INVESTIGATIONS INTO CHEMICAL TREATMENT OF MEDIUM DENSITY COIR FIBRE BOARDS FOR ENHANCING FIRE RETARDANT PROPERTIES

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INVESTIGATIONS INTO CHEMICAL TREATMENT OF MEDIUM DENSITY COIR FIBRE BOARDS FOR ENHANCING FIRE RETARDANT PROPERTIES

Ph. D. Thesis under the Faculty of Engineering

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THESIS CERTIFICATE

Certified that the work presented in this Ph.D. thesis entitled "Investigations into Chemical treatment of Medium Density Coir Fibre Boards for enhancing Fire Retardant properties" is based on the authentic record of research done by Shri Rejeesh C. R. (Reg. No: 4723) under my guidance in the School of Engineering, Cochin University of Science and Technology, Kochi-682022 and has not been included in any other thesis submitted for the award of any degree.All the relevant corrections and modifications suggested by the audience during the pre-synopsis seminar and recommended by the Doctoral Committee has been incorporated in the thesis.

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DECLARATION

I hereby declare that the work presented in the thesis entitled "Investigations into chemical treatment of medium density coir fibre boards for enhancing fire retardant properties" is a bonafide work carried out by me under the supervision and guidance of Dr. K. K. Saju, Professor, Division of Mechanical Engineering, School of Engineering, Cochin University of Science and Technology, Kochi, Kerala, India as Research guide. No part of this thesis has been presented for the award of any other degree from any other institution.

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- - - Dedicated to my family - - -

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ABSTRACT

In recent years, there has been an increasing interest in finding new applications for coir fibre reinforced composites that are traditionally used for making ropes, mattress, mats, and carpets. Coir fibre is a natural fibre extracted from the husk of coconut and medium density fibre boards made from coir are being investigated worldwide for their ability to substitute wood. Coir fibre is estimated as a promising reinforcement to be used in composite boards on account of its economy, low density, high specific strength and elastic modulus, non toxicity, ease of availability, renewability and lower energy requirement for processing.

Increasing popularity and use of wood substitutes for building and construction sector makes coir fibre boards a possible construction material as a substitute to engineered wood products like plywood. Since the decreased ability of a material to catch fire accounts to better safety, especially in residential buildings, it is a preferable choice when the materials are treated with fire retardant (FR) chemicals. Fire retardant properties of the panel boards made from coir fibre need to be enhanced for its wider acceptance.

In places where severe degradation conditions are prevailing, it is also desirable to have better resistance to degradation along with fire resistance. Boron compounds are extensively used in chemical treatments for wood preservation. A large variety of fire-retardant chemicals and their treatment methods are being studied including the combination of Borax (BX) and Boric acid (BA). Coir fibre based medium density panel boards were subjected to treatment with a solution of BX and BA for enhancing their fire-retardant properties.

The investigations suggested great improvements to thermal properties of medium density coir board samples when chemically treated with a preservative solution containing a combination of boric acid and borax. Chemically treated medium density coir board samples were found to be thermally more stable over untreated samples throughout the range of temperatures. The Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) analysis revealed that the first decomposition temperature and crystallization enthalpy has increased after chemical treatment. The derivative weight loss of FR treated samples were found to be decreased which shows that the treated samples offered better resistance when subjected to heat. The research suggests that the method of chemical treatment using boron compounds in medium density coir board samples could increase the thermal stability.

The investigations included chemical treatment of fibre boards with the aqueous solution containing preservative boron compounds, and subjecting to fire resistance tests like flammability test, flame penetration test, rate of burning test and resistance to flame spread test. The fire retardant treated (FRT) samples showed significant improvements in all the four tests and gave reduced flammability, lesser flame penetration, reduced rate of burning and resistance to spread of flame.

The test for limiting oxygen index showed reduced values of ignitability and analysing all these results suggests that the method of

chemical treatment using boron compounds with medium density coir fibre board samples could also increase the flame retardation properties to a great extent.

Investigations on the mechanical behaviour of coir board samples were made to compare the properties of FRT coir fibre boards with untreated coir boards. Tensile tests confirmed that boron treated coir board samples exhibited a higher maximum tensile strength than untreated samples. A similar behaviour was observed for flexural strength wherein, treated samples exhibited higher flexural strength.

Evaluation of the flame resistance of chemically treated coir composite panel boards for different weight percentage of borates in the solution were also done. An optimum composition of water-borne preservative solution was identified which gave better results out of the selected samples under investigation. Regression analysis successfully predicted the values of flammability, flame penetration time, rate of burning and resistance to flame spread with respect to the weight percentage of FR boron present in the solution for chemical treatment.

The results from present research work indicates that FR treatment of coir fibre boards with a combination of BA and BX enhances the fire-retardant properties of coir fibre based panel boards and could increase the use of coir fibre boards as substitute for wood based applications. These results further encourages the use of coir fibre boards for more varied applications that involves stringent fire safety norms. The research also suggests the possibility of developing a new class of fire proof coir fibre based panel boards that can cater to a wide range of applications.

The major contribution of this research includes the following,

- Development of a new fire retardant fibre board made from coir fibre.
- Development of a simple and economically feasible method to impart fire resistance to coir fibre boards.
- Assessment of fire resistance characteristics and thermo-mechanical properties of coir fibre boards after fire retardant treatment.

KEYWORDS:

Medium density coir fibre boards; Fire retardant (FR); Borax; Boric acid; Fire retardant treatment (FRT); Flame resistance tests; Limiting oxygen index; Mechanical properties; Thermal stability; Regression analysis.

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ABBREVIATIONS

AS	Ammonium Sulphate
BA	Boric Acid
BX	Borax
MF	Melamine Formaldehyde
PF	Phenol Formaldehyde
FR	Fire Retardant
MC	Moisture Content
FRT	Fire Retardant Treated
LOI	Limiting Oxygen Index
LPG	Liquefied Petroleum Gas
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
FTIR	Fourier Transform Infrared Spectroscopy
GC-FTIR	Gas Chromatography - Fourier Transform Infrared Spectroscopy
BBP	Butyl Benzyl Phthalate
PVC	Poly Vinyl Chloride
FRP	Fibre Reinforced Polymer
FSP	Fibre Saturation Point
MAP	Mono-Ammonium Phosphate

DAP	Di-Ammonium Phosphate
DOT	Disodium Octoborate Tetrahydrate
ATH	Alumina Trihydrate
LVL	Laminated Veneer Lumber
MOR	Modulus of Rupture
BIS	Bureau of Indian Standards
MDF	Medium Density Fibre
SEM	Scanning Electron Microscope
DOE	Design and analysis of Experiments
ANOVA	Analysis of Variance
CRD	Completely Randomized Design
RCBD	Randomized Complete Block Design
RECP	Rolled Erosion Control products
RHPB	Rice Husk Particle Boards
CICT	Central Institute of Coir Technology
MSME	Ministry of Micro, Small & Medium Enterprises

NOTATION

\$	Dollar
μm	Micrometer
R^2	Coefficient of determination
F	F value as the variance ratio
p	probability value
α	significance level
n'	Replication
ΔH	Crystallization Enthalpy in J/g
ρ	Density of coir fibre board samples
CV_{gas}	Calorific Value in kJ/m^3
Q	Rate of Heat Energy added, in kJ/s
V_g	The volume of gas burnt in l/s
m	Mass flow rate in kg/s
ΔT	The change in temperature of water in ^{0}K
C_p	The specific heat of water in kJ/kg-K
n	Limiting Oxygen Index values in %
O	Oxygen concentration in the inflow gases in %
N	Nitrogen concentration in the inflow gases in %
M_i	Initial mass of sample in g
M_o	Oven-dry mass of sample in g

L	Length of the sample in cm
w	Width of the sample in cm
t	Thickness of the sample in cm
E	modulus of elasticity in MPa
k	Thermal conductivity in W/m-K
R	Thermal resistance in m^2.K/W
L	Thickness of the specimen in m
T	Temperature in K
q	Heat flow rate in W/m^2

CHAPTER 1

INTRODUCTION

Researchers have been interested in use of natural plant fibres for engineering applications, from ancient times, wherein papyrus was used to retain soil, to the modern era where cotton fibre is used in foam insulation. During the last few years, biomass based materials derived from different kinds of natural resources had become a topic of interest because of their ease in availability and renewable nature [1, 2]. Today, the interest in natural plant fibres has soared with global demand for more efficient, less expensive, lightweight and renewable green products. This global demand also provides a unique opportunity to several developing nations, as a large percentage of the world's natural plant fibres are being grown there [3].

1.1 Introduction to natural fibres

Natural fibres are eco-friendly, renewable materials which are biodegradable as well as economical. This new generation reinforcement material supplements for polymer based materials. Fibres from plants such as cotton, hemp, jute, sisal, pineapple, ramie, bamboo, banana, as well as wood and seeds of flax are used as the reinforcement in polymer matrix composites. Factors such as ease of availability, lower density, high strength to weight ratio, lower price and competitive mechanical properties, makes them an attractive reinforcement to substitute glass, carbon and other man-made fibres. Biomass based materials can be easily degraded through the enzymatic action of bacteria, fungi, algae and other living organisms, into carbon dioxide, methane, water, and/or various inorganic compounds. By combining the synergistic effect of different properties from bio based polymers, the properties of the resulting bio composite materials can be enormously improved [4–8].

1.2 Classification of Natural Fibres

There are a wide variety of cellulose fibres like wood fibres and a variety of agro-based fibres derived from bast, stems, leaves, stalks and seed hairs of different plant species that can be used to reinforce thermoplastics and other matrices. These fibres are extracted from renewable resources and are found to be cultivated in abundance in India and various parts of the world [4–6]. Other major sources include recycling agro-based products such as paper or veneer and agricultural residues like rice husk from rice processing plants [6].

Natural fibres can be classified according to their origin,as
(a) Vegetable or cellulose base fibres like cotton, jute and coir;
(b) Animal, or protein base fibres like wool, mohair and silk;
(c) Mineral class fibres like wollastonite and asbestos.

Cellulose based vegetable fibres are further classified on the basis of the part of the plant from which they are derived. Figure 1.1 represents the classification of different types of vegetable fibres and their sources [3].

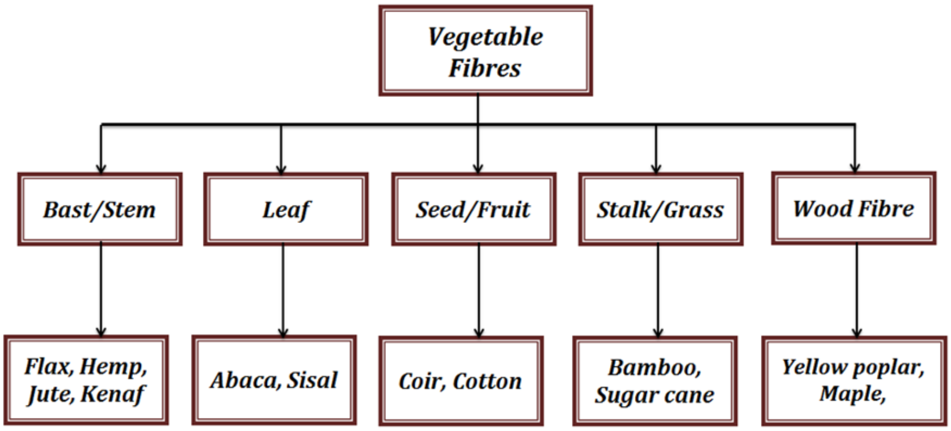


Figure 1.1: Classification of some natural plant fibres

(1) Bast fibres

These fibres (bundles) come from the inner bark (bast) of the stems of dicotyledonous plants [7, 8]. Flax, hemp, jute, and kenaf are all examples of bast fibres extracted from the inner bast tissue of plant stems made up of overlapping cells.

(2) Leaf fibres

Leaf fibres run lengthwise through the leaves as a part of the fibro-vascular system of monocotyledonous plants such as sisal, henequen and abaca [7, 8]. These fibres, are referred to as hard fibres, and are most commonly employed as reinforcing agents in plastics [4].

(3) Seed and fruit fibres

They are extracted from the seed-hairs and flosses of plants. Cotton and coir are examples of fibres originating as hair borne on the seeds or inner walls of the fruit, where each fibre consists of a single, long, narrow cell [4, 8].

(4) Grasses and reeds

These fibres come from the stems of monocotyledonous plants such as bamboo and sugar cane (bagasse) [7, 8]. Both types of fibres can be used to reinforce plastics [4].

(5) Wood fibres

These fibres come from the xylem of angiosperm (hardwood) and gymnosperm (softwood) trees. Examples of such fibres includes those extracted from trees like maple, yellow poplar and spruce.

1.2.1 Sources of cellulose based natural fibres

Natural fibres of mineral origin largely find application in industrial insulation purposes, whereas animal fibres are mostly used for domestic insulation, clothing and carpets. The natural fibres which are significant and most beneficial from an environmental point of view are those of vegetable origin. Among vegetable fibres, pure cotton is made up of α-cellulose and is costly. Lignocellulosic fibres are composed of lignin and cellulose and they are found to be cheap and strong. Lignin is a thermoplastic natural resin whereas cellulose is a reinforcing fibre, thus lignocellulosic fibres are regarded as a self-reinforcing composite fibre [9].

Cellulose based natural fibres are preferable in engineering applications due to their enhanced strength, elongation and durability when compared to mineral or animal fibres. They are classified based on the origin of fibre from a part of the plant. For example, bast/stem or soft fibres reinforce the stems of dicotyledonous plants. Dicotyledonous plants are flowering plants that typically have tap root systems and leaves with net venation (arrangement of the veins). Bast/stem fibres generally have higher tensile strength than other vegetable fibres. Leaf or hard fibres reinforce the leaves of monocotyledonous plants. Monocotyledonous plants are flowering plants that typically have root systems that do not contain a main root and have leaves with parallel venation. Seed/fruit fibres protect the seeds and fruits of plants while Stalk fibres include straws and grasses [10]. Figure 1.2 shows the pictures of plant sources of different natural fibres.

(a) Cotton plant (b) Sisal plant (c) Jute plant

(d) Coconut tree (e) Flax plant (f) Sugarcane

(g) Hemp field (h) Kenaf plant (i) Bamboo grass

Figure 1.2: Images of plant sources of some natural fibres

1.2.2 Profile of common cellulosic natural fibres

Plants that produce natural fibres are classified into primary and secondary in terms of utilization. When plants are cultivated primarily for their fibre content, they are classified as a primary plant, while fibres from secondary plants are mostly treated as by-products after some other primary utilization.

Fibres like Jute, hemp, kenaf, sisal and cotton are thus categorized as primary plants, while pineapple, cereal stalks, agave, oil palm and coir are considered as secondary plants [11]. Figure 1.3 shows some of the common natural plant fibres which are extensively cultivated and traded in the world.

(a) Coir fibre (b) Cotton fibre (c) Flax fibre

(d) Hemp fibre (e) Jute fibre (f) Sisal fibre

Figure 1.3: Different natural plant fibres

(1) Coir fibre

Coir fibre is extracted from the tissues surrounding the seeds of coconut palm *(Cocos nucifera)*, which is grown extensively throughout the tropical land. The two types of fibre are: the brown fibre, obtained from mature coconuts, and finer white fibre, extracted from immature green coconuts after soaking for up to 10 months. Coir fibres measure up to 35 cm in length and have a diameter of 12-25μm.

Among vegetable fibres, coir has one of the highest concentrations of lignin, which makes it stronger, but is found to be less flexible than cotton and thus unsuitable for dyeing. The tensile strength of coir is low when compared to abaca, but it has good resistance to microbial action and salt water damage [11–13].

(2) Cotton fibre

The cotton fibre grows in the seed of a variety of plants of the genus *Gossypium*. Out of the four cotton species cultivated for fibre, the most important are *G. hirsutum*, originated from Mexico and accounts for 90% of the world's cotton production, and *G. barbadense*, of Peruvian origin, that accounts for another 5%. The world average of cotton yields is around 800 kg per hectare.

Cotton is almost pure cellulose, with softness and breathability, which makes it the world's most popular natural fibre. Fibre length varies from 10 to 65 mm, and diameter from 11 to 22 μm. It readily absorbs moisture, that makes cotton clothes to be a comfortable wear in hot weather [11–13].

(3) Flax fibre

Flax fibres obtained from the stems of the plant *Linum usitatissimum* are used mainly to make linen fabric. The plant has been used for fibre production since prehistoric times. It grows best at northern temperate latitudes, where moderately moist summers yield fine, strong but silky flax.

In Europe, a hectare of flax plants typically yields 1.5 to 3.5 tonnes of fibre. Similar to cotton, flax fibre is also a cellulose polymer, but its structure is more crystalline, making it stronger, crisper and stiffer to handle, and more easily wrinkled. Flax fibre ranges in length up to 90 cm, and average 12 to 16 μm in diameter. They absorb and release water quickly, that makes linen also a comfortable wear in hot weather [11–13].

(4) Hemp fibre

Hemp fibre is obtained from the bast of the plant *Cannabis sativa L.* (The L was included in the name in honour of the botanist Carl Linnaeus.) which grows easily to a height of 4 m and captures large quantities of carbon. The resemblance of the plant with marijuana *(Cannabis sativa)* has posed restrictions for production of hemp in some places.

The optimum yield of hemp fibre is more than 2 tonnes per hectare, while average yields are around 650 kg. Long, strong and durable, hemp fibres have diameters ranging from 16 to 50 μm. Hemp fibre conducts heat, dyes well, resists mildew, blocks ultraviolet light and has natural anti-bacterial properties [11–13].

(5) Jute fibre

Jute is extracted from the bark of the white jute plant, *Corchorus capsularis* and to a lesser extent from *tossa jute (C. olitorius).* It flourishes in tropical lowland areas with humidity of 60% to 90%. A hectare of jute plants consumes about 15 tonnes of carbon dioxide and releases 11 tonnes of oxygen. Yields are about 2 tonnes of dry jute fibre per hectare.

Jute is long, soft and shiny, having a length of 1 to 4 m and diameter from 17 to 20 μm. It is one of nature's strongest vegetable fibres and ranks second only to cotton in terms of production quantity. Jute has high insulating and anti-static properties, moderate moisture regain and low thermal conductivity [11–13].

(6) Sisal fibre

Sisal fibre is obtained from *Agave sisalana*, a native of Mexico. The plant grows well in a variety of hot climates, including dry areas unsuitable for many other crops. After harvest, the leaves are cut and crushed in order to separate the pulp from the fibres.

The average yield of dried fibres is about 1 tonnes per hectare, although it has been reported that yields in East Africa reaches up to 2.5 tonnes. This lustrous, creamy white sisal fibres measure up to 1 m in length with a diameter of 200 to 400 μm. It is a coarse, hard fibre unsuitable for textiles or fabrics, but is strong, durable and stretchable, does not absorb moisture easily, resists saltwater deterioration and has a fine surface texture that accepts a wide range of dyes [11–13].

1.3 Annual production of cellulose fibres

Attempts to make use of natural fibres to manufacture a wide variety of traditional and novel products ranging from textiles, ropes, nets, brushes, carpets, mats, mattresses and even paper or panel boards pertain in almost every country. The long fibres extracted from their respective sources, are transformed to threads or yarns which are used to join, connect or attach to form networks or weaves [14, 15].

The fibre and textile industries stand among the most labour intensive sectors and therefore play a major role in industrialisation of several countries where labour is cheaply available. Textile production is often a major economic output of these countries. However, in many of the less developed countries the fibre and textile sector is still poorly developed, but stimulates the socio-economic development.

This development should be sustainable and not at the expense of the environment or by exploiting workers. In the value addition chain of fibre crop production and their supply to markets, the environmental impacts incurred are distinguished. The impact factor over the environment is related to the production volumes of fibre products and the size of the end-use market [14, 15].

The average global production volumes for different natural fibres derived from agricultural crops, the expected production volume of major natural fibres from India and the average expected cost of fibres in USD per tonne are provided in Table 1.1. These projected values (in 10^3 metric tons per year average) for the year 2017 are estimated from data over the recent years [14–16].

