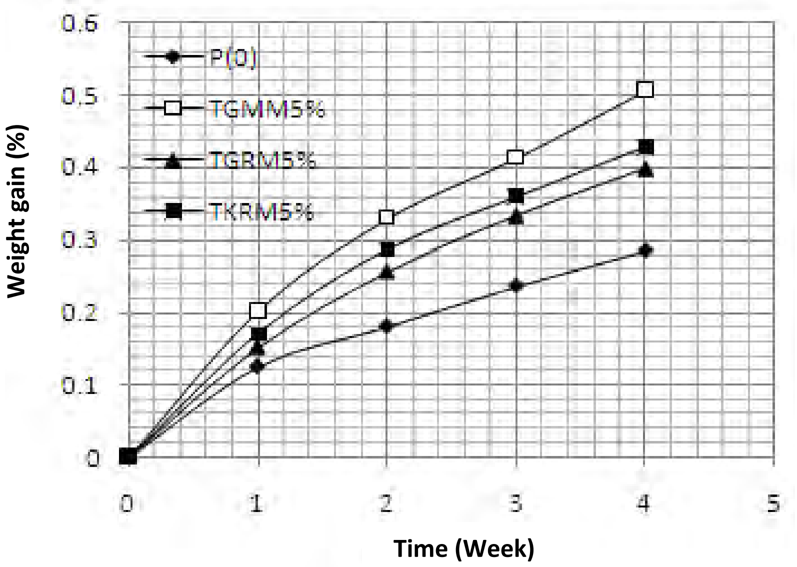

Figure 4.61: Effects of types of wood particle on water absorption behaviour of medium size treated 5% wood polyester composites.

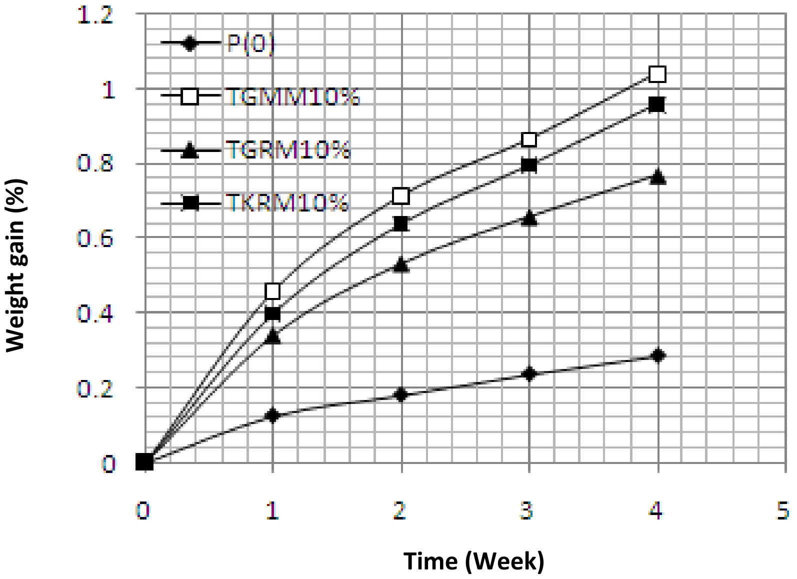

Figure 4.62: Effects of types of wood particle on water absorption behaviour of medium size treated 10% wood polyester composites.

From Figs. 4.57-4.62, it is clear that, for all cases, pure polyester showed the lowest level water absorption. Another observation is that composites reinforced with Garjan wood particle, irrespective their content and sizes, showed the lowest water absorption among all other composites. On the other hand, Gamari wood (lowest strength and density) particle showed the highest level of water absorption. In general highly dense wood contains lower level of porosity.

As inherent porosity seriously interfere the degree of water absorption, so wood having lowest level of density resulted higher water absorption.