Table 1.1: Estimated annual production of cellulose fibres in 2017 [14–16]

Fibre source	World production (10^3Tons)	Production in India (10^3 Tons)	Cost (USD/Tonne)
Coir	851.6	531.5	250-550
Cotton	25,000	6,423	1500-2200
Jute	3036.3	1580	650 -770
Flax	810	—-	500-1500
Hemp	215	—-	600-1800
Sisal	281.6	12	1300 - 1600
Banana	100	1.63	100-150
Kenaf	255.6	130	250-300

1.3.1 Coir

Globally, about 500,000 tonnes of coir at a total value estimated at $100 million are produced annually. India and Sri Lanka are the main exporters, followed by Thailand, Vietnam, the Philippines and Indonesia. Around half of the coir produced is exported in the form of raw fibre. Smaller quantities are exported as yarn, and as mats and matting. Coconuts are typically grown by small-scale farmers, who use local mills for fibre extraction [14, 15, 17].

1.3.2 Cotton

Cultivated in around 80 countries, cotton is one of the world's most widely produced crops and uses about 2.5% of the world's arable land area.

Cotton is fundamental to the economies of many developing countries, particularly in West and Central Africa, where around 10 million small farmers depend on this sector for their income. The world produces around 25 million tonnes of cotton every year. Six countries - China, Brazil, India, Pakistan, the USA and Uzbekistan accounts for more than 80% of total production [14, 15, 17].

1.3.3 Jute

India and Bangladesh are the world's main jute producers, with Myanmar and Nepal following the line. Collectively in India and Bangladesh, about 4 million farmers earn their living and support 20 million dependants from jute cultivation, while many work in the jute manufacturing sector which shows the economic significance. Jute production fluctuates, influenced by weather conditions and prices. Annual output ranges from 2.3 to 2.8 million tonnes, on par with wool. India produces 60% of the world's jute, with Bangladesh, accounting for most of the rest [14, 15, 17].

1.3.4 Flax

The leading producers of flax fibre are France, Belgium and the Netherlands. Other significant producers are China, Belarus and the Russian Federation. The total area dedicated to flax cultivation for fibre is estimated at around 120,000 ha in Europe, and around 320,000 ha worldwide.

In 2007, the European Union produced 122,000 tonnes of flax fibre, making it the world's biggest producer, followed by China with about 25,000 tonnes. China is also a major buyer of raw flax for processing, with imports of 60,000 tonnes a year, including most of Europe's flax fibres. Bulk linen production has shifted to Eastern Europe and China, but niche producers in Ireland, Italy and Belgium continue to supply the market for high quality fabrics in Europe, Japan and the USA [14, 15, 17].

1.3.5 Hemp

The world's leading producer of hemp is China, with smaller production in Europe, Chile and the Democratic People's Republic of Korea. In the European Union hemp is grown on around 15,000 ha of land. Major producers are France, Germany and the UK. China is the largest exporter of hemp textiles, mainly to Europe and North America, where the market for hemp clothing is growing rapidly. They also export hemp-based fibre boards [14, 15, 17].

1.3.6 Sisal

Sisal is cultivated for fibre in Brazil, China, Cuba, Kenya, Haiti, Madagascar, and Mexico. Production patterns differ between countries. In Tanzania and Kenya sisal is predominantly a plantation crop, while production in Brazil is largely small-scale. World production of sisal and a similar agave fibre, henequen *(Agave fourcroydes)*, is estimated at around 300,000 tonnes, valued at $75 million. The major producers are Brazil, Tanzania and Kenya [14, 15, 17].

1.4 Natural Fibre as an Engineering Material

Similar to agriculture, textiles also play a major role in human history since the dawn of civilization. Natural fibres have been extensively used in the textile industry. Fragments of cotton articles dated from 5000 BC have been excavated from sites of Mexico and modern day Pakistan. Chinese tradition believes that the history of silk begins in the 27th century BC. The oldest wool textile found from Denmark, dates back to 1500 BC, and the oldest wool carpet from Siberia to 500 BC. Fibres such as jute and coir have been cultivated in India since 1500 BC [17].

The thought of using natural fibres to reinforce composite material dates back to ancient civilizations where they used grass or straw to reinforce mud blocks and form adobe bricks. Later on composites made from coconut fibre/natural rubber latex were popular in the automotive industry as they reduce the overall weight and hence improved performance. However, cellulosic fibres were gradually substituted by newly developed synthetic fibres owing to their better performance and since then, the use of cellulose fibres has been limited to the production of ropes, strings, mats, carpets, apparels and other decorative products [5, 6].

Over the past few years, there has been an increased interest in using natural fibres as reinforcement material in composites and to some extent in replacing plastics. This sudden resurgence of interest is due to the positive shift in the global view of green and sustainable manufacturing and also the environmental benefits of using renewable and biodegradable materials [6].

Today, most natural fibres still have found application in fabrics and containers and to insulate, soften and decorate our living spaces.

Increasingly, however, natural fibres are being used for industrial purposes as well as reinforcements for composite materials, in medical implants, and Geo textiles [17].

Among natural fibres, wood has been the most extensively used reinforcement in polymer matrices. However, due to expensive manufacturing processes and the desire for sustainability, a shift in interest has occurred towards other sustainable natural fibres that would otherwise be treated as agro-wastes [18, 19].

1.4.1 Chemical composition of vegetable fibres

The cell walls comprise of sugar-based polymers like celluloses, hemicelluloses and lignin. Unlike celluloses and hemicelluloses, lignin is resistant to microbial attack and anaerobic processes, which allows it to slowly degrade in aerobic conditions, whereas hemicelluloses strengthens lignocellulosic fibres, while maintaining their flexibility. Lignin thus plays a significant role in providing the mechanical strength or rigidity to a plant as well as in preventing invasion by pathogens and pests [3, 16].

Fibres also contain varying amounts of extracts, such as pectin, fats, and waxes. The physical and chemical composition of a plant fibre can vary over a wide range due to difference in origin (species, geographic location, climate, and part of the plant) and age of fibre along with the methods adopted for measurement [16, 20].

Chemical composition can also vary within a plant's structure. Fibre width can also vary along the length of a fibre. For example, Pritchard et al. (2000) [21] reports that coconut fibres tend to be thicker in the middle of the fibre and tapered at the ends. Table 1.2 shows the chemical composition of some common cellulose based plant fibres.

Table 1.2: Chemical composition of selected vegetable fibres [16, 20]

Type of Fibre	**Cellulose** (%)	**Lignin** (%)	**Hemi celluloses** (%)	**Pectin** (%)	**Ash** (%)	**Water Solubles** (%)	**Wax/fat** (%)
Coir	32-53	40-45	0.2-0.3	3-4	—	4.5	—
Kenaf	36-72	9-19	20-21	2	—	—	—
Jute	51-84	5-14	12-24	0.2-4.5	.17-.7	0.5-2	0.4-0.8
Hemp	57-92	2.8-13	6-22	0.8-2.5	0.7-3	0.8-2.1	0.7-0.8
Abaca	56-68	5-13	19-25	0.5-1	—	1.4	0.2-3
Sisal	43-88	4-14	10-15	0.5-10	.14-.55	1.2-6	0.2-2
Flax	60-81	2-5	14-21	0.9-3.8	1.5	3.9-10.5	1.3-1.7
Cotton	82-96	0-1.6	2-6	0-7	0.8-2	0.4-1	0.6

The different chemical and physical composition of these fibres also leads to variations in mechanical properties (see Table 1.3). Although variations in strength are found between fibres, natural plant fibres provide engineers with a viable resource of lightweight materials that can add value, lower cost, and improved sustainability of engineering projects [3].

1.4.2 Mechanical properties

The selection of fibre for various applications depend on factors like length, strength, and purpose of usage. Cell dimension of lignocellulosic fibres depends on the species, maturity and location of plant growth and also on the fibre extraction conditions [23]. Table 1.3 reports various physical characteristics and mechanical behaviour of selected cellulosic fibres, with their respective density, microfibril angle, fibre elongation and Young's modulus of fibres.

Table 1.3: Physical and mechanical behaviour of natural fibres [16, 20, 22]

Type of Fibre	**Density g/cm^3)**	**Moisture absorption (%)**	**Tensile Strength (MPa)**	**Elongation at break (%)**	**Young's Modulus (GPa)**	**Microfibril angle (Deg)**
Cotton	1.5-1.6	8-25	287-597	3-10	5.5-12.6	20-30
Jute	1.3-1.46	12	393-800	1.5-1.8	10-30	7-10
Coir	1.2	10-12	175-220	15-25	4.0-6.0	30-49
Banana	1.35	10-12	529-754	1-10	7.7-20.8	10-12
Sisal	1.33-1.5	11	400-700	2.0-14	9-38	10-25
Flax	1.4-1.5	7	345-1500	1.2-3.2	27.6-80	5-10
Pineapple	1.44	11.8	413-1627	1.6-3	34.5	6-18
Hemp	.75-1.48	8	550-900	1.6	70	2-6.2

These properties are strongly influenced by many factors like chemical composition and internal fibre structure which differs between different parts of a plant as well as between different plants. Other factors that may affect the fibre properties are maturity, separating process, microscopic and molecular defects such as pits and nodes, type of soil and weather conditions under which they were grown [4, 16].

1.4.3 Utilization of cellulose fibres

Cellulose fibres are generally lignocellulosic in nature, and consists of helically wound cellulose micro fibrils in an amorphous matrix of lignin and hemicelluloses. Retting, scrapping and pulping are the various methods that can be employed to separate fibres from the plant [8]. Natural fibres offer several unique advantages over synthetic fibres as they are abundant, cheap and renewable, which makes them attractive to be used as a reinforcement in composite materials. They also ensure continuous fibre supply and significant savings in material cost to the plastics and composites fabrication industry. Cellulose fibres, despite their low strength, can lead to the development of composites with high specific properties because of their low densities [7].

Contrary to synthetic fibres like glass and carbon fibres which are brittle, cellulose fibres are flexible and will not fracture when processed over sharp curvatures. They are non-abrasive in nature and permits a high volume fraction of filling during processing, and this results in high mechanical properties without the usual machine wear problems associated with synthetic fibres especially glass and ceramic[6, 7]. Cellulose fibres are also non-toxic, easy to handle and brings no health problems unlike glass fibres that can cause skin irritations and respiratory diseases when the fibrous dust is inhaled [24].

They offer good scope for surface modification, require low amounts of energy for processing, and are economical, renewable and biodegradable. In terms of socio-economic issues, the use of cellulose fibres as a source of raw materials proves to be beneficial when it generates an opportunity for economic development of non-food farm products in rural areas. These advantages and benefits are not likely to be ignored by the manufacturing industry for use in the automotive, building, appliance and other applications [7].

1.4.4 Bio degradation of cellulose fibres

Lignocellulosic materials including cellulose fibres degrade easily when exposed to nature. Nature builds lignocellulosic resources from carbon dioxide and water, and it has all the tools to recycle them back. Possible ways of degradation include biological, thermal, aqueous, photochemical, chemical, and mechanical means of degradation. In order to produce cellulose fibre-based composites with a long service life, the degradation processes caused by nature need to be retarded [24].

One of the most notable features of natural fibres is their ability to absorb water and degrade, which is particularly beneficial in short-term applications, such as erosion control and filtration. Natural plant fibres absorb moisture when they are exposed to humid atmospheric conditions or when immersed in water. They swell and contract because their cell walls contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding [3]. Although this is mainly because of the presence of hemicelluloses, cellulose also contains numerous hydrophilic hydroxyl groups [10].

When water comes in contact with plant fibres, the cell walls swell until they are saturated with water, reaching the fibre saturation point (FSP). Once this occurs, any remaining water will exist as free water within the void structure and will not contribute to further swelling. This is a reversible process, where the fibre will begin to shrink once it loses water below its FSP. The swelling of fibres can lead to micro cracking and reduction in mechanical properties, when exposed to frequent wetting and drying due to differential swelling when different parts of the fibre have different moisture contents [3, 10].

1.5 Engineering Applications

Natural plant fibres have been extensively used for centuries in a variety of different applications. Fibres such as hemp, kenaf, abaca, sisal, coir, and straw have been widely used as rope, twine, and cordage. Natural fibres have also been extensively used for manufacturing paper, clothing, or carpets and are finding their way into everything from tea bags and animal bedding to packaging materials.

Natural plant fibres provide engineers with a renewable resource of light weight materials that can add value, lower cost, and improve the sustainability of engineering materials. They also provide engineers with tremendous opportunities to develop a variety of new and different products that offer important advantages. The following section summarizes some of the applications of natural fibres in engineering applications.

1.5.1 Automotive materials

The automotive industry has opened up a whole new market for the use of natural fibres in non-structural composite applications in cars, trains, trucks, and air planes. Nonstructural applications are those where the product will not be required to carry a load. The main biomass-based natural fibres which are used in the automotive sector are wood, cellulose, flax, jute, sisal, hemp, and kenaf fibres in which kenaf, hemp, flax, jute, coir, and sisal, have been used in door panels, seat backs, and dashboards [3,23]. In order to improve the fuel consumption and efficiency of vehicles, there has been a continuous effort in the automotive industry to seek high strength raw biomass-based materials that are more eco-friendly [25].

1.5.2 Building materials

Natural fibres are finding tremendous application in both structural and non-structural building applications, particularly in residential construction. Hemp and flax fibres are being evaluated for use in composite load-bearing beams and structural panels.

Natural fibres are also being used in non-structural building applications. Wood fibres have been extensively used in producing particleboard, fibre board, and flooring. Recent advances have also been made in the use of hardwood, softwood, rice husk, kenaf, and coir fibres in foamed composite decking, siding, and window blinds. Natural fibres, such as kenaf and coir, are also found in insulation materials [3, 12].

1.5.3 Geo-environmental materials

Natural plant fibres have also been used in a variety of different geo-environmental applications to improve or protect soil and water. Natural plant fibres can be used as filters to remove particulates from drinking water, storm water, air, and wastes. Natural plant fibres like kenaf/jute fibres are also used as sorbents, which sorb oil out of seawater and filter heavy metals from storm water [3].

Geo-textiles are made out of both natural, typically coconut or jute, and synthetic fibres and can be either woven or nonwoven and are used in a variety of separation, reinforcement, filtration, and drainage applications, such as around conventional pipe under drains, behind retaining walls, in earth dams, in landfills, and as wick drains. They are also used to reinforce soft subgrades and retaining walls. Natural plant fibres, such as coconut, jute, straw, and wood, are also commonly used in rolled erosion control products (RECPs). RECPs are made of degradable (natural fibre) materials for temporary applications and non-degradable (synthetic fibre) materials for long term applications. They are manufactured or fabricated into rolls designed to reduce soil erosion and assist in the growth, establishment, and protection of vegetation [3, 10].

1.5.4 Applications of biomass-based composites

A composite may be defined as the combination of two or more individual materials and it shows selected properties of each constituent. A suitable combination of different constituents results in a superior substance or material from practical point of view [9, 25].

Biomass-based composites are used in many different areas such as furniture for the garden and house, parts for cars and technical devices. Along with these applications, biomass-based materials are also being used in the preparation of products for medicine and nanotechnology. Depending on the application, the fibre content in a composite varies between 20 to 90% by weight.

A number of everyday household consumer products, such as furniture, automotive parts and decking materials, are today being fabricated from biomass-based composites using different processing techniques such as compression moulding, injection moulding and extrusion. The easy availability, non-abrasiveness, non-toxic nature, light in weight and low specific gravity of biomass-based materials as reinforcement makes possible the development of new eco-friendly materials. All these properties of the biomass-based materials thus give the resulting composite materials an added advantage over composites which contain inorganic fillers [9, 25].

1.6 Limitations of Natural fibres

Despite all these advantages mentioned in the previous sections, use of cellulose fibres in thermoplastics has not been extensive. Possible reasons that contribute to unsatisfactory final properties of the composite includes:

(i) Limited thermal stability is exhibited when the typical melt processing temperatures reach around 200^0C. This restricts the range of thermoplastics that can be used with these fibres [7].

(ii) Poor dispersion characteristics shown in the non - polar, olefinic thermoplastic melt due to strong hydrogen forces between the fibres [24].

(iii) Limited compatibility with many of the thermoplastic matrices due to their highly hydrophilic character which results in poor mechanical properties of the resulting composites [24].

(iv) High moisture absorption of the fibres which can affect the dimensional stability of the composite and the interfacial bond strength [6];

(v) High biodegradability when exposed to the environment which limits the service life of composites, particularly in outdoor applications [24].

Other factors that may hamper increased use of cellulose fibres in plastics are problems and costs associated with the collection and storage which are not yet mechanized and standardized to produce fibres of high and uniform quality [24].

1.7 Scope for improvement of properties

The common limitations that restrict the widespread use of natural fibres in bio composites needs to be overcome through modification of natural fibres by chemical treatment. The mechanical properties and physical behaviour of the material also needs to be modified through chemical treatments.

1.7.1 Chemical modification of cellulose fibres

Natural plant fibres are most suitable for chemical modification to enhance their properties. Lignocellulosic fibres are hygroscopic in nature, flammable and biodegradable, which restricts the service life of composites; many methods are tried to neutralise these drawbacks by chemical modification. Chemical modification through plasma activation or graft polymerization with vinyl monomers, such as acrylonitrile showed improved performance in composites, however, there was considerable increase in fibre cost.

It is reported that acetylation improves thermal stability and moisture absorption capacity of fibreboards made from kenaf/jute fibres. Fibre dispersion problem is seen to be overcome with pretreatment of the fibres with polymer coating materials. The introduction of polymer coatings on the fibre surfaces helps to separate fibres from each other, eliminating the hydrogen bonding that holds them together [3, 24].

For example, cellulose fibres can be surface-coated with butyl benzyl phthalate (BBP) - plasticized Poly Vinyl Chloride (PVC) (commercial name - Santoweb W), and be used as reinforcements. Coupling agents such as isocyanates and silanes when applied modifies the fibre-matrix interface by forming a bridge of chemical bonds between the components which results in improved fibre-matrix adhesion, and is reflected in the mechanical properties of the composite [24].

1.7.2 Improvements in thermal stability and flame resistance

Bio composites are generally flammable and degradable in nature. Almost all reported attempts to improve the thermal stability of natural fibre bio composites focused on treatment of fibres alone and any attempt to treat the composites (end product) are sparsely reported. A simpler method of imparting thermal stability would be when the treatment is done on the bio composites which are rather easier to mechanize and would be suitable for large scale production.

The flame resistant properties of bio composites are a major concern when major applications projected for its use includes building materials and furnishing in high rise apartments, or use of it in railway coaches where the number of people directly affected is more. This necessitates the study of thermal stability and flame resistant properties of bio composites and the development of a suitable and simple method for efficient treatment of bio composites is very much required for widespread commercial use of bio composites to be feasible.

1.8 Background & Motivation

Although the research on green materials has become widespread, studies pertinent to fabricated boards made of coir fibre are found to be quite limited. Coir fibre is a cellulosic natural fibre and is a biomass by-product by nature. Hence there is a great potential for the coir fibre-based composite materials to replace the existing commercial prefabricated wood products. For creating a viable market for coir composite panel boards, it is essential to incorporate novel applications where plywood or traditional synthetic polymer composites failed to penetrate. Though coir fibre based composite boards could match with the properties of wood or plywood, they are highly inflammable and are susceptible to fire [26, 27].

The increasing urbanization and lifestyle changes have resulted in a mushrooming growth of high rise buildings. These skyscrapers are regarded as potential sites of mass casualties in case of fire due to the heat, noxious fumes and other gases. In such occasions, fire retardant materials in the form of panel products, doors and windows play a vital role in saving life and property by reducing flammability of material or by delaying their combustion.

Fire retardant treated (FRT) panel products contain special fire retardant chemicals to induce the required properties like lower flammability, reduced rate of burning or resistance to flame spread. Fireproofing chemicals like Zinc Borate, Boric acid, Borax and Ammonium sulphate are found to be helpful for imparting fire proofing qualities such as lower flammability, reduced rate of burning, resistance to flame spread or higher flame penetration time in FRT panel boards [26, 27].

An FRT coir fibre board can find major applications in high rise buildings, interior woodwork in the kitchens, restaurants, theatre halls, compartments of trains and any other such places where the risk and spread of fire is critical and has to be reduced. Another application can be a fire check door where a fire-resistance rating is used as a part of passive fire protection system to reduce the spread of fire or smoke between compartments and to enable safe egress from a building or structure [27]. The requirements for fire resistant wood based composites have expanded from an initial focus on fire resistance to encompass other factors such as smoke inhibition, environmental impact and economic aspects.

The research on improvement of fire resistance characteristics of coir composite boards and their treatment methods, leading to developing better techniques, materials and products has assumed great significance. Though the mechanisms of fire resistance have been fairly well established for wood, specific research on coir fibre reinforced composites is minimal and the methods of fire retardation and means to prevent flame propagation needs to be explored. Despite the wide applications of coir fibre reinforced composite products, little research, has been seen to evaluate the composite's flammability and the effect of chemical treatment on composite fibre boards and corresponding changes in its properties.

CHAPTER 2

LITERATURE SURVEY

The rapid pace of urbanisation coupled with stringent regulations that restrict mass deforestation and growing environmental concern have created a dearth in forest based building materials like wood. The manufacturing of non-wood lignocellulosic fibres for industrial products and the idea of using these green construction materials can be promoted only by integrating cost effective, environment friendly technologies with locally available materials and traditional techniques. The disposal of Agro-Industrial solid wastes are also a serious concern in many developing countries like India, however the effective conversion of agro-waste to green construction materials could minimise the use of natural resources and energy [28–30].

2.1 Sustainability - Role of coir composites

Environment friendly technologies to extract fibres from different agricultural residues and their processing into value added composite products using formaldehyde based resins have been established. Adoption of similar technologies leads to implementing a green solution for solid waste disposal as an alternative to crop residues being burnt or disposed and thus favours environmental protection and sustainable development by recovering and re-utilizing organic wastes. Although green composites from several sources have been identified and explored to develop appropriate manufacturing techniques, only a limited number of these have been commercialized to date.

Even though these eco-friendly composites offer a new alternative, the product has to satisfy a variety of challenging requirements and has to be competent in every sense. However, the market and commercialization of bio composites are anticipated to expand in the future with novel applications, technological innovations, relentless socio-environmental considerations and investments from the state [28–31].

2.2 Coir fibre: Production and Global Market

Coconuts are grown in more than 93 countries in the world over an area of 12.29 million hectare, producing approximately 64.3 billion coconuts globally. The total area under coconut cultivation in India, is about 1.89 million ha and some 16,943 million nuts are produced annually [14, 32].

About 6.7 million tonnes of husk are produced annually in India, assuming the weight of husk in each nut as 0.4 kg. The coir dust is estimated to be about 70% of the weight of the coconut husk for every coconut [33]. Therefore, assuming that if 50% of the total husks are available for fibre extraction, the potential availability of coir fibre in India is roughly 1.15 million tonnes but only around 0.5 million tonnes of coir fibre are produced in India [14] which shows the underutilised potential in this sector. To sum up, if the by-products of coir industry from the husk used for coir like pith, shearing waste and coir dust are added an enormous quantity of this residue is potentially available.

The production of coir fibre during recent years is illustrated in figure 2.1 which clearly depict the position of India as a world leader among the other coir producing countries.

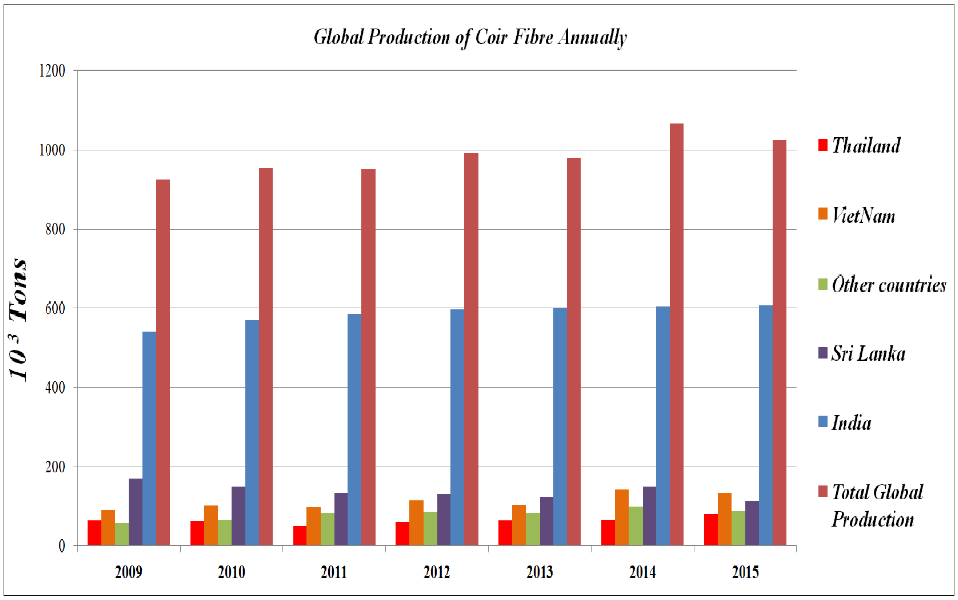


Figure 2.1: Global Production of coir fibre in 10^3 Tonnes

Approximately 60% of the total global coir fibre production is from India. Over the last 25 years, India has generally been the leading producer of coir fibre in the world. India experienced rapid growth in the production of coir fibre in the early 1990s, which has been unmatched by any of the other raw coir-producing countries like Sri Lanka, Thailand, Philippines, and Vietnam.

India's exports of coir fibre have, been steadily increasing in recent years, going from 1% of the world's coir fibre export market in 1996 to nearly 11% in 2002. In 2016 it has reached nearly 50% of the world exports of coir fibre [3, 14, 34].

Table 2.1 shows the quantities of coir fibre produced in the leading countries in recent years.

Table 2.1: Production of coir fibre in Thousand Tonnes [14, 34, 35]

Year	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
Brown fibre - India	394.00	415.50	425.00	431.50	455.80	458.80	460.60
White fibre - India	97.00	100.00	100.50	100.00	80.40	81.00	81.40
Curled fibre - India	49.50	54.20	59.00	63.80	63.80	64.70	65.00
Total Production (India)	**540.50**	**569.70**	**584.50**	**595.30**	**600.00**	**604.50**	**607.00**
Sri Lanka	169.60	149.80	135.00	131.20	123.70	150.40	112.50
Thailand	64.60	63.70	49.90	61.70	64.80	68.10	80.50
VietNam	91.10	103.00	97.60	115.10	105.90	143.30	135.60
Other countries	57.70	66.50	81.90	84.80	79.30	97.60	87.90
Total Global Production	**923.6**	**952.7**	**949**	**988.9**	**977.1**	**1065.8**	**1023.6**

Although coir fibre is an important commodity, the industry has seen very little growth over the last 30 years, with the exception of China, which has seen unprecedented growth in coir fibre imports over the last decade. In fact, approximately 65-70% of the total production of coir fibre is imported by China.

Having an expanding middle class of 109 million people with increased purchasing power, China produced 1.3 million coir fibre mattresses a month in 2009 and now they require coir fibre of about 572,200 tons/year and this requirement is expected to persist for another decade or more [36]. However, the major historic importers of coir fibre, Germany, United States, UK, and Japan, have seen decreasing trends in coir fibre imports since the early 1970s, although Germany still accounts for 8% of the market, the contribution of USA has declined to nearly 1.2% [14, 34]. But around 15% of the coir and coir products exported from India in 2015 is absorbed by USA [35]. Table 2.2 shows the import statistics of coir fibre by various countries in the world showcasing leading importers.

Table 2.2: Import statistics - Coir fibre [14, 34, 35]

	2009	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**
Canada	33.30	52.30	76.60	66.30	29.10	45.20	82.30
USA	7.70	5.70	8.60	10.30	10.80	10.10	9.80
European Union	86.70	80.30	88.80	92.10	90.40	106.60	116.20
Other Europe	1.30	1.30	1.10	1.80	1.80	1.10	1.00
Russia	0.50	2.40	3.70	4.20	5.70	5.90	7.30
Australia	2.00	2.40	2.50	4.20	3.80	4.40	6.20
New Zealand	1.50	1.80	1.80	1.70	1.90	1.70	1.90
Japan	2.20	3.50	3.30	2.00	2.30	2.90	3.40
South Africa	2.40	3.30	3.50	4.50	4.40	3.90	5.10
China	**352.90**	**387.00**	**412.20**	**505.20**	**515.00**	**572.20**	**573.7**
Taiwan	5.90	5.80	6.10	4.80	4.90	5.30	4.90
South Korea	17.70	23.50	38.90	51.60	90.80	76.50	81.60
Pakistan	3.60	4.80	11.40	4.10	3.00	1.30	1.30
Saudi Arabia	4.00	3.10	4.40	5.00	4.10	5.00	5.00
Malaysia	1.90	2.00	1.20	2.60	1.40	1.50	2.70
Others	1.30	1.50	1.30	1.10	5.20	5.30	5.20
Global statistics	**524.80**	**580.60**	**665.50**	**761.40**	**774.80**	**848.20**	**907.70**

In recent years, new markets have also emerged in the Netherlands, Spain, and Slovenia. In fact between 2002 and 2004, the top two importers of coir fibre from India were the Netherlands (41%) and Belgium (14%) [3, 34]. Although these have been viable markets for India's coir fibre, these countries made up only 10.6% of total Indian coir fibre imports in 2014 [35].

2.2.1 Potential of Coir Composites

An annual estimated 877,100 tonnes of coir fibre are produced globally, in which India (607,000 tonnes) is a main contributor, followed by Vietnam (135600 tonnes), Sri Lanka (112500) and Thailand (80500 tonnes). India together with Sri Lanka contributes around 80% of the estimated annual production of coir fibre worldwide. Apart from the main contributors, Coconuts are grown in around 90 countries in the world, including Indonesia, Malaysia, Brazil and the Philippines and they produce coir fibre and collectively accounts to around 12,000 tonnes of total annual production [14].

Thus a developed coir industry with value added products and market expansion is feasible economically with more employment opportunities. Around 80% of the total production of coir fibre is exported as raw fibre and the rest as yarn, mats, matting and rugs. An innovative product like green composite based on coir fibre has entered the markets of India to substitute plywood and has all the properties of phenol-bonded plywood. Fibre reinforced phenol bonding results in additional strength, has high abrasive wear resistance and withstands variations in temperature with respect to thermal expansion/contraction.

A major share of coconut husk is seldom used for making value-added products and therefore is treated as an agricultural by-product or waste. The development of medium-density coir boards from a green material like coir fibre has been gaining attention as it facilitates value addition [14, 37]. Taking into account this largely unused potential, investment on manufacturing facilities to produce coir fibre based panels looks justified. It also opens up new job opportunities particularly in developing countries which helps to wipe out poverty and unemployment.

Substituting timber products with coir composite boards could save a substantial amount of tropical trees being logged. A manufacturing unit of coir composite boards with an average production rate of 40 m^3 a day would be able to save approximately 5000 trees annually. Based on an assumption that one such tree requires 40 m^2, and then such a unit could save about 260 acres of tropical forest every year. If 10% of the total production of coir fibre is utilized in the manufacture of coir composite boards, it would save about 880,000 tropical trees per year, which corresponds to 8,800 acres of tropical forest per year [38, 39].

The chemical composition of coir fibre is similar to that of wood fibre and consists of cellulose forming 30-45%, hemicellulose with 5-15%, lignin accounting for 30-45% and the remaining is water soluble, ash and pectin. The lignin content of coir fibre is found to be comparable with that of wood fibre which is up to 37% [38, 40, 41]. Lignin is a hydrophobic aromatic polymer thermally stable than cellulose and hemicelluloses. Hemicelluloses are low molecular weight polysaccharides that grow around cellulose fibres while cellulose forms cell walls and give tensile strength and lignin accounts to rigidity (compressive and shear strength) of the material and contributes to char formation than cellulose and hemicelluloses which reduces the emission of combustible gases and reduce further thermal degradation [26, 42, 43].

2.3 Nature and composition of coconut husk

The external appearance of the husk varies from dull brown when fully ripe to bright green while immature; however, some varieties are found to be golden yellow or yellow brown in colour. The husk is full of long, coarse fibres, all running in the same direction. The fibres are embedded in a matrix of material called coir dust [33, 44].

The composition of a matured coconut husk that gives brown fibre is as illustrated in Table 2.3. The main constituents in the husk of a mature coconut and their quantity in percentage are as listed. Since coconut husk is a porous material, they absorb and retain water.

Table 2.3: Composition of coconut husk [33, 44]

Constituents	**Percentage**
Moisture Content	15.0
Lignin	43.0
Ash Content	8.26
Alkalinity of ash (as K_2O)	37.5

Figure 2.2 shows a whole coconut and its cross section. It has the outer husk which is a fibrous covering (5-10 cm thick) that envelops the nut and protects it while falling from the tree. Inner nut shell is the hard shell structure (3.5 mm thickness) of coconut fruit and white coconut meat (copra), from which coconut oil is extracted [33, 44].

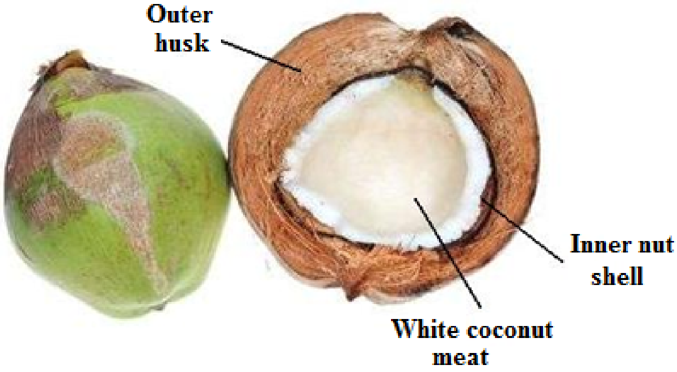


Figure 2.2: Coconut (whole) and Cross-section

The average approximate dry weight of husk present in each coconut fruit is about 0.3 - 0.4 kg. It has been established that 30% of the husk can be obtained as commercial coir fibre while the remaining 70% becomes coir fibre dust. Of this extractable fibre, 40% is coarse type usually referred to as bristle fibre and 60% is the finer material mattress fibre [33, 34].

2.3.1 Coir Fibre

Coir fibre is a seed fibre which is thick and coarse in nature and are extracted from the fibrous husk *(mesocarp)* of the outer layer of coconuts (seeds of coconut palm *Cocos nucifera L.*). The coconut husk consists of the epicarp (water-resistant outer skin) and the mesocarp (fibrous zone). The mesocarp consists of fibre bundles embedded in non-fibrous parenchyma tissue [45].

Natural fibres like coir provide engineers with tremendous opportunities to develop more efficient, less expensive, lightweight, renewable green products, and they are increasingly being used due to their unique properties in terms of strength, elongation and durability. They are also mouldable, anisotropic, hygroscopic, non-abrasive, combustible and compostable in nature [41]. Their low density, good thermal insulation and comparable mechanical properties coupled with favourable factors like reduced tool wear and problem-free disposal makes them an attractive choice [19, 46]. Coir fibre can substitute for synthetic fibres in a variety of applications. In addition to exhibiting good mechanical and acoustical properties, they are also biodegradable and buoyant [47].

The composition of coir fibre is shown in table 2.4 and the main constituents include lignin and cellulose. Cellulose, hemicelluloses and pectin make up the bulk in the ground tissue of the husk and lignin is responsible for the stiffness and partly for natural colour of coir fibre.

Table 2.4: Composition of coir fibre (in percentage of dry weight) [33, 46]

Coir Fibre	**Old Nut (%)**	**Young Nut (%)**	**Very Young Nut (%)**
Water soluble substances	26	29	38. 50
Pectin, others soluble in boiling water	14. 25	14. 85	15. 25
Hemicelluloses	8.5	8.15	9. 00
Lignin	29. 33	31. 64	20. 13
Cellulose	23. 87	19. 26	14. 39

There are two types of coir fibre: white and brown coir. White fibre bundles originate from immature green coconut while brown coir is obtained from ripened coconuts that have lost their green colour. The colour of brown coir fibre varies from light brown to very dark brown, depending on the variety and maturity of the coconut from which it is extracted [45].

These fibres are stiff, coarse, resilient and pliable apart from being resistant to bacterial attack and also exhibits good water resistance. The fibres are made up of elementary fibre cells with an approximate length of about 0.7 mm and thickness of 12mm to 20 mm. The largest of the fibres are seen to have a length of up to 35 cm and thickness from 0.3 mm to 1.5 mm in the middle [33].

The favourable properties of coir fibre when compared to synthetic fibres are less weight, easy availability, low cost and high specific strength. Coir fibre is a renewable natural resource and is also biodegradable. The ever increasing concern and customer awareness over environment promotes the usage of recyclable raw materials and productsderived from it. This in turn could increase the demand for lignocellulosic composite materials like medium density coir fibre boards [48].

Composites with favourable properties like design flexibility, high strength-to-weight ratio, high impact strength, stiffness and durability have come along in replacing conventional materials including metals, alloys and wood. The development of coir composite boards from an environment friendly green material like coir fibre to substitute wood gets more significant because it facilitates the eco-friendly recycling of coir fibre which is otherwise treated as waste and burned for disposal [37].

Scanning Electron Microscope (SEM) images of coir fibres are shown in figure 2.3. Figure 2.3 A and 2.3 B gives the cross sectional view of coir fibre at a magnification of 50 μ m and 500 μ m respectively and Figure 2.3 C and 2.3 D gives the longitudinal view of coir fibres at a magnification of 200 μ m and 100 μ m respectively [45].

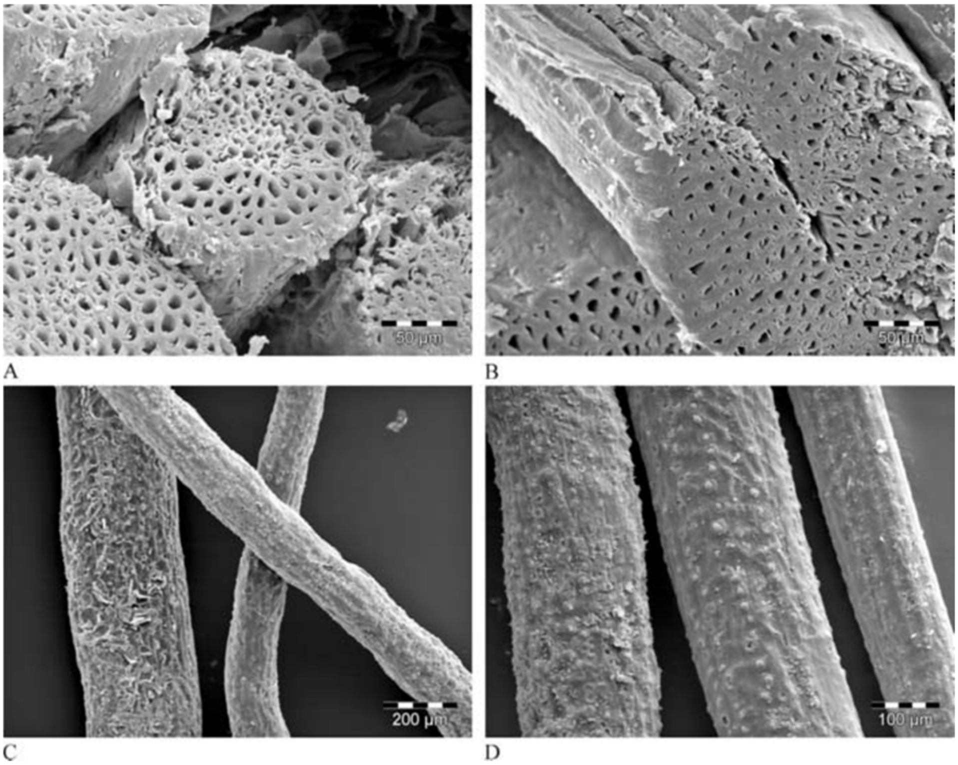


Figure 2.3: SEM images of coir fibres.
A and B - cross sectional views; C and D - length wise views

When viewed under the microscope in cross-section, the fibre is seen to be a simple vascular bundle surrounded by a sheath of thickened schlerenchymatous cells (supporting or protective tissue composed of thickened, dry and hardened cells). Coir fibres are long, tube-like structures made up of cells and their basic structure consists of a central void or lumen surrounded by a cell wall. As seen in Figure 2.3, coir fibres have pore spaces that allows for the transport of water through the fibre which influences the ultimate strength and elongation of coir fibres.