Chapter 5

CONCLUTIONS AND RECOMMENDATION FOR FUTURE WORK

5.1 CONCLUSIONS

In this study wood saw dust of three different types of woods as Garjan, Kerosene and Gamari having significantly different tensile strengths (high, medium and low strengths) were used to develop polyester based composites. In order to know the effects of saw dust particle size, medium and coarse wood dust were used. Chemical treatment changes the physico-chemical properties of the wood saw dust. As a result, both as-received and chemically treated dust particle were used. After moulding of the composites, their tensile strength, breakdown voltage and thermal conductivity were measured for observation of the composite behaviours. Compositional analysis was carried out by Fourier Transform Infrared Radiation (FTIR) spectrometer. Thermal degradation was investigated by Thermo-Gravimetric Analysis (TGA). Water absorption of composites was also performed. Before moulding of the composites, some physical properties (density, porosity, etc.) of the saw dust particle were also studied. The experimental investigation of wood saw dust particle reinforced polyester based composites leads to the following conclusions.

- Among three woods Garjan, Kerosene and Gamari woods resulted the highest, medium and the lowest tensile strength respectively. For the apparent and true densities of these woods similar trends like tensile strengths have been found. In the case of Garjan, porosity of the coarsest size was observed, whereas it was the smallest size for Gamari wood.
- For all cases, tensile strengths of composites were found to decrease with increase in the wood particle contents. Compared to medium size particle, coarse saw dust particle was found to deteriorate the composites more.
- Woods of various tensile strengths were used to develop the wood particle reinforced composites. In this case, saw dust particle of higher strength wood resulted relatively higher tensile strength of the developed composites.

- For pure polyester, no consistent result has been found for percentages of elongation. However, in the case of the developed composites, percentages of elongation were found to decrease with increase in the proportions of wood particle.
- Polyester based composites reinforced with chemically treated wood particle resulted relatively higher strength, which have been found to be true for all types of wood particle and particle size.
- The fracture surfaces study of wood saw dust reinforced polyester composite after the tensile test has been done. From this study it has been concluded that fracture in the pure polyester sample was started from some foreign dust particles. However, for wood composites, crack was found to initiate either from the wood particle stacking or air bubbles formed during the moulding process.
- With increase in the wood particle contents more wood particle stacking and air bubbles were found.
- For the breakdown voltage of pure wood, wood strength or density has not found to influence. However, size of the in the individual pore rather than total volume fraction of pore was found to influence the breakdown voltage.
- Similar to tensile strength, breakdown voltage was found to decrease with increase in the proportion of the wood particle. Similar trend of decrease in the tensile strength was observed for both the treated and untreated wood particle reinforced composites. However, chemical treatment has been found to improve the breakdown voltages of composites of all wood particles.
- FTIR results suggested that chemical treatment removed part of the lignin of the wood particle.
- Pure polyester has been found to have lower thermal conductivity compared to that of any wood and that wood strength or true density has been found to influence the thermal conductivity of the as-received pure very mildly.
- With increase in the dust particle content, thermal conductivities of all composites have been found to increase for both the untreated and chemically treated wood particle.

- From the FTIR graphs, it is clear that no reactions are occurred between reinforced wood saw dust and polyester.
- Thermo-Gravimetric Analysis (TGA) was used to monitor the decomposition of composites as a function of increasing temperature. It has been seen that wood particle has no significant influence on the thermal stability of the pure polyester up to 350^oC, however, the thermal stability of the composite increased slightly. This high temperature thermal stability further improved with higher proportion of wood particle.
- Chemical treatment improved the thermal stability of the composite. In this case improved thermal stability has been found from around 150^oC.
- Water absorption of both pure polyester and wood particle reinforced composites has been found to increase with increase in time.
- Either increase in the proportion of wood particle content in the composite or particle size of the wood dust increased the water absorption.
- Chemical treatment improved almost all properties. However, in the case of water absorption, it has been found to reduce the water absorption resistance of the composites, which indirectly suggest that lignin has better water absorption resistance than that of wood fiber.

5.2 RECOMMENDATION FOR FUTURE WORK

All objectives were fulfilled in the study, which were outlined in the thesis specification. The objective outlines a comparison of mechanical, electrical and thermal properties with varying amounts and size of untreated and treated saw dust reinforced composites. There is a very wide scope for the future researchers to explore this area of research. The following recommendations are suggested for future work.

- The cellulose, hemicellulose and lignin contents of woods should be quantitatively analyzed because the properties of wood and its composites depend on these components.
- Initiative might be taken to treat the wood particles with fire retardant (NH_4) to reduce the degree of flammability of composites products under service.