2.4 Coir processing from coconut husk

First process involves dehusking of whole coconuts after harvesting of mature coconut crops. Coir fibre is extracted from coconut husks which surrounds the coconut shell. Coir fibre is seen as only a by product in many parts and the majority of the husk goes unused in terms of fibre production. Owing to the high content of potash, coconut husks are left in the field as a fertiliser or burnt to ash to get potassium for use as fertiliser [33, 49, 50].

2.4.1 Fibre Extraction and processing

There are mainly two methods to extract coir fibre from coconut husk:

- Traditional fibre extraction process and
- mechanical extraction process.

Coir fibre can also be processed through a bio technological approach by using microbial treatment.

(1) Traditional fibre extraction

The traditional fibre extraction process from coconut husk is simple and needs little investment for equipments but is laborious as the process is done mostly manual. It is also time-consuming as the husks have to be retted in water for several weeks to loosen the fibres from the pith. After the separation of husks, they are processed using retting techniques usually in a creek or waterhole having brackish water for about 3-6 months or in backwaters or lagoons which requires 10-12 months of anaerobic (bacterial) fermentation. The husks are softened after retting and can be decorticated while the fibres can be extracted by beating manually followed by washing and drying [49, 50].

The remaining residual pith was considered a waste until recently wherein it has found a profitable market in horticulture applications. Traditional fibre extraction process yields the highest quality white fibre for spinning and weaving and these fibres find increased applications like in coir geo textiles. Retted fibres from green husks are most suitable for dyeing and bleaching. Production of more coarse brown yarns requires shorter periods of retting [49, 50].

(2) Mechanical Extraction

Mechanical processes use either de-fibering or decorticating equipments that could process the husks after only five days of immersion in water tanks. The husk is crushed in a breaker to open the fibres. By using revolving drums, the coarse long fibres are separated from the short woody parts and pith. The stronger fibres are washed, cleaned, dried, hackled and combed [49, 50].

The extracted raw coir fibre itself is of commercial value and is exported, but they are also used to make value added products through various processes. A flow diagram for the processes involved in converting coconut husk for various applications and products is shown in Figure 2.4.

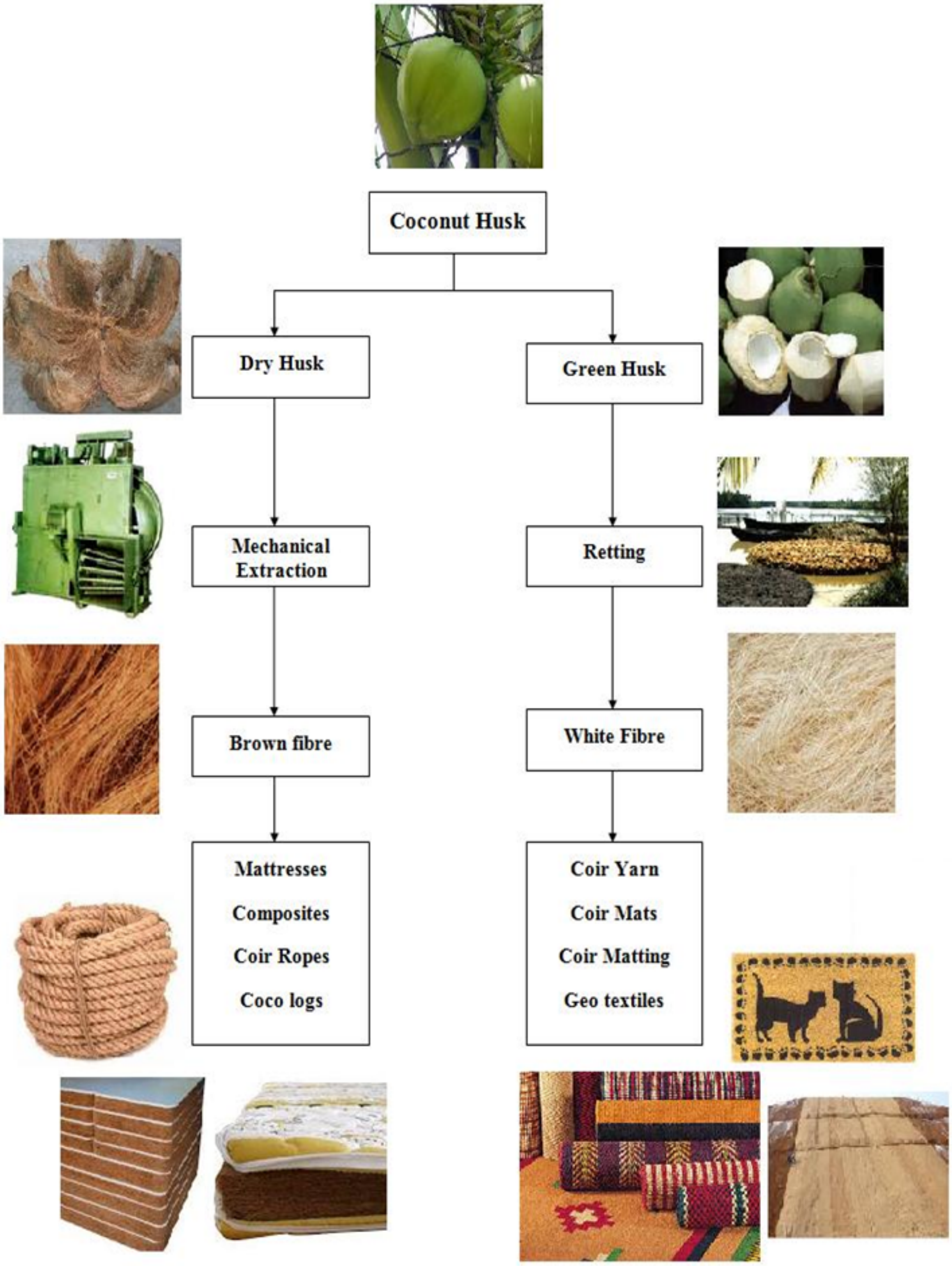


Figure 2.4: Flow Diagram for coconut husk and its applications

(3) Green decortications and Microbial treatments

New environmentally friendly methods for fibre production are of interest and can be locally exploited on relatively smaller-scale, and have the potential to produce constant quality of fibres. Novel developments by the Central Coir Research Institute, Kalavoor a research institute under Coir Board, India uses a bio-technological approach with specific microbial enzymes that have substantially reduced the retting time to three to five days maintaining the same high quality in fibre production. Similar protocols can be developed to enhance the properties of fibre with regard to surface properties such as smoothness and porosity. By using specific (microbial) lignolytic enzymes (laccase/ phenoloxidase), the fibre surface can be bleached or activated to react more easily with the dyes [49, 50].

2.5 Wood as an engineering material

The use of wood for industry and construction has been long established. Wood is considered as a renewable resource and one of the most useful engineering materials because of its wide range of applications. It is also one of the most preferred materials in the field of construction due to its physical and mechanical properties along with aesthetics [26, 42, 51]. However, external factors like climate, direct sunlight or a biological factor like termite attack can adversely affect the physical and chemical properties of wood and forms a major limitation for its outdoor applications [52, 53].

The ever increasing demand for wood as a feasible raw material has resulted in fast depletion of the forest reserves. However, the alarming rate of depletion in forest resources has led to stringent environmental regulations to check their exploitation in several countries including India. This mandates the conservation of available wood resources and makes it a necessity to find alternative sources. Furthermore, this decline of resources has made it more difficult to stably acquire the wood needed for manufacturing various products. The large amount of timber used for a variety of applications, reminds about the importance of conserving this precious resource and ensuring its availability for extended usage [54–56]. The above factors call for the reduction in wastage of wood as well as to develop alternative engineered wood products that could substitute wood.

With the development of wood composites, several natural fibre reinforced materials are being produced wherein inexpensive and sustainable alternatives like agricultural waste have attracted considerable interest [51, 57, 58]. The research and development of new materials to substitute wood, is being driven by rising environmental concern and demand for alternative resources, which in turn has initiated the use of renewable materials like biomass [18, 19]. Lignocellulosic materials like coir fibre are considered as excellent reinforcing materials since they are a renewable natural resource. The growing concern about our environment promotes usage of recyclable raw materials and products, emphasizing the demand for lignocellulosic composite materials [59].

From the point of view of substituting wood and wood-based materials, coir composites can be considered as an ideal solution. Coir fibre boards that can substitute wood will have the potential to penetrate the market, especially when it is based on renewable resources. Coir fibre reinforced composite boards offer good potential to substitute wood and other wood-based material in several applications [37].

2.6 Wood substitutes available in the market

In the past, there have been several attempts to develop suitable substitutes for wood, which in turn have resulted in the development of plywood, MDF boards and laminated particle boards. Currently plywood and MDF board are two popular options available in Indian market as a substitute for wood. The basic difference between the two is that, plywood is made from solid wood, whereas MDF board is made from wood fibres [60]. Wood and its substitutes remain the major raw materials in the furniture and construction industries, while composite panel board manufacturing accounts for one of the fastest growing sectors of the wood and allied products industries [29, 30]. Wood being expensive is used mainly in the manufacture of high class furniture and products, whereas laminated particle boards have limited applications.

2.6.1 Plywood

Plywood is made from good log of wood called peelers. Thin layers are peeled from the log by rotating it along its horizontal axis. The sheets of veneer thus obtained are cut to the desired dimensions, dried, patched, glued together and then baked in a press at 140 ^{0}C (284 ^{0}F) and 1.9 MPa (280 psi) to form plywood panels [61].

Plywood has minimal wastage during production stage as every bit of wood that is cut from the tree is utilized in making plywood. An advantage of plywood is that it could tolerate extreme cold conditions without affecting its dimensions or strength and also exhibit consistent strength across both directions.

2.6.2 Medium Density Fibre Boards

Medium Density Fibreboard (MDF) is an engineered wood product formed from wood fibres obtained by breaking down hardwood and softwood using a defibrator. The wood fibres are then glued together using wax and resin adhesive and made into panels by applying high temperature and pressure. Made from recycled wood fibres and resin, MDF is machine dried and pressed to produce dense, stable sheets[60].

The advantage of MDF is that wastage of wood is minimal during manufacture as it can be made by mixing wood fibres of different types, whereas plywood can be made only by using a single type at a time. Earlier, both plywood and MDF were used for making furniture. But now plywood has been replaced by MDF in the furniture industry. Plywood is now mostly used as a packing material as it has a clear advantage of strength when compared to MDF.

Solid wood boards have a nature of expanding and contracting both horizontally and vertically when temperatures and humidity increases or decreases. Unless these conditions are carefully monitored and controlled, solid wood panels could shrink, crack or buckle in unfavourable conditions of temperature and humidity. Hence MDF is a better option than solid wood due to its stability and adaptability to abrupt changes in heat and humidity [60][62].

Though MDF is made of wood fibres and have superior surface finish than plywood, they exhibit poor screw holding capacity and are adversely affected by moisture. The cross graining of plywood improves dimensional stability by reducing expansion and shrinkage, but due to its non-directional grain structure, MDF boards are ideal when subjected to cutting, machining and drilling. However MDF is not as stiff as plywood and could sag with weight and get damaged by rough handling [60][62].

2.6.3 Bio-composite boards

Coir fibre and its possibilities to form composites have been investigated most extensively in India due to the large availability of coconuts. Coir fibre has been recognised as a highly durable fibre in different types of matrices, like polymer, bitumen, cement, gypsum, flyash-lime and mud. Technologies to manufacture compregs, coir trays (to replace existing metal trays) and coir corrugated sheets have been developed. The properties of these composites meet the requirement of strength with reference to relevant specifications. Investigations have also been carried out to develop particle boards, insulation boards and hardboards using the coconut coir pith and have yielded positive results [63, 64].

Table 2.5 gives the comparison of the mechanical properties of various agro based bio composite boards including coir hardboard, medium density coir board and medium density fibreboard which is very commonly available in the market and is a wide choice in the construction industry.

Table 2.5: Mechanical properties of some bio-composite boards [64–67]

Sl. No.	Properties (Unit)	Coir Hard Board	Medium density Coir board	Medium density fibre board	Wood Particle Board	Rice Husk Particle Board	Bamboo Mat overlaid RHPB*	Wheat straw Particle Board	Ground nut Husk Board	Bagasse Board
1	Density (Kg/m^3)	800	650-900	600-900	755	790	845	800-900	700	683
2	Water absorption (%)									
	After 2 Hrs soaking	—	6	9	7.5	16	7	—	—	20.7
	After 24 Hrs soaking	40	30	45	16	30	15.5	61	—	60.4
3	Thickness Swelling (%)	25	4	7	6.5	7.4	3.3	25	23.65	9
4	Modulus of Rupture (N/mm^2)	30	31	28	16.6	14.53	32.9	25.74	11.8	21
5	Modulus of Elasticity (N/mm^2)	3000	2800	2800	2800	2290	3700	3169	1396	2617
6	Tensile (Internal Bond) strength (N/mm^2)									
	Dry	0.25	0.9	0.8	0.55	0.37	0.39	0.368	0.3	0.52
	Wet	—	—	—	0.18	0.12	0.13	—	0.15	0.13
7	Screw withdrawal strength (N/mm^2)									
	Face	—	2300	1500	1475	1375	2200	—	—	2220
	Edge	—	1700	1250	950	1100	1275	—	—	977

*RHPB stands for Rice Husk Particle Boards

A comparison among the values shown in Table 2.5 reveals that the relevant properties of coir based green composite boards are comparable to any such products currently available for use. Many of these boards are still in the laboratory stage and not currently available in open market. These non standardised boards show little uniformity in their properties, as the source of raw material can seriously affect the test results and thus are recorded sparingly in literatures.

Coir fibre, like any other natural fibre is lighter, less expensive, requires lesser energy in production, is environmentally sound being renewable and biodegradable and also have superior specific strength when compared to synthetic fibres. Coir fibre reinforced in traditional thermoplastic matrices offer good performance at lower prices thus creating a huge potential to replace similar materials in automotive applications where even a fraction of the total weight saving can make significant contribution to fuel efficiency by reducing gasoline consumption and with an added advantage of being eco friendly. Automotive, construction, packaging and consumer products are major segments where coir fibre based bio composites may find applications. Demand for natural fibre based green composites is growing mainly because of their increased acceptability, awareness and also due to consumer affinity towards greener products. The most important feature of green composites is their long-term durability, reduced carbon footprint and complete biodegradability although every green composite made from bio fibre are not biodegradable [18, 19].

The feasibility of manufacturing particle boards from the retted green husk or by chipping dry husk has been established. Similarly, the feasibility of producing hard boards by defibration and manufacturing of fibre board from shearing waste/coir dust has also been established [64–67]. It is estimated that, nearly a million tonne of coir fibre and dust are burnt as a fuel or for disposal. If the economical use of this bio material could be made, the surplus fibre after meeting the demand of coir industry would be available for the panels and board manufacturing industry [63].

2.6.4 Coir Composite boards

Native supply of veneer for manufacturing engineered wood has become limited in India and a subsequent rise in felling acquisition cost while importing them made the producers to assay plausible alternatives [37]. This is where coir fibre board falls into place as they could offer a valuable alternative for the customers with competitive properties like high stiffness, high strength and low density at less weight and could decrease the overall utilization of wood resources [18].

In India, coir is regarded as a versatile fibre which is locally available in abundance and hence is cheaper. India enjoys an immense potential in the coir fibre composites industry as the country is endowed with a large area of coconut cultivation and is the leading producer and exporter of coir fibre in the world [41].

The properties favouring coir fibre in comparison to synthetic fibres are less weight, good availability, low cost, and high specific strength. Coir is a biodegradable, renewable natural fibre containing 32.8% lignin [40, 53]. Coir fibre boards do not pollute the environment to significant levels and are generally easily workable. Being a cost-effective material, its outstanding physical and mechanical properties coupled with aesthetics offer a distinctive advantage over other similar materials and can be used for various applications [42]. Bio composites based on natural fibres like coir fibre would get a wider acceptance as a substitute of wood in the area of building and construction, having enhanced fire resistant capabilities. The application of coir fibre boards in the post disaster management of rehabilitation and rebuilding could become cost competitive compared to other conventional building materials [28–30].

Unconventional building materials like natural fibre reinforced composites are obviously more environment friendly than conventional building materials due to their renewability and ease of availability while standardising the process of manufacturing can make them economical also. The Bureau of Indian Standards (BIS) have adopted the following four different types of coir fibre based panel board products developed by Central Institute of Coir Technology (CICT), Bangalore - a premier research institute under the Ministry of Micro, Small & Medium Enterprises (MSME), Government of India.

(1) Coir faced block boards

Coir faced block boards generally have wooden strips to form the core and is glued between two or more outer surface layers of coir ply boards to form a sandwich structure. The core strips can be also made of compressed coir fibre and border strips are mostly wood [68].

(2) Coir hard boards

Coir hard boards are manufactured from coir needled felt (non-woven) by the application of heat and pressure. The primary bond is derived from felting of fibres and synthetic resins are added as binders. Coir hard boards have uniform thickness and a density exceeding 800 kg/m^3 [67].

(3) Coir veneer boards

The coir veneer board uses a combination of coir fibre needled felt, veneer and jute fibres along with kraft paper. The blended mass of glued fibres is processed through needled felt machine to make a uniform mat in different densities and forms the outer skin of the board. The impregnated fibre mats are sandwiched with wood veneer in a press under controlled temperature and pressure [69].

(4) Medium density coir boards

The medium density coir board is a panel board manufactured from coir and jute fibres combined with synthetic resins like phenol formaldehyde binder. The boards are processed to a specific gravity of 0.5 to 0.9 using a hot press by applying heat and pressure for a required time [66].

2.7 Market potential of coir fibre boards

The growing concern over environment on account of deforestation has led researchers to explore the possible alternatives for wood and wood products where the majority of consumption accounts for applications like building and construction, interior decoration, furniture etc. The focus of research has shifted to composite boards manufactured from environmentally safe, renewable raw materials like agricultural wastes such as coir fibre, bagasse fibres, jute fibre, rice husk, pineapple leaf etc. [29, 30].

Coir fibre is a biodegradable natural fibre extracted from the husk of the coconut (Cocos nucifera L.) and is treated as a low value by-product from coconut harvest and its presence is limited to traditional markets for making mattresses, mats, carpets, ropes etc., but there is an increasing interest in finding additional applications and is now being used in various Fibre Reinforced Polymer (FRP) composites [18, 19].

Coir fibre is cheap, non-toxic and is regarded as a promising reinforcement in composite boards which enables coir composite boards to enjoy excellent potential as a substitute for wood or other engineered wood products and gain popularity in manufacturing household furniture and non-structural construction materials [29, 30].

Unlike various species of solid wood that are affected by micro-organisms, coir composite boards show a higher resistance to biodeterioration. However, they are still susceptible to biological attack under favourable conditions [70]. Fire- retardant chemicals in treated panels are susceptible to leaching from moisture when exposed to weathering, and it decreases the effectiveness of the chemical against biodeterioration [58, 71]. Different approaches and standards have been devised for manufacturing several of the coir fibre based composite boards and medium density coir board is a potential candidate in capturing market demand [66–69].

However the physical, mechanical, thermal and flame retardant properties of non-standardized boards are sparsely recorded. Risk of fire in a skyscraper or in railway coaches could be fatal and the strategy of fire control using construction materials inspires the use of fire retardant treatment for interior decoration/furnishing. An increase in research and development of new technologies has been observed in the fire retardant treatment of plastics, textile materials and even Nano-composites [72, 73]. However the transfer of these technologies to a commercially viable situation is found to be limited. The application of fire retardant/preservative chemicals could impart self-extinguishing characteristics of wood as well as fibre boards [26].

2.8 Flame-retardant coir fibre board

Coir fibre based composite boards manufactured to substitute particle boards or plywood is a relatively new application and their flammability characteristics have been investigated in this study. Using coir composite boards for potential applications makes it necessary to add knowledge on their flammability characteristics to widen their usability and range of applications [29, 30].

Coir fibre, being a lignocellulosic material, is flammable, and fires can propagate very fast [40, 41]. Coir fibre has evident environmental and sustainability benefits over its synthetic counterparts, and applying flame retardants could further help in meeting relevant fire standards. Reducing the risk of fire also contributes to substantial improvements in quality of living. A fire accident and the successive combustion of wood, or any wood substitutes, in habitable structures like high-rise apartment buildings and other skyscrapers can be life-threatening. To reduce the risk of flammability and improve safety, composite panel boards can be treated with fire-retardant chemicals [26, 74].

Fire resistance of composite fibre boards is an important concern as the market for structural panel boards expands; these products are being used in wider and more demanding applications, including the construction sector and the furniture industry. It is widely accepted that the fire performance of wood-based composites can be considerably improved through various chemical treatments, making them suitable for a variety of applications.

Fire retardant-treated (FRT) panel board products are gaining acceptance where an improved level of fire safety is desired [75]. Boron-based compounds are particularly effective for cellulosic materials, and their neutral pH, preservative effectiveness, and relatively low impact on mechanical properties make them a desirable choice as a fire retardant [70, 77, 78]. They are also well known for their excellent protective characteristics against biodegrading micro-organisms like termites [76].

The boron treated samples offer greater resistance to fire and flame propagation than untreated samples of wood and other engineered wood products like plywood which contain combustible organic carbon and hydrogen compounds [79, 80]. In a scenario where the usefulness and viability of the mass-scale production of biocomposite materials are still debated, it is critical to ensure that the material is compatible with various treatments and is capable of self-extinguishing any fire, making it suitable for a variety of functions.

Several attempts have been made to improve the fire resistance of natural fibre-based composite panel boards by treatment with different fire-retardant additives, and boron has been extensively reported for this purpose. The general procedures followed in for preservation and treatment of wood for extended life and fire resistance were expected to hold well for medium density coir fibre board samples as well, due to the presence of common constituents like lignin, cellulose or hemi celluloses in the structure [40]. Nevertheless, it is unlikely to conclude that all lignocellulosic materials behave in a generalized manner when treated with boron.

2.9 Types of fire retardants

Numerous chemicals having different structure and physical properties could impart fire resistant characteristics when used alone or in combinations. Combinations of different fire retardants with varying proportions can produce either an additive effect, synergistic effect or even an antagonistic effect. The synergy of combining fire retardant chemicals has gained considerable practical importance as they are usually less expensive. Fire retardant chemicals can be either chemically bonded reactive type or the additive type where the retardant is applied as a coating to the base material and the retention of retardant is by weak secondary bonding and not covalent bonding unlike the former [27, 42]. The various classes of fire retardant chemicals, particularly suitable for treatment of lignocellulosic materials are described in the next section.

2.9.1 Phosphorus based fire retardants

Phosphorus based fire retardants are particularly effective for cellulosic materials with high oxygen content. It works in the condensed-phase, by accelerating char formation, thereby avoiding the gas-phase of further volatile decomposition products. The char formation provides thermal insulation to underlying structure, limits oxygen diffusion and depriving the gas-phase of volatile fuel in order to terminate the combustion cycle. Phosphorus based fire retardants generally inhibit burning through formation of a coating on the condensed combustible layer, thereby cooling the surface and by limiting the supply of oxygen to suppress ignition. In addition, a carbon char formed, acts as a protective surface over the remaining condensed combustible layer from propagating flame [26, 42].

The steps involved in the mechanism are:

(i) Thermal decomposition of fire retardant to form phosphoric acid and

(ii) Esterification of acid and dehydration of oxygen-containing polymer to form char.

Materials treated with fire retardants like monoammonium phosphate (MAP), after exposure to elevated temperatures from solar radiation experienced in-service reduction of strength and as a result, boron treatments were advocated as the solution. Phosphorus based chemicals like elemental red phosphorus, phosphonates or phosphinates can also impart fire resistance; but ammonium phosphates are found to be more effective when applied on lignocellulose materials [26, 42].

2.9.2 Nitrogen based fire retardants

Nitrogen based fire retardants are used often in combination with phosphorus or sulphur containing fire retardants owing to the synergistic effect observed. Nitrogen used with phosphorus is found particularly effective for cellulose, polyester, and polyurethane products [27]. Some of the commonly used nitrogen based fire retardants are melamine and melamine phosphates. Guanidine phosphates are effective in wood and textiles, while melamine and melamine phosphates are used in intumescents, flexible polyurethane foams and in polyolefins. Melamine based fire retardants employ one or more of the following mechanisms to flame retard a material [26, 42].

- Production of volatile gases is prevented by cross-linking of molecular structures.
- The release of nitrogen containing compounds can dilute flammable gases produced during combustion.
- Synergy with phosphorus based fire retardants to enhance fire resistance.
- Release of ammonia during endothermic decomposition forms a superficial char layer, which acts as an insulating barrier to heat flow and inhibits further thermal decomposition.

2.9.3 Halogen based fire retardants

The relative effectiveness of halogens to resist the flame propagation is directly proportional to their atomic weights and follows the order of Fluorine < Chlorine < Bromine < Iodine [20]. High sensitivity of the carbon - iodine bond to thermal and photochemical degradation and high cost makes Iodine, the least preferable applicant [42]. The carbon-fluorine bonds are less likely to thermal degradation until relatively high temperatures are reached. Fluorine based flame retardant materials such as the chlorofluorocarbons are used as blowing agents in elastomers like polyurethane foam and polystyrene [27].

Bromine acts as a flame retardant in its gaseous phase. When exposed to flame, bromine dissociates from the material and this dissociation disperses heat, and the generated bromine gas acts as a thermal barrier around the material and prevents flame spread by inhibiting oxygen supply and slowing down the heat transfer. Among the four halogens, Chlorine and Bromine are the most widely used. Chlorine based fire retardants release Hydrochloric Acid over a wider temperature range and thus becomes less effective due to relatively lower concentrations. Chlorine compounds while low in cost and high in light stability are not as effective on a weight basis as the corresponding bromine compounds [27, 42].

2.9.4 Inorganic based fire retardants

Organic compounds like nitrogen based fire retardants evaporates under heat or fire, thus diluting the fuel gases and oxygen near the combustion source. Unlike these, inorganic fire retardants seldom vaporise under the influence of heat, instead, they decompose via endothermic reactions and release non-flammable compounds like water, carbon dioxide, sulphur dioxide, or hydrogen chloride. In the gaseous phase, inorganic fire retardants act by diluting the mixture of flammable gases and by protecting the surface of the substrate against oxygen attack. An inorganic fire retardant acts simultaneously on the surface of the solid phase by cooling the substrate via an endothermic breakdown process along with resisting the formation of pyrolysis products [42].

(1) Fire retardant boron compounds

Borates are naturally found in the earth as compounds of boron. Two of these minerals, borax and boric acid, are particularly well suited for use as flame-retardant materials due to their complementary flame-retardant characteristics. Their low toxicity to humans and the environment makes them qualified for use as flame retardants in residential settings [58, 74].

Boric acid (BA), borax (BX), and their combinations have been widely used in the fire protection industry due to their thermal resistance because they retards flame propagation on the wood surface [58, 81]. Boron compounds have relative advantages like, being a good preservative that resists decay, neutral pH value and no change in mechanical properties when compared to other flame retardant chemicals like phosphorous, hence they are often considered a good flame retardant [77]. In addition to the char forming catalytic effect, they have low melting point and form glassy films when exposed to high temperatures in fire. The formation of this glassy film barrier inhibits the flow of combustible volatiles to fire exposed surface.

The presence of BX tends to eliminate flame spread but promotes glowing, while BA enhances carbonization and suppresses smouldering or glowing; consequently, they exhibit a synergic effect when used in combination [82, 83]. The mixtures of BA and BX are complementary nature and adding more BX can compensate for the adverse effects of BA on board strength, as it buffers the acidic pH to neutral pH levels [84]. An estimated loading levels of at least 7.5% boron are required for the material to be flame-retardant post the treatment [77].

2.10 Mechanism of fire retardation

Presence of Fire retardant chemicals in a material inhibits or resists the continuous combustion cycle through various mechanisms. Based on the nature of fire retardants, they act chemically and/or physically in the solid, liquid or gaseous phase in order to retard the combustion processes [85].

2.10.1 Fire retardation by physical action

There are three major physical processes involved in the resistance to the propagation of flame. They are listed below as,

(1) Cooling process:

Any chemical reaction in which energy absorbing (endothermic) processes are initiated by additives and/or the chemical release of water, cools the substrate to a lower temperature that is required for sustaining the combustion process, e.g. magnesium hydroxide [27, 42, 85].

(2) Coating process:

By forming a protective layer or coating that shields the underlying combustible layer of material from heat and oxygen necessary for the combustion process, e.g. phosphorous and boron compounds [27, 42, 85].

(3) Dilution process:

By incorporating inert substances (e.g. Fillers) and additives that release inert gases, water or Carbon dioxide during decomposition and dilute the fuel in the solid and gaseous phases so that the lower ignition limit of the gas mixture is not exceeded, e.g. aluminium hydroxide releases water [27, 42, 85].

2.10.2 Fire retardation by Chemical action

The most significant chemical reactions that interfere with the combustion process takes place either in the solid and gaseous phases.

(1) Reaction in the gaseous phase:

Chemical reactions in the flame (i.e. Gaseous phase) can be interrupted by fire retardants, thus ending the exothermic processes which results in cooling of the system, reducing the supply of flammable gases and eventually suppressing the combustion, e.g. halogenated fire retardants [27, 42, 85].

(2) Reaction in the solid phase:

Polymers melt by breaking down (by the action of fire retardants) and are allowed to flow away from the flame, resulting in building up a layer of char on the polymer surface and thus shielding it against oxygen thereby providing a barrier against heat sources. For carbon-based materials, solid phase flame retardants cause a layer of carbonaceous char deposited over the surface. This layer of char is much harder to burn and prevents further burning, e.g. phosphorus compounds [27, 42, 85].

2.11 Methods of Applying Fire Retardant Treatment

The common methods adopted for waterborne preservative treatment of timber is taken into consideration, as boron compounds act as a good fire retardant apart from being a good preservative resisting decay and termite attack [77]. Application of boron compounds as a fire retardant/ preservative over coir composite boards is further elaborated. Preservative solutions are generally applied either by coating on the surface of the material, or by incorporating into the structure using a vacuum pressure impregnation technique [86, 87]. Since there appear to be no relevant standards referring to fire retardant coir composite boards, the existing standard for Fire Retardant Plywood has been taken into consideration. In order to conform to the standards of IS 5509:2000 the following types of treatment are applicable.

2.11.1 Pressure impregnation processes

The application of pressure to force fire retardant into the material in a pressurized cylinder is the most efficient way of treating to obtain good absorption values. The treatment is done in a pressure plant equipped with pressure cylinder, gauges, storage tank, pressure pumps and vacuum. The most common processes for treatment are the full cell process (Bethell) and the empty cell process (Rueping) [86, 87].

(1) Full-Cell Processes

The full-cell (Bethel) process is adopted when the objective is to obtain maximum retention of preservative. Preservative retention is controlled by regulating the concentration of preservative in treating solution. The sample is sealed in the pressure cylinder and a preliminary vacuum of not less than 560 mm Hg is applied for 30 minutes to remove the air in the cylinder and in the void spaces of sample. The preservative (at ambient temperature) is pumped into the cylinder without breaking the vacuum and after filling the cylinder, pressure is applied to get the maximum or required retention of the fire retardant is made [86, 88, 89].

A pressure of 2 to 12 kg/cm^2 is applied for a specified time, and after that the fire retardant solution is pumped out from the cylinder and a final vacuum is used to remove the dripping solution from the sample. Initially the vacuum removes air from the cavities of the composite board to give space for the fire retardant solution, and is then forced deep into the structure under high pressure. The degree of penetration depends on the permeability of the material and while impregnating, the structure of coir composite board is viewed as being similar to that of a sponge, with cell cavities and cell walls. The pressure and temperature applied shall be limited so that there is no damage to the structure of the specimen [86, 88, 89].

(2) Empty-Cell Processes

The empty-cell processes are mainly used for materials which require deep penetration with relatively lower retention of fire retardant chemical and is also recommended for treatment with oil borne preservatives. There are two empty cell processes, named after Rueping and Lowry; even though both employ the force of compressed air to drive out a portion of the fire retardant chemical absorbed during the pressure process, the Rueping empty cell process is more popular and often called empty cell process with initial air.

Compressed air is blown for a few minutes into the treatment cylinder, which contains the sample. The air pressures employed generally range between 25-100 psi (172-689 kPa), depending on the required net retention of chemical and afterwards the preservative is pumped into the cylinder. As the preservative is forced into the treatment cylinder, the air inside escapes from the treatment cylinder into an equalizing tank (Rueping tank) at a rate at which constant pressure is maintained in the cylinder.

After the treatment, cylinder gets filled with preservative, the pressure is raised and kept above initial air pressure until the specimen absorbs enough preservative for the required retention. At the end of the pressure period, the fire retardant solution is drained from the cylinder, and surplus fire retardant is removed from the sample with vacuum. About 20 to 60 % of the total preservative pumped into the cylinder can be recovered after the vacuum has been applied [86, 88, 89].

2.11.2 Non pressure processes

There are numerous processes that do not use pressure and differs widely in the level of penetration, retention and degree of protection that can be achieved. The service life of non-pressure treated materials become comparable to that of pressure treated materials when similar retention and penetration levels are achieved. Non-pressure processes are particularly useful where expensive methods are irrelevant as only little protection is required or when more rigorous treatments are impractical [88].

To apply a protective surface coating to the sample, it can be painted, sprayed or dipped into a solution of fire retardant, all of which are modifications of soaking treatment. Soaking treatment is carried out by submerging the specimen in fire retardant solution for a sufficiently long period until the required absorption of the retardant is obtained [89, 90].

(1) Brushing/spraying

This method requires minimum solution strength of 3% preservative applied on the sample. Depending on the moisture content and surface roughness of the sample, up to three coatings can be applied, however, this method has only limited effect on fire resistance [86, 87].

(2) Soaking/cold soaking

The sample to be treated has to be soaked in a solution having 3-5% fire-retardant chemical for a minimum of 24 hours in this method. Soaking gives better fire protection than brushing or spraying. The sample must be dry and the treatment is best done during the summer when temperatures are warm [86, 87].

(3) Hot and cold bath

Hot and cold bath treatment is considered as the most effective non pressure treatment and owing to the initial and maintenance cost for pressure treatment plant, is recommended for operations on a smaller scale. The hot bath has to be equipped with a heating source and in order to prevent corrosion from preservatives; the bath is made of stainless steel. The sample is submerged in the preservative and heated for up to 3 hours at 80 ^{0}C to 105 ^{0}C.

Then the sample is transferred to the cold bath filled with the same solution maintained at 38 ^{0}C, where it is again kept for 8-24 hours. In both tubs the sample is kept totally immersed. In this method during the hot bath, air entrapped in the sample expands and is driven out and during the cold bath, the residual air in the specimen contracts, creating a partial vacuum in the cells that allows the preservative to penetrate into the structure. Retentions of 40-140 kg of preservative per cubic metre have been achieved [86, 87].

(4) Diffusion processes

Simple diffusion process - It is a simple but time consuming method in which the sample is immersed in a solution containing 20% boron at 40^{0}C for a minimum period of 20 minutes. Diffusion occurs during storage of samples, while they are stacked and covered in polythene sheets (to retain moisture) for a period of 6-10 weeks. An overall retention of about 3% boron can be achieved in the oven-dry sample weight over this period [91].

Double diffusion process - The sample is immersed into a hot (80 ^{0}C) fire retardant solution for 3 to 6 hours and cooled overnight. After 24 hours the sample is transferred to a tank containing a cold solution, where it is kept for up to 48 hours. The double diffusion process gives better retention rates and can be applied to samples without seasoning, thereby reducing processing time as well as the danger of fungal or insect attack [77, 88, 91]. All these methods act towards reducing the rate of heat release during combustion, delay in time to ignition, and reduction in flame spread.

2.12 Fire-retardant treatment of wood and composites: Review

Gon et al. (2011) [37] developed a jute fibre based composite board to substitute wood and found that humidity and moisture adversely affects the tensile strength of the boards and was prone to de-lamination. Lowden & Hull (2013) [26] reviewed different types of fire retardants and their mechanism of fire inhibition. They also reviewed the various techniques for studying fire retardant treated timber.

Turekova et al. (2011) [92] investigated the influence of treating mono ammonium phosphate (MAP) on lignocellulosic materials and found optimum concentration of MAP. Tests like Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC) and Limiting Oxygen Index (LOI) were used in the process.

Baysal (2011) [79] investigated on combustion properties of Calabrian pine wood specimens impregnated with aqueous solutions of Ammonium sulphate (AS) and di-ammonium phosphate (DAP). DAP and AS were impregnated into wood at 10, 20 and 30% concentrations. Results indicated that chemical treatments improved the combustion properties of specimens and those impregnated with 30% DAP gave the best results.

Barnes & Murphy (2005) [93] treated medium density fibre boards, using vapour boron and observed no negative effects on composite bending and tensile properties.

Ozcifci et al. (2007) [57] investigated the fire properties of laminated veneer lumber (LVL) treated with some fire retardants. Boric acid (BA), borax (BX) and di-ammonium phosphate (DAP) were used as impregnation chemicals. LVL were manufactured using the melamine formaldehyde (MF) and phenol formaldehyde (PF) adhesives. Results indicated that the mass loss was found to be lower for the specimens using MF treated with DAP and (BA-BX) mixture.

Lebow et al. (2005) [94] studied the penetration of boron on different species of wood with pressure treatment and found that boron in disodium octaborate tetrahydrate (DOT) could penetrate the samples for more than 10 mm immediately after treatment.

Yalinkiliç et al. (1998) [84] used a mixture of BA and BX in the ratio of 5:1 on particle boards made from waste tea leaves and observed that the treatment improved its fire resistance. The mixture of BA and BX is complimentary as the adverse effect of BA on board strength is suppressed by adding small amounts of BX, by buffering its acidic pH to neutral pH levels, while BA reduced the glowing or smouldering effect of BX.

Saxena (2014) [27] reported that Boric acid and Borax were found to be helpful for imparting fire proofing qualities such as lower flammability, reduced rate of burning, resistance to flame spread or higher flame penetration time in FRT panel boards.

Nagieb et al. (2011) [58] studied the physical and mechanical properties of flame retardant saw dust composite boards treated with the chemicals boric acid (BA) and borax (BX) and observed that the boards exhibited better flame retardance after treatment, however the bending strength decreased after treatment.

Wang et al. (2004) [95] treated wood with boric acid and the gas chromatography - Fourier Transform Infrared Spectroscopy (GC–FTIR) spectra indicated that compounds generated by treated wood is different from untreated wood. They concluded that boric acid promotes dehydration and other oxygen-eliminating reactions of wood at relatively low temperature (approx. 100-300^{0}C) and partly contributes to the effects of promoting charring and fire retardation of wood.

Laufenberg et al. (2005) [96] evaluated fire and bending properties of block boards with various fire retardant treated veneers. The veneers were treated with either boric acid (BA), disodium octoborate tetrahydrate (DOT), alumina trihydrate (ATH), or a BA/DOT mixture. It was found that the treatments resulted in significant reduction in the flame spread rate and had little effect on flexural strength, but flexural stiffness were reduced.