- Higher percentage of wood dust could be incorporated through modern machine. To get better composites, hot press molding might be used. Hybrid could be done for enhancing the mechanical properties and dimensional stability of the composite.
- In the case of practical issue (such switch board and circuit board of electronics devices), dielectric constant and dielectric loss should be measured of these composites.

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Appendix 1

Table A-1: Tensile strength of untreated wood saw dust reinforced polyester matrix composites

Name of the composites	% of particles	Tensile strength (MPa)
Pure polyester, P(0)	0	42±0.7
Kerosene medium (KRM)	5	32±0.5
Kerosene medium (KRM)	10	26±1.5
Kerosene coarse (KRC)	5	26±1.7
Kerosene coarse (KRC)	10	22±1.2
Garjan medium (GRM)	5	32±0.84
Garjan medium (GRM)	10	28±0.7
Garjan coarse (GRC)	5	27±1.7
Garjan coarse (GRC)	10	24±0.5
Gamari medium (GMM)	5	30±1.2
Gamari medium (GMM)	10	25±0.5
Gamari coarse (GMC)	5	24.5±1.7
Gamari coarse (GMC)	10	21±0.25

Table A-2: Tensile strength of treated wood saw dust reinforced polyester matrix composites

Name of the composites	% of particles	Tensile strength (MPa)
Pure polyester, P(0)	0	42±0.7
Treated Kerosene medium (TKRM)	5	33.5±0.56
Treated Kerosene medium (TKRM)	10	31±0.79
Treated Kerosene coarse (TKRC)	5	28±0.8
Treated Kerosene coarse (TKRC)	10	24±1.1
Treated Garjan medium (TGRM)	5	34±.082
Treated Garjan medium (TGRM)	10	32±0.4
Treated Garjan coarse (TGRC)	5	31±0.48
Treated Garjan coarse (TGRC)	10	29±0.81
Treated Gamari medium (TGMM)	5	32±0.47
Treated Gamari medium (TGMM)	10	28±1.11
Treated Gamari coarse (TGMC)	5	25±1.04
Treated Gamari coarse (TGMC)	10	22±1.05

Table A-3: Breakdown voltage of untreated wood saw dust reinforced polyester matrix composites

Name of the composites	% of particles	Breakdown voltage (KV-cm)
Pure polyester, P(0)	0	70±0.57
Kerosene medium (KRM)	5	61±1.0
Kerosene medium (KRM	10	57±0.1
Kerosene coarse (KRC)	5	57±0.58
Kerosene coarse (KRC)	10	51±0.5
Garjan medium (GRM)	5	64±0.57
Garjan medium (GRM)	10	56±0.1
Garjan coarse (GRC)	5	62±0.58
Garjan coarse (GRC)	10	53±0.58
Gamari medium (GMM)	5	65±1.0
Gamari medium (GMM)	10	58±1.5
Gamari coarse (GMC)	5	65±1.5
Gamari coarse (GMC)	10	54±1.15

Table A-4: Tensile strength of untreated wood saw dust reinforced polyester matrix composites

Name of the composites	% of particles	Breakdown voltage (KV-cm)
Pure polyester, P(0)	0	70±0.57
Treated Kerosene medium (TKRM)	5	75±1.15
Treated Kerosene medium (TKRM	10	64±1.15
Treated Kerosene coarse (TKRC)	5	74±0.58
Treated Kerosene coarse (TKRC)	10	60±1.5
Treated Garjan medium (TGRM)	5	72±0.58
Treated Garjan medium (TGRM)	10	59±0.5
Treated Garjan coarse (TGRC)	5	71±1.15
Treated Garjan coarse (TGRC)	10	60±1.52
Treated Gamari medium (TGMM)	5	70±1.15
Treated Gamari medium (TGMM)	10	60±1.5
Treated Gamari coarse (TGMC)	5	70±1.15
Treated Gamari coarse (TGMC)	10	58±1.0

Appendix 2

List of publication

1. M. Faruk Hossain[1] and M.A. Islam[2], "TENSILE AND ELECTRICAL PROPERTIES OF WOOD SAW DUST REINFORCED POLYMER MATRIX COMPOSITES", Published in the Proceedings of the Conference on Engineering Research, Innovation and Education (CERIE) 2011, 11-13 January 2011, Sylhet, Bangladesh.