Levan & Tran (1990) [77] investigated the role of boron in providing flame retardancy to wood. Different loading levels of borax-boric acid, ranging from 0 to 20 percent by weight, were applied to wood. It was estimated from fire test results that loading levels of more than 7.5 percent add-on of BX and BA are required for the material to be flame retardant.

Pedieu et al. (2012) [80] investigated the fire retardant performance of wood particle boards, treated with three percentages (8, 12 and 16%) of boric acid. Particles were treated during blending with adhesive. Particle boards, treated with 16% BA showed the best fire-retardant properties, particularly in terms of weight loss, flame spread speed and after flame time. The use of BA decreased the flame spread speed and also improved the internal bond and thickness swelling of particle boards.

2.13 Scope of the Research

The coir fibre market has witnessed the entry of several new countries like Belgium, China, Germany, Indonesia, Spain, United States, and Venezuela in recent years, which will likely drive down the price of coir fibre. The global coir fibre market is diminishing, except China. As a result, more countries are involved in processing their own coir fibre with the trends in coir exports. However, with an increase in export of a variety of different coir products to many different countries, the global coir product market seems promising [14, 34].

In addition to conventional products like coir, carpets or mattresses, the future also encourages export of coir fibre based composite boards. The development of an environmental friendly technology that turns coir fibre (agricultural residues) into quality, value-added composite products using formaldehyde-based resins are in practise now a days.

Implementation of such technology would result in efficient utilization of coir fibre as a sustainable resource for manufacturing commodity products like particle boards and fibreboards that could substitute wood and other engineered wood products. It could also help release the pressure on our forests to meet growing demands [35, 36].

The primary applications of wood include construction for acoustic reasons. Large amounts of wood are also used when making furniture, speakers and soundproof rooms, due to the merits of wood in terms of acoustic performance, function, design and texture. The development of alternative materials that could reproduce the desired properties of wood for specific applications is the current requirement. Recently a lot of focus has been made on the technological developments that could easily contribute to the efficient use of wood resources [28–30].

Coir fibre-based panels act as an excellent substitute to wood in view of their low cost, availability and pollution free production. As a result of these advantages, natural fibre reinforced composite boards could gain popularity in manufacturing household furniture and non-structural construction applications [32].

In order to enhance the development and commercialization of coir reinforced composite boards, improved laboratory-industry linkages are needed. Coir reinforced composite boards will have good requirement in India as a substitute for plywood and medium density fibre boards, for low-cost housing needs. Scope of the present work is to develop techniques to reduce fire risk and improve thermal conductivity and fire retarding properties of coir fibre based panels [32].

2.14 Research Objectives

- ⋆ To develop a suitable manufacturing processes for developing fire resistant coir fibre boards.
- ⋆ To explore the potential of coir fibre-based composite boards and to develop fire retardant treated coir fibre boards without compromising structural properties.
- ⋆ To identify a simple and economical method to improve fire retardation properties of coir fibre boards.
- ⋆ To investigate the possibilities of chemical treatment to minimize flame propagation, while promoting sustainability.
- ⋆ To evaluate the effect of boron compounds on the fire resistance properties of coir fibre boards.
- ⋆ To assess the change in different mechanical and physical properties of coir fibre boards after fire retardant (boron) chemical treatment.

2.15 Conclusion

A broad range of fire retardant chemicals along with their mechanism for fire inhibition and methods of application have been researched in the recent years. It is observed that the combinations of phosphorus and nitrogen continue to prove themselves as very powerful fire retardants for ligno-cellulosic materials, while borax, boric acid and other retardants which supplies boron particles to the samples deliver fire resistant characteristics without affecting much of its mechanical properties and preserves the composite from decay and termite attack.

In the near future silicon may become a possible replacement to boron, operating via similar mechanisms combining economy, flammability reduction and smoke inhibition with less toxic fume. These materials and methods have shown promising results in the laboratory. There is scope for more research in this area to develop a fire retardant coir composite board with self-extinguishing characteristics which can be used for a wide range of applications.

CHAPTER 3

METHODOLOGY

3.1 Design and Analysis of Experiments

Formulation of an experimental design is crucial as the validity of an experiment is directly affected by its construction and execution. Design and analysis of experiments (DOE) in this research involved the planning and execution of experiments that could generate appropriate data on flame resistance with respect to the amount of Fire-Retardant (FR) chemicals used for treatment, which is the problem under investigation. It also includes the subsequent process of drawing out suitable inferences from the available data. The structuring of dependent and independent variables, the choice of their levels in the experiment and the method of recording and tabulation of data are all details that go with the design and analysis of this experiment [97–99].

3.2 Principles of experimentation

There are three basic principles, (i) randomization, (ii) replication and (iii) local control. These three principles increase the accuracy of the experiment and provide a valid test of significance. It is obvious that any number of repeated measurements would not overcome systematic error, whereas it is very likely that the random errors would cancel out with repeated measurements. These principles are devices to avoid the systematic error and to control the random error [97, 98].

3.2.1 Randomization

In a randomized experimental design, samples are randomly assigned to experimental groups. Randomization in experiments along with expert judgement, guarantees the elimination of systematic error (experimental bias). It further ensures that any error component that persists in the observations is purely random in nature, which provides the basis to make a valid estimate of random fluctuations which is essential in testing the significance of genuine differences. The variations of randomized experimental designs include Completely Randomized Design and Randomized Complete Block Design [97, 98].

3.2.2 Replication

Replication is the repetition of experiments on a large group of subjects under identical conditions but in the context of experimental design, it refers to the number of distinct experimental units under the same treatment. Replication improves the significance of experimental results, as the long-term averaging effect helps to determine the true effect of treatment. Replication also helps in reducing variability in experimental results and increases their significance and confidence level with which conclusions are drawn about an experimental factor. Replication, with randomization, provided the basis for estimating error variance. In the absence of randomization, any amount of replication does not lead to a true estimate of error [97, 98].

3.2.3 Local Control

Local control means the control of all factors except the ones about which we are investigating. Local control is a device to reduce or control the variation due to extraneous factors and increase the precision of the experiment. In order to reduce experimental bias, the experiment has to be conducted in identical conditions and controlled for outside variables favouring specific outcomes. Without controlling outside variables, it is unlikely to conclude that the effects observed were a direct result of treatment. This type of local control to achieve homogeneity of experimental units, will not only increase the accuracy of the experiment, but also help in arriving at valid conclusions. In short, it may be mentioned that while randomization is a method of eliminating a systematic error (i.e., bias) in allocation, thereby leaving only random error component of variation, the other two principles; replication and local control keep this random error as low as possible. All the three however, are essential for making a valid estimate of error variance and to provide a valid test of significance [97, 98].

3.3 Completely Randomized Design

A completely randomized design (CRD) is the one where all treatments are assigned completely at random so that each experimental unit has the same chance of receiving any one treatment. For a CRD, any difference between experimental units receiving the same treatment is considered as experimental error and hence, CRD is more appropriate for experiments with homogeneous experimental units, where the environmental effects are relatively easy to control. CRD is rarely used for field experiments, where there is generally a large variation among experimental plots in environmental factors [97, 98].

3.3.1 Analysis of Variance

Analysis of variance (ANOVA) is a technique of partitioning the overall variation in the responses observed in an investigation into different assignable sources of variation, some of which are specifiable and others unknown. Further, it helps in testing whether the variation due to any particular component is significant as compared to residual variation that can occur among the observational units. The two sources of variation among observations obtained from a CRD are the variation due to treatments and experimental error. The relative size of the two is used to indicate whether the observed difference between treatments is real or is due to chance. The treatment difference is said to be real, if the treatment variation is sufficiently larger than experimental error [97, 98].

3.4 Randomized Complete Block Design

The randomized complete block design (RCBD) is suited for experiments where the number of treatments are not large and there exists a conspicuous factor based on which homogenous sets of experimental units are identified. The primary distinguishing feature of RCBD is the presence of blocks having equal size, each of which contain all the treatments. A randomized block design is preferred over a completely randomized design, when specific differences among groups of subjects within an experimental group is known. In randomised block design, subjects are initially divided into homogeneous blocks and are then randomly assigned to a treatment group. In a block design, both control and randomization are considered [97, 98].

3.5 Experimental design

Experiments in this research work used fire-retardant chemical treatment of the subjects (coir fibre board samples) and analysed their observed response. All experiments in this research are single factor experiments. As greater number of replications helps to improve precision in the experiment results, it was decided to test three specimens, n'=3 replicates, for each level of the factor. In this research a completely randomized design CRD was adopted for chemical treatment with Fire retardant (FR) solution. The process of random allocation of samples for FR treatment was done using Minitab software version 17. Objects were assigned to treatment groups using Minitab, where the SAMPLE command was used to select a random sample of a specified size from a list of objects. Fire retardant treated (FRT) coir fibre board samples were randomly assigned through CRD to various tests for assessing the improvements in thermal stability like Thermogravimetric Analysis (TGA) and Differential

Scanning Calorimetry (DSC).

FRT coir fibre board samples were also subjected to various tests to determine their physical and mechanical properties by random allocation through CRD. The experiments conducted to decide the most economical combination of FR solutions that imparted fire resistance to coir fibre boards were designed using RCBD. Four blocks were identified for each type of fire resistance tests employed in this research, namely flammability, flame penetration, rate of burning and resistance to flame spread.

The significance threshold was set at a level of significance of $\alpha < .05$, for all the four fire performance tests. The value of sum of squares were used to compute the mean squares and F values. The tabular F values from the Table of F-statistics (P = 0.05), for corresponding degrees of freedom at the 5% level of significance were recorded. The computed F values were compared against the tabular F value to decide on the significance of difference among treatments. The variation due to treatments are considered significant, only if the computed F value is larger than the tabular F value at 5% level of significance.

3.6 Experimental Methods

Coir fibre board samples were subjected to hot and cold bath treatment using an aqueous solution containing a combination of laboratory grade BA, H_3BO_3 and BX, $Na_2B_4O_7.10H_2O$. Physical properties like density, moisture content, thermal conductivity and thermal resistance of both treated and untreated coir fibre board samples were determined and the effect of FR treatment was ascertained.

A detailed flow chart showing the experimental methodology adopted for the research is shown in figure 3.1.

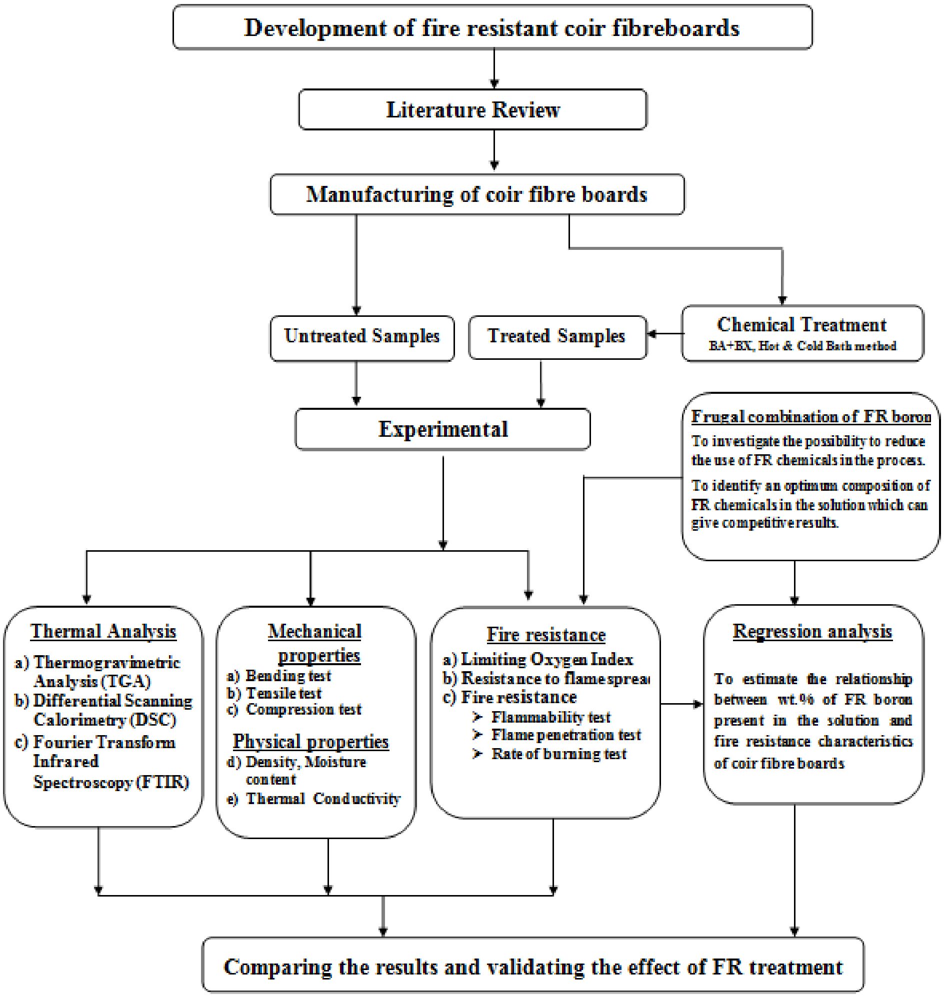


Figure 3.1: Experimental Methodology

The changes in mechanical properties of coir fibre board samples after FR chemical treatment were assessed by subjecting them to tensile, bending and compression tests. The chemically treated samples were also subjected to various tests like Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC) and Fourier Transform Infrared

Spectroscopy (FTIR) for assessing the improvement in thermal stability. Various fire resistance tests like Resistance to spread of flame test, Flammability test, Flame penetration test, Rate of burning test and Limiting Oxygen Index test were performed to assess the improvement after chemical treatment. A regression analysis was also done to predict the fire resistance of coir fibre board samples with respect to the amount of FR boron retained in the board after chemical treatment.

3.7 Preparation of coir fibre boards

Medium density coir fibre board samples having a size of 300 x 300 x 12 mm, were manufactured as per IS 15491:2004 by hot pressing coir fibre mats impregnated with phenol formaldehyde resin. Figure 3.2 shows medium density fibre boards manufactured from coir fibre.

Figure 3.2: Medium density fibre boards manufactured from coir

The manufacturing of boards were done in close cooperation with Central Institute of Coir Technology (CICT) at their laboratory facility at Peenya, Bangalore.

The various stages involved in the manufacturing of coir fibre boards can be described as follows.

3.7.1 Coir fibre extraction

Coir fibre (brown fibre) required for manufacturing the boards were mechanically extracted from mature dry coconut husks using a coir defibering machine as per IS 9308 [48]. Figure 3.3 shows the mobile fibre extraction machine that was used to separate coir fibre from coconut husks.

Figure 3.3: Mobile Fibre Extraction Machine

3.7.2 Fibre conditioning

The extracted fibre was classified and cleaned before manufacturing coir felt. The brown fibre separated from husk was first soaked in water for about 72 hours. It was then thoroughly washed and dried in sunlight to remove moisture content [67].

3.7.3 Coir felt preparation

Coir fibre was fed through a non-woven needle punch machine to make a uniform coir fibre mat (felt) of sufficient density [67]. The uneven edges of the prepared coir felt were cropped before further processing. The density of coir needle felt can be controlled by the amount of fibre going through the needle board or by overlapping the needled felt. Figure 3.4 shows the needle punch felt machine (Fehrer, Stylus NL9/S) that stitches coir fibre in a random fashion to form fibre mats.

Figure 3.4: Needle Punch Felt Machine

3.7.4 Resin impregnation

The coir fibre mats produced by the needle punch felt machine were then impregnated with phenol formaldehyde resin using a glue spreading machine and was dried before further processing. The felt was immersed in a bath of phenol formaldehyde resin while being pulled off at a constant feed rate using a pair of rotating rollers and finally rolled up to form logs of coir felt.

The figure 3.5 shows the glue spreading machine which is used to impregnate the coir fibre mat with phenol formaldehyde resin.

Figure 3.5: Glue Spreading Machine

3.7.5 Hot pressing

The figure 3.6 shows the hydraulic hot press in action with coir fibre mats pressed to form composite boards.

Figure 3.6: Hydraulic Hot Press

The prepared samples were then subjected to chemical treatment. After drying, the impregnated fibre mats were stacked one over the other for the required thickness and is pressed at a temperature of 140^0C and a pressure of $12kg/cm^2$ for about five minutes with a hydraulic hot press to form coir fibre boards [67].

3.8 Fire retardant chemical treatment

The sample surface was cleaned to remove any impurities, followed by chemical treatment with a water-borne preservative solution containing a mixture of Boric Acid (BA) and Borax (BX) in the ratio 1:1.5 by their weight, which accounted to around 35% fire retardant (FR) boron for 65% water to form a solution that yielded the maximum solubility in water.

Even though longer soaking periods would result in better absorption and penetration of the fire retardant, it may also result in thickness swelling of coir fibre boards. Therefore a modified hot and cold bath method of FR treatment was employed on the coir board samples. In order to protect the material from thickness swelling, the soaking periods were reduced which resulted in a quicker treatment process.

The method of hot and cold bath is found to be an effective method in seasoning coconut palm stem timber. Table 3.1 shows the required soaking time for preservative treatment of coconut palm timber. In line with this a modified hot and cold bath method was employed for the fire retardant chemical treatment of coir fibre boards.

Table 3.1: Soaking time to preserve coconut timber [86]

Timber Thickness (mm)	**Soaking Time (hours)**	
	Hot bath	**Cold bath**
12	1	8 – 12
25	2	Max. 12
50	Determined during process	18
Thicker		24

The dry sample was first dipped in the FR solution at 100^0C for 30 minutes and then transferred to the cold bath where it is soaked for a further 6 hours at about 38^0C followed by drying in the sunlight until the moisture content is removed. During hot bath process, the rapid heating of samples resulted in the expansion of air molecules present in the sample that were subsequently driven out, whereas the residual air present in the samples contracted during rapid cooling during the cold bath process which created a partial vacuum and allowed the preservative boron to penetrate [86].

CHAPTER 4

THERMAL STABILITY OF FR TREATED COIR FIBRE BOARDS

Wood products do not require supplemental treatment with fire retardant (FR) chemicals in most applications, whereas coir fibre boards lack fire resistant properties which decreases their utility value. However, where situations demand a higher level of fire safety, if fire retardant treated, they can substitute traditional non-combustible materials and thereby widen their utilization options [80, 96]. This chapter describes the improvements in thermal stability of coir fibre board samples after FR treatment with boron compounds.

4.1 Experimental

Medium density coir fibre boards were manufactured by hot pressing of phenol-formaldehyde impregnated coir fibre felt. A mixture of Boric Acid and Borax were used to chemically treat the fibre boards. A modified hot and cold bath method was employed on the coir fibre board samples to impart fire resistance. Improvements in thermal stability of coir fibre boards subjected to fire retardant treatment were evaluated through Thermogravimetric analysis (TGA) and Differential scanning calorimetry (DSC) tests. Fourier Transform Infrared Spectroscopy (FTIR) was used to investigate chemical bonds present in both treated and untreated samples.

4.1.1 Thermogravimetric Analysis (TGA)

The analysis was carried out on 7-14 mg of treated and untreated powder samples at a heating rate of 10^0C/min with air as the atmosphere using a thermogravimetric analyser (PerkinElmer STA 6000). The samples experienced thermal decomposition in a programmed temperature range of 40^0C to 700^0C and continuous weight loss and temperature were recorded and analysed.

4.1.2 Differential Scanning Calorimetry (DSC)

DSC analysis was carried out on 5-7 mg of treated and untreated powder samples at a heating rate of 10^0C/min using a Differential Scanning Calorimeter (METTLER STARe SW 8.10) with air as the atmosphere. All measurements of each sample were made in a programmed temperature range of 30^0C to 300^0C.

4.1.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy uses electromagnetic radiation frequencies between 4000 cm^{-1} and 400 cm^{-1} (wavenumbers) to determine the organic structure of compounds. The radiation in this region is absorbed by inter-atomic bonds and different chemical bonds absorb varying intensities at varying frequencies.

The infrared spectra for both untreated and FR treated coir fibre board samples in powdered form were recorded in an FTIR Spectrophotometer with a transmittance range of scan from 4000 cm^{-1} to 500 cm^{-1}. FTIR spectroscopy collected the absorption information and was rendered in the form of a spectrum. The chemical bonds in the sample after chemical treatment was investigated and compared using FTIR spectroscopy [58, 59].

4.2 Results and Discussion

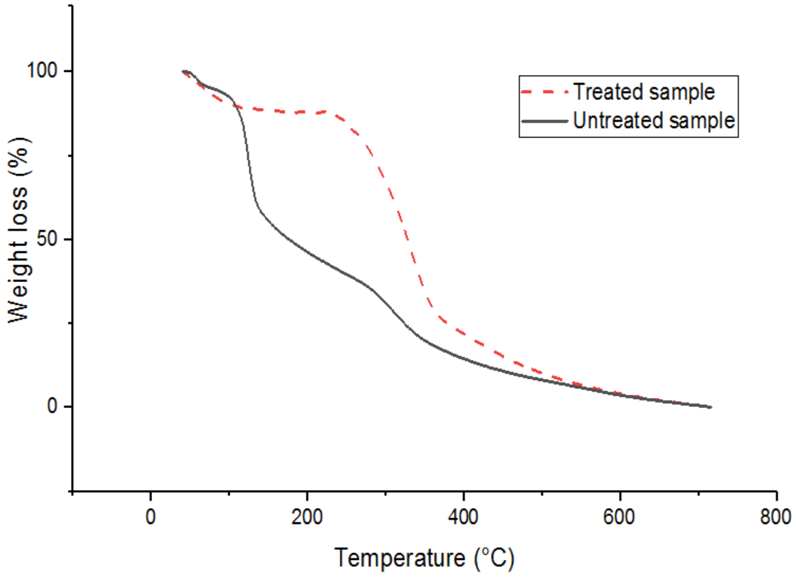
The thermal stability of FR treated coir fibre boards were evaluated through TGA and DSC tests. Weight loss (%) and derivative weight loss (%) of both samples with respect to temperature rise were plotted after TGA. DSC analysis provided the heat flow (mW) during the experiment against temperature rise and the endothermic and exothermic peaks are located and crystallisation enthalpy is determined.

FTIR Spectroscopy technique was used to investigate chemical bonds present in both FR treated and untreated samples. The presence of boron in FR treated coir fibre board samples were confirmed from FTIR spectrum.

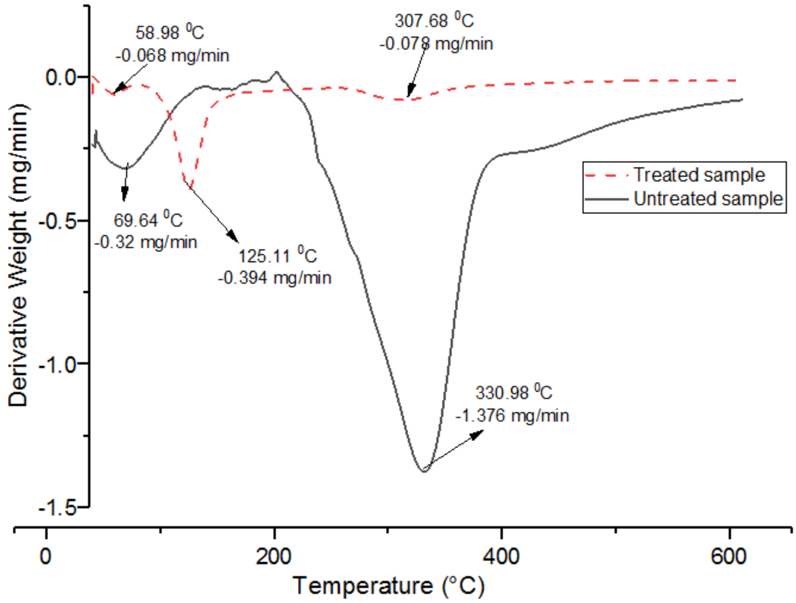
4.2.1 TGA of coir fibre boards

The derivative weight (mg/min) of the samples were analysed and it is observed that the untreated samples show a total reduction of 1.696 mg/min of which 1.376 mg/min is at a temperature of 330.98^0C, and a reduction of 0.32 mg/min at 69.64^0C. Whereas treated samples exhibit very limited derivative weight loss with a total of 0.54 mg/min.

Figure 4.1 represents the TGA curves of boron treated and untreated medium density coir board samples.



(a) Weight loss vs. Temperature



(b) Derivative weight loss vs. Temperature

Figure 4.1: Thermogravimetric analysis of coir fibre board samples

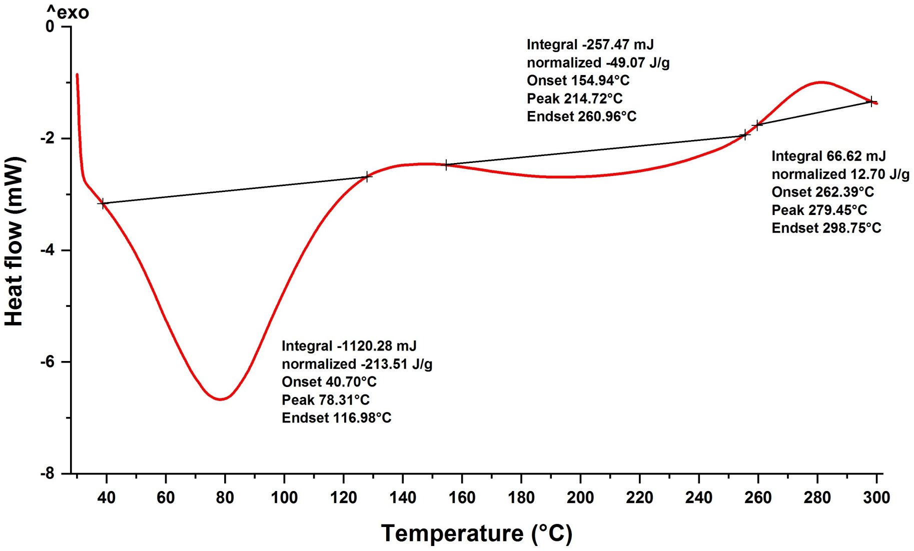
Figure 4.1 (a) depicts the weight loss versus temperature curve from TGA that shows a steep decline for the first weight loss for untreated coir board samples between 110^0C to 132^0C which is attributed to the dehydration of samples; [59] however the boron treated samples show very limited weight loss during the temperature range between 110^0C to 132^0C which indicated that the boron treated samples are more thermally stable after the treatment.

Figure 4.1 (b) depicts the derivative weight loss versus temperature curve from TGA which shows that the decomposition of untreated samples is at a faster rate between 251^0C and 365^0C but the boron treated coir fibre board samples show very little signs of accelerated decomposition and almost follow a steady state of weight loss. A strong resistance for thermal decomposition is observed between the temperature range 278^0C to 335^0C for the boron treated sample.

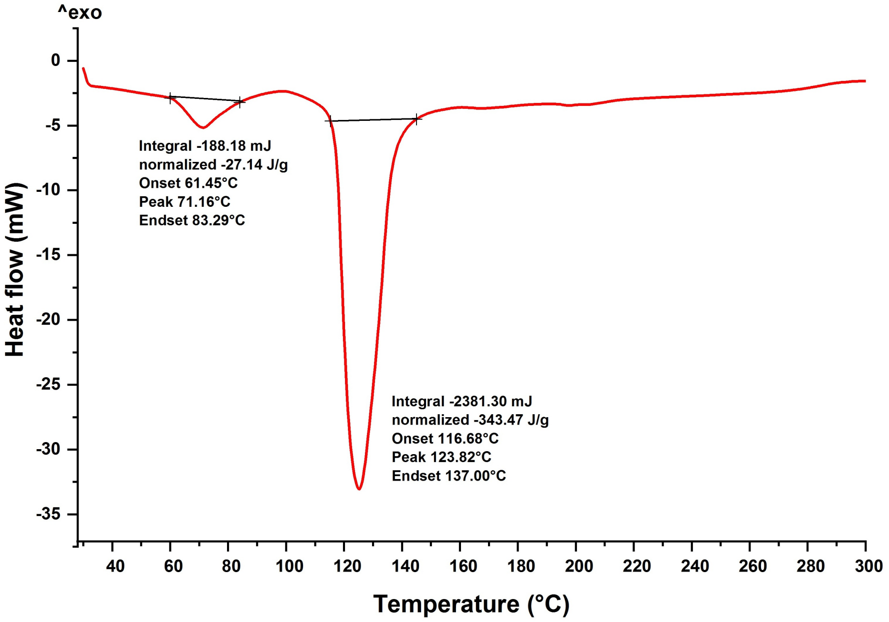
The treated samples exhibit derivative weight loss at three different temperature points first at 58.98^0C, a reduction of 0.068 mg/min and then at 125.11^0C, a reduction of 0.394 mg/min both accounting to dehydration and lastly at 307.68^0C, a reduction of 0.078 mg/min, altogether a total of 0.54 mg/min only. Both the curves suggest that thermal stability of chemically treated coir fibre board sample is significantly more than untreated samples.

4.2.2 DSC analysis of coir fibre boards

The thermal stability of both FR treated and untreated medium density coir fibre board samples characterised by DSC is shown in Figure 4.2. The DSC curves for untreated coir fibre boards are shown in Figure 4.2 (a) whereas, Figure 4.2 (b) shows the DSC curves for FR treated coir fibre boards.



(a) Untreated samples



(b) FR boron treated samples

Figure 4.2: DSC curves for Medium Density Coir Board samples

DSC analysis of both untreated and FR treated coir fibre board samples exhibited a broad endotherm in the temperature range of 50^0C to 150^0C which indicates the presence of water molecules in the fibres. The first and second endothermic peaks for boron treated sample falls in this temperature range. Thermal decomposition of wood fibres (cellulose, hemicelluloses and lignin) are initiated at temperatures around 200^0C to 360^0C. The highly crystalline cellulose is thermally more stable than amorphous hemicelluloses and lignin. Acetyl groups in hemicelluloses make it least thermally stable and lignin decomposes slowly over a wide range of temperature in line with the results obtained previously.

The second endothermic peak for untreated sample is 214.72^0C. The second endothermic peak, at a higher temperature than 200^0C, indicated decomposition temperature of the wood fibre present in the untreated sample in line with the previously obtained results. The first decomposition temperature is 61.45^0C for the treated sample and it exhibits a higher value than the untreated sample with first decomposition temperature 40.70^0C, indicating that treated sample is more thermally stable.

The crystallization enthalpy ($-\Delta H$) of the treated sample is determined as 370.61 J/g and is higher than that of untreated sample which is 262.58 J/g. The higher crystallization enthalpy exhibited by treated samples is due to the presence of flame retardant boron particles with uniform dispersion in FR treated coir fibre boards. Larger the area under the endothermic peaks, the more efficient they will be in protecting polymeric materials against thermal degradation because heat is absorbed by the sample during enthalpy change [58, 59].

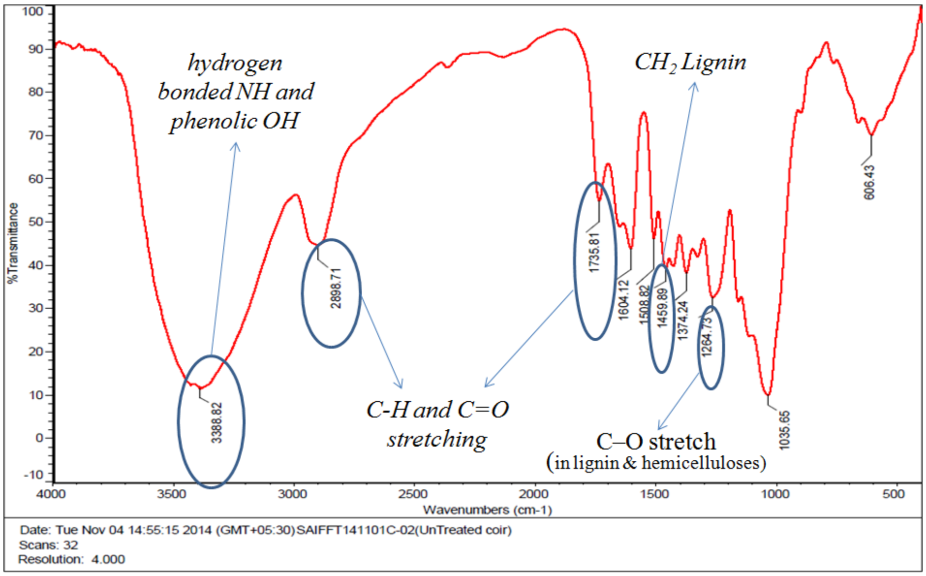
4.2.3 FTIR spectroscopy of coir fibre boards

The Infra-red (IR) absorption range for a wide variety of chemical bonds in various environments are quickly referenced through tables of IR assignments for absorption peaks and compared to the spectrum under investigation. Frequencies at which there are absorptions of IR radiation can be correlated directly to the bonds present in the samples [58, 59]. Infrared assignments for absorption peaks of medium density coir fibre board samples are shown in Table 4.1.

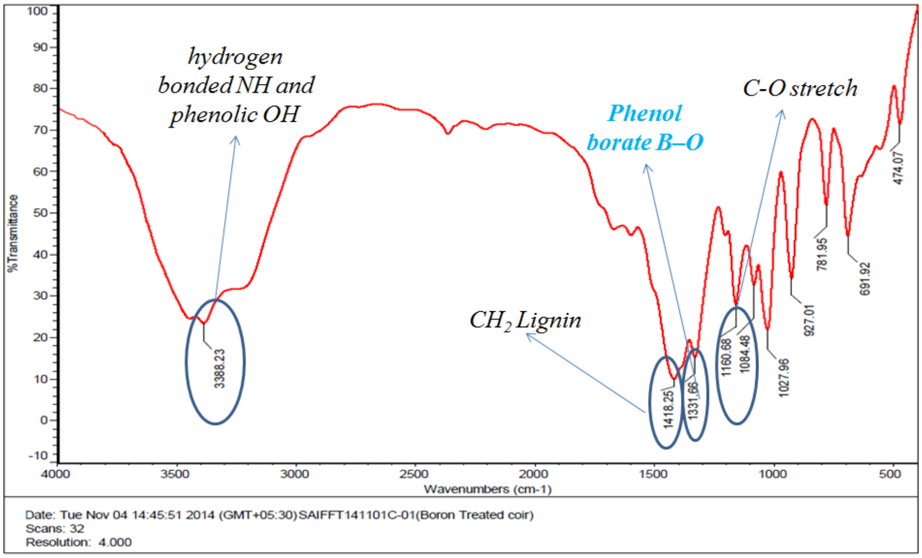
Table 4.1: Infrared assignments for absorption peaks [58, 59]

Wave number (cm^{-1})	**Assignment of peaks**
3500 - 3300	O–H stretching
2800 - 3000	C–H stretching in methyl and methylene groups
2920 - 2850	Aliphatic CH_2 asymmetric stretch
1750 - 1730	C=O stretching in carbonyl
1640 - 1618	C=C alkenes
1510 - 1504	C=C aromatic skeletal vibrations
1462 - 1425	CH_2 lignin
1350 - 1310	Phenol borate B-O
1260 - 1234	C-O stretching vibration in lignin and hemicelluloses
1170 - 1153	C-O-C asymmetric bond
756	Out-of-plane ring deformation
691	COH out-of-plane ring bending

Figure 4.3 (a) illustrates the absorption bands of medium density coir board samples treated with boron compounds and Figure 4.3 (b) illustrates the absorption bands of untreated medium density coir board samples.



(a) Untreated samples



(b) FR boron treated samples

Figure 4.3: FTIR spectrum for medium density coir fibre boards

FR boron treated medium density coir board samples show a strong absorption band at 3388.23cm^{-1}. This band is broad and typical of hydrogen bonded NH and phenolic OH. FTIR spectra of treated samples clearly show the presence of the C-H stretching vibration in the region of 1418.25 cm^{-1}. The peaks at 1160.68 cm^{-1}, 1084.48 cm^{-1} and 1027.96 cm^{-1} are due to the C-O stretch and the absorption band at 1331.66 cm^{-1} is attributed to the presence of the phenol borate B–O band between 1310 cm^{-1} and 1350 cm^{-1} [58, 59].

Untreated medium density coir board samples showed a strong absorption band at 3388.82 cm^{-1}. This band is broad and typical of hydrogen bonded NH and phenolic OH. The FTIR spectrum of the untreated sample clearly shows the absorption bands in the region of 2898.71 cm^{-1} and 1735.81 cm^{-1} due to C-H and C=O stretching vibration respectively. These absorption bands are due to the carbonyl group of acetyl ester in hemicellulose and carbonyl aldehyde in lignin. Comparison of FTIR spectra suggests that the absorptions situated at 1510 and 1600 cm^{-1} (aromatic skeletal vibrations) are caused by lignin, and the absorption located at 1730 cm^{-1} is caused by holocelluloses; this indicates the C=O stretch in non-conjugated ketones, carbonyls and in ester groups in line with previously obtained results [58, 59].

4.3 Conclusion

The study showed that the thermal properties of medium density coir board samples have greatly improved by the chemical treatment with specially prepared preservative solution containing appropriate proportions of boric acid and borax. Chemically treated medium density coir board samples are thermally more stable over untreated samples throughout the range of temperatures. The TGA and DSC analysis reveals that the first decomposition temperature and crystallization enthalpy increases after treatment. The derivative weight loss decreases after treating the sample with boron. The study suggests that the method of chemical treatment using boron compounds on the medium density coir board samples could increase the thermal stability. This gives an encouraging result to utilise medium density coir boards for more varied applications.

CHAPTER 5

MECHANICAL PROPERTIES OF FR TREATED COIR FIBRE BOARDS

The research on developing a new fire retardant coir fibre board without losing their properties after fire retardant (FR) treatment assumes great significance. Though promising results have been reported for fire retardant treated (FRT) particle boards, little is reported about the corresponding changes in mechanical and physical properties of coir fibre boards after FR treatment. This chapter reports the investigations on the effect of FR chemical treatment of medium density coir fibre board samples and the subsequent changes it imparts to some of the physical and mechanical properties of medium density coir board samples.

5.1 Physical properties

The effect of FR treatment on various physical properties of coir fibre board samples were investigated using standard procedures. Change in properties like density and moisture content were determined. Thermal insulation properties like thermal conductivity and thermal resistance were also determined. Three samples each were tested for both FR treated and untreated coir fibre boards for the entire range of experiments.

5.1.1 Density and moisture content

Density and moisture content of both FR boron treated and untreated coir fibre board samples were determined as per IS: 1734 (Part 1) – 1983 standards. Samples having a size of 75 x 150 x 12 mm were initially weighed before drying it. The coir fibre board samples were oven dried at around 100^0C for short intervals of 5 minutes time and subsequently weighed until further mass loss is not observed.

Thereafter equations (5.1) and (5.2) were used to determine density and moisture content (MC) respectively[101].

$$Density,\ \rho = \frac{M_o}{L \times w \times t} \quad in \quad g/cm^3 \tag{5.1}$$

$$Moisture\,Content,\ MC = \frac{(M_i - M_o)}{M_o} \quad \times\ 100 \quad in\,\% \tag{5.2}$$

Where,

M_i = initial mass of sample in g,

M_o = oven-dry mass of sample in g,

L = length of the sample in cm,

w = width of the sample in cm, and

t = thickness of the sample in cm

5.1.2 Thermal conductivity

Thermal conductivity (k) is the time rate of steady state heat flow through a unit area of a homogeneous material induced by a unit temperature gradient in a direction perpendicular to that unit area. The unit of thermal conductivity is expressed as W/m-K. The ability of a material to conduct heat is measured by its thermal conductivity, k. The higher the k value, the greater the ability of the material to conduct heat and the lower the k, the higher the insulation value.

Figure 5.1 shows the principle of operation of a heat flow meter used to measure the thermal conductivity of coir fibre board samples. The sample was placed between two heated plates controlled to a user-defined sample temperature and temperature drop. The plate temperatures were controlled by bidirectional Heating/Cooling Peltier systems, coupled with a closed loop fluid flow with an integrated forced air heat exchanger.

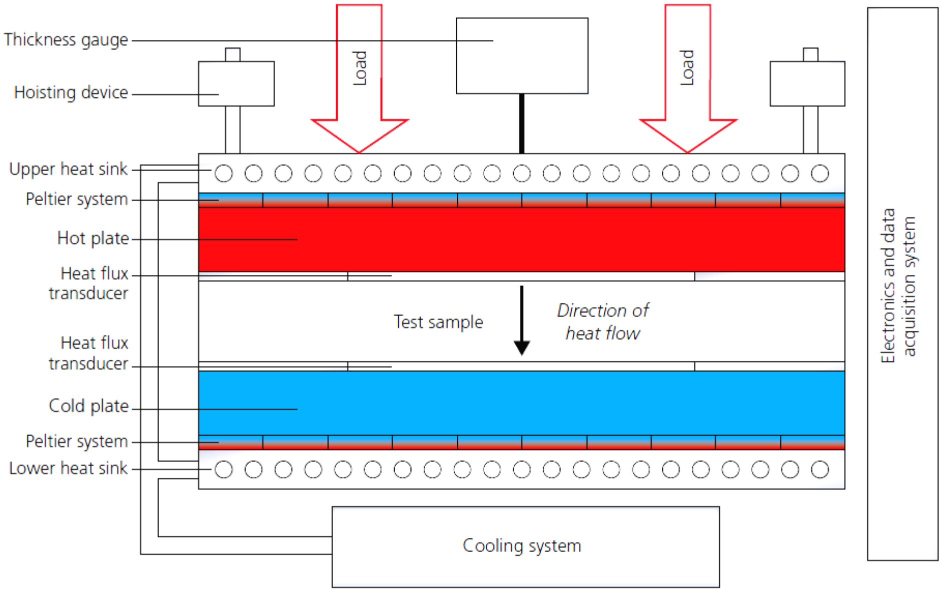


Figure 5.1: Schematic design of thermal conductivity apparatus NETZSCH HFM 436 Lambda (plate temperatures between 0^0C-100^0C)

A heat flow meter (model: HFM 436 Lambda, Make: NETZSCH, Germany) was used to measure the steady state thermal transmission through flat slab samples having a size of 305 x 305 x 11 mm. The thermal conductivity and thermal resistance of both treated and untreated samples were determined as per ASTM C518 standard and were used to compare the thermal insulation performance of FR treated coir fibre boards against untreated fibre boards [102].

The values of thermal conductivity were determined from the equation (5.3), where it is proportional to heat flow rate. It also shows the different factors affecting the thermal conductivity of samples.

$$Thermal\ Conductivity,\ k\ = q \times \frac{L}{\Delta T} \qquad in \quad W/m - K \tag{5.3}$$

Where,

L = Thickness of the specimen in m,

T = Temperature in K,

q = Heat flow rate in W/m^2.

Thermal resistance (R) is the temperature difference, at steady state, between two defined surfaces of a material or construction that induces a unit heat flow rate through a unit area, m^2.K/W. According to this definition, Equation (5.4) can be obtained. The value of thermal resistance is determined using Equation (5.4). The higher the R value, the more effective the insulation [102].

$$Thermal\ Resistance,\ R\ = \frac{\Delta T}{q} \quad = \frac{L}{k} \quad in \quad m^2.K/W \tag{5.4}$$

Where,

k = Thermal conductivity in W/m-K,

L = Thickness of the specimen in m,

T = Temperature in K,

q = Heat flow rate in W/m^2.

5.2 Mechanical properties

The FR treated and untreated coir fibre board samples were tested in a universal testing machine (Z 100, Zwick Asia Private Ltd, Singapore) to measure and compare their mechanical properties (Figure 5.2). The effect of FR treatment on coir fibre boards over mechanical properties like strength were investigated as per ASTM standards.

Figure 5.2: Universal Testing Machine for measuring tensile, bending and compressive properties of coir fibre boards

5.2.1 Tensile Test

Tensile test performed on the samples was as per ASTM D3039 standards. Medium density coir fibre board samples measuring 250 x 50 x 12 mm were used for testing. The samples were subjected to a maximum operating force of up to 1.3 kN at a test speed of 10 mm/min [103].

Mechanical properties like Tensile strength (MPa), Tensile Modulus (MPa), Maximum tensile force to failure (N) and Longitudinal strain at maximum tensile force (%) were determined for both FR treated and untreated coir fibre board samples.

5.2.2 Bending Test

The flexural properties of both treated and untreated samples were determined as per ASTM D790 – 02 test standard. The three point bending test was performed on coir fibre boards having a size of 250 x 50 x 12 mm at a crosshead speed of 5mm/min and preload of 0.1 MPa [104]. Properties like flexural strength (MPa) and flexural modulus (MPa) were obtained to compare the performance of both FR treated and untreated coir fibre board samples.

5.2.3 Compression test

Coir fibre board samples having a size of 12 x 12 x 24 mm were tested as per ASTM D 695. Compression test on both treated and untreated samples were performed at a standard test speed of 1 mm/min [105]. Compressive strength (MPa) of coir fibre board samples were determined and compared to measure the performance of FR treated coir fibre board samples against untreated samples.

5.3 Results and Discussion

The various physical and mechanical properties of FR treated coir fibre boards are experimentally determined and are compared against the values found from testing untreated coir fibre board samples. The average values of different physical and mechanical properties obtained from testing of both FR treated and untreated coir fibre board samples with their respective standard deviations are consolidated and are shown in Table 5.1.

Table 5.1: Average values determined for different properties

Sl. No:	**Properties**	**Determined values with standard deviation**	
		FR Treated samples	**Untreated samples**
1	Density (Kg/m^3)	846 (5)	650 (3.51)
2	Moisture content (%)	7 (0.57)	5 (1)
3	Tensile strength (MPa)	1.26 (0.04)	0.8395 (0.015)
4	Compressive strength (MPa)	11.6 (0.73)	15.7 (0.35)
5	Flexural strength (MPa)	33.9 (12.33)	17 (5.47)
6	Flexural modulus (MPa)	3.03 (0.42)	2.16 (0.42)
7	Tensile Modulus (MPa)	4846 (51.05)	2469 (18.68)
8	Max. tensile force to failure (N)	1260 (40)	839 (14.53)
9	Longitudinal strain at maximum tensile force (%)	0.026 (0.006)	0.034 (0.005)
10	Thermal conductivity (W/m-K)	0.0938 (0.005)	0.183 (0.002)
11	Thermal resistance (m^2 K/W)	0.117 (0.003)	0.060 (0.002)

5.3.1 Effect of FR treatment on Physical properties

The prescribed value of density of medium density coir fibre boards (Grade-1) as per IS 15491 falls within a range of 650 - 900 Kg/m^3 [67]. The untreated samples have a density of 650 Kg/m^3 when tested while samples after FR treatment exhibited an increase in density and recorded 846 Kg/m^3. This increase in density is due to the added weight of borates present in FR treated coir fibre board samples. The percentage of moisture content present in coir fibre board samples have to be in a controlled level of within 5-15% as per IS 15491. The untreated samples have a moisture content of 5%, whereas FR treated samples contained 7% moisture content, but is still within the range specified by the relevant BIS standard-IS 15491. The moisture content of FR treated samples increases as a result of the chemical treatment done using a water-borne solution for about seven hours.

FRT coir fibre boards are found to have a reduced value of 0.0937 W/m-K for thermal conductivity, whereas the untreated samples exhibited 0.183 W/m-K. The thermal conductivity values for coir fibre boards are comparable to thermal conductivity of hardwood species like Yellow-poplar (0.11) as well as softwoods like Douglas-fir (0.12) and Redwood (0.10) in W/m-K [106]. The thermal resistance of FRT samples show a considerable increase to a value of 0.117 m^2 K/W from a resistance of 0.060 m^2 K/W for untreated samples. The more resistant and less conductive FRT coir fibre board samples turns out to have better thermal insulation characteristics.

5.3.2 Effect of FR treatment on Mechanical properties

The values for tensile strength perpendicular to the surface (Internal bond) and modulus of elasticity of treated coir fibre boards are found. As per IS 15491 the average values stipulated for tensile strength of coir fibre boards are within 0.8 - 0.9 MPa and the tensile modulus is expected to be between 2500 - 2800 MPa [67]. The average tensile strength of FRT coir fibre boards are found to have increased to 1.26 MPa while untreated boards are only exhibiting 0.8395 MPa. The tensile modulus (E), of untreated fibre boards are found to be 2469 MPa while the FRT fibre boards show a substantial increase of 4846 MPa.

The flexural strength (modulus of rupture) and flexural modulus of coir fibre boards are found using a three point bending test. The average flexural strength of FRT samples increases to 33.9 MPa from 17 MPa of untreated samples, while the flexural modulus of treated samples increases to 3.03 MPa from 2.16 MPa of untreated samples. As per IS 15491 the average values stipulated for modulus of rupture (MOR) of coir fibre boards are within 17–31 MPa. The results of bending test on FRT coir fibre boards confirm a substantial improvement in the properties flexural strength and flexural modulus.

The average compression strength for a coir fibre board is comparable to that of similar wood fibre composite boards for general purposes. When compared to the average compressive strength of sheathing-grade oriented strand boards having values between 10.3 – 17.2 MPa, coir fibre boards retained the required compressive strength after fire retardant treatment [107]. However the average compressive strength of treated samples show a negligible fall to 11.6 MPa from 15.7 MPa of untreated samples.

5.4 Conclusion

Coir fibre boards were subjected to FR treatment with a specially prepared chemical solution containing a combination of BX and BA to enhance their fire retardant properties. The samples after FR treatment conformed to the required values for density and moisture content as put up by IS 15491 standard. The density of samples increased from 650 Kg/m^3 to 846 Kg/m^3 after FR treatment whereas the moisture content increased from 5% to 7%.

The thermal conductivity of FRT coir fibre boards were reduced to 0.0937 W/m-K from 0.183 W/m-K whereas, the thermal resistance of fibre boards increased from 0.060 m^2 K/W to 0.117 m^2 K/W. The treated samples exhibited reduced values for thermal conductivity and improved thermal resistance when compared to untreated samples, which means there is an overall improvement in thermal insulation characteristics of FRT coir fibre boards.

The study also determined the mechanical behaviour of FR treated coir fibre boards using a combination of tensile test, bending test and compression test. The obtained values were used to evaluate the performance of samples after FR treatment. The tensile strength of coir fibre boards increased from 0.8395 MPa to 1.26 MPa and flexural strength increased from 17 MPa to 33.9 MPa after FR treatment. However, the compressive strength was reduced from 15.7 MPa to 11.6 MPa after FR treatment.

Experimental investigations show that the fire retardant treated coir fibre boards exhibited better physical properties than that of untreated coir. The penetration of boron particles in to the porous structure of coir fibre board has increased the density of treated boards and subsequently influenced other physical and mechanical properties. The soaking time required for fire retardation treatment of coir fibre board samples in hot and cold bath method were also considerably reduced to check the subsequent adverse effects of hydration. These two factors contributed to the better physical and mechanical properties exhibited by the fire retardant treated coir fibre boards over untreated coir fibre boards.

The results also suggested that there was no substantial loss of strength to coir fibre boards after FR treatment. The FRT coir fibre boards exceeded the minimum required values for strength set by IS 15491 standards. The results were encouraging and suggest the use of FR treatment on coir fibre boards for applications involving stringent fire safety norms. The study also proposes the possibility of developing a new class of fire proof coir fibre boards that can cater to a wide range of applications.

CHAPTER 6

FLAME RESISTANT PROPERTIES OF FR TREATED COIR FIBRE BOARDS

Carbon-based lignocellulosic materials, such as bio-based composites, undergo combustion, pyrolysis, or burning when subjected to radiant energy or sufficient direct heat leading to concerns about medium-density coir boards for its potential ignitability and flame-resistant characteristics [26, 57, 96]. Flame retardant treatment can be investigated to overcome these drawbacks and enable medium-density coir boards to satisfy better fire safety requirements and make it suitable for a wider range of applications [42]. Boron compounds are common preservatives used for protecting wood against fungi and insects, especially termites.

Borates act as both wood preservatives and flame retardants and have low toxicity, low volatility, and are colorless and odourless [57, 58]. The primary mechanism for imparting fire resistance is to form a coating or protective layer on the surface at high temperature. A combination of borax (BX) and boric acid (BA) exhibits a synergic effect on flame-resistant properties of the sample [77, 95]. BX tends to reduce flame spread, but promotes glowing, whereas BA suppresses glowing and improves mechanical properties such as internal bond strength and limit swelling of panel boards [80].

This chapter enumerates a study on the effect of chemical treatment on medium-density coir board samples through a set of standard fire tests. An aqueous solution of BA and BX have been used for improving the flame-retardant properties of samples of medium-density coir board.

6.1 Limiting Oxygen Index (LOI) Test

Limiting Oxygen Index (LOI) characterizes the ability of a material to burn even at reduced oxygen concentration, especially during fire in buildings where the air supply to the area is very limited [92, 108, 109]. Figure 6.1 shows the limiting oxygen index tester used to find oxygen index values of coir fibre boards.

Figure 6.1: Limiting Oxygen Index Tester

The LOI of both FR treated and untreated coir fibre board samples measuring about 60 x 40 x 12 mm was determined as per ASTM D 2863 in an oxygen index tester (model: CSI 178, make: Custom Scientific

Instruments Inc., India). Equation (6.1) were used to determine the limiting oxygen index values.

$$Limiting\ Oxygen\ Index,\ n = \frac{100\ \times\ O_2}{(O_2\ +\ N_2)}\quad in\quad \% \tag{6.1}$$

Where,

O = Oxygen concentration in the inflow gases in %,
N = Nitrogen concentration in the inflow gases in %,

Since air comprises about 20.95% oxygen by volume, any material with an LOI less than this will burn easily in air. Therefore, values below 21 are generally considered as flammable or combustible and a high index is indicative of a material that has lower ignitability and flammability [110]. As per ASTM D 2863 the sample has to burn continuously for a period of 180 seconds after ignition to complete the test and obtain LOI value [111]. Table 6.1 shows the general nature of flammability of substances with respect to their Limiting Oxygen Index values.

Table 6.1: LOI and Nature of Flammability

Oxygen index (LOI)	**Nature of Flammability**
Up to 16	Highly inflammable
16 – 20	Flammable
21 – 24	Slow burning
25 – 29	Limited fire resistance
30 – 43	Self extinguishing

6.2 Determination of Fire Resistance

Both treated and untreated samples were prepared and tested according to IS 5509:2000 for assessing their fire proofing characteristics. To evaluate the extent of fire resistance of samples after the chemical treatment, a combination of three fire resistance tests were carried out as per IS:1734 (Part 3), 1) flammability test, 2) flame penetration test, and 3) rate of burning test [89, 112].

A Junkers gas calorimeter was used to find the calorific value of the fuel used i.e., liquefied petroleum gas (LPG) and a gas flow meter (wet type) was used to record the volume flow rate [113, 114]. Figure 6.2 shows the Junkers gas calorimeter apparatus which was used to determine the rate of heat energy added to the system during experiments. The apparatus was also equipped with a gas flow meter (wet type) to measure the volume flow rate of LPG.

Figure 6.2: Junkers gas calorimeter apparatus

Equation (6.2) and (6.3) were used to determine the calorific value of LPG and rate of heat energy added during the experiments.

$$Calorific\,Value,\;CV_{gas}\;=\;\frac{(m\;\times\;C_p\times\Delta T)}{V_g}\quad in\quad kJ/m^3 \tag{6.2}$$

$$Rate\;of\;Heat\;Energy\,added,\;Q\;=\;m\times C_p\times\Delta T\quad in\quad kJ/s \tag{6.3}$$

Where,

V_g = The volume of gas burnt in l/s,
m = Mass flow rate in kg/s,
ΔT = The change in temperature of water in ^{0}K,
C_p = The specific heat of water in kJ/Kg.K,

The tests were performed at a constant volume flow rate of 28.8 L/h for all the three tests and the calorific value of fuel in the gaseous state was found to be 94 MJ/m^3. The rate of heat energy added during the tests was found to be 752 J/s.

6.2.1 Flammability test

The flammability test was conducted on six set of treated and untreated samples as per IS 1734. LPG was fed to the burner and burned as a blue flame and ignited the face of the lower specimen, which in turn ignited the opposite face of the higher specimen. The variable, flammability, was the time taken (min) for the higher specimen to be ignited after igniting the lower specimen. The specimen was deemed ignited if the burning is uninterrupted for at least 50 seconds [56]. The arrangement for the test is shown in figure 6.3 and figure 6.4 shows the flammability test apparatus in operation fixed with a Bunsen burner so that the flame was directed against the inner face of the specimens.

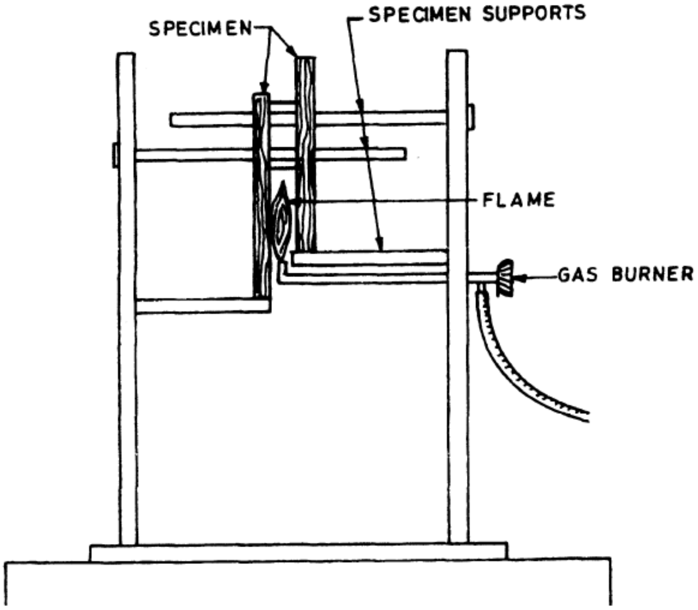
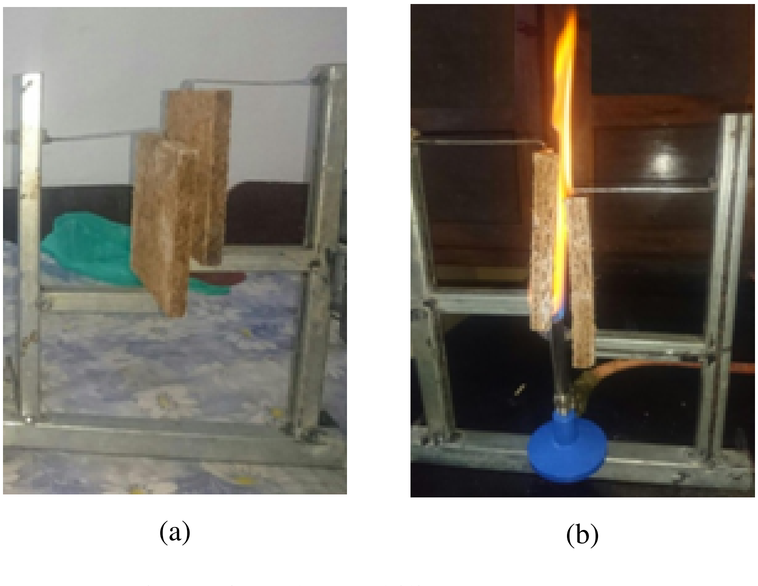


Figure 6.3: Schematic arrangement for flammability test


(a)

(b)

Figure 6.4: Flammability test apparatus

Two test specimens measuring about 125 x 125 x 12 mm were held vertical, 15 mm apart, with one held 40 mm higher than the other. Flammability test apparatus was equipped with a Bunsen burner fitted horizontally so that the flame plays against the lower end of the inner face of the lower specimen [56].

6.2.2 Flame penetration test

Flame penetration test as per IS 1734 was conducted on three set of treated and untreated samples measuring about 125 x 125 x 12 mm. The schematic arrangement for flame penetration test is shown in Figure 6.5.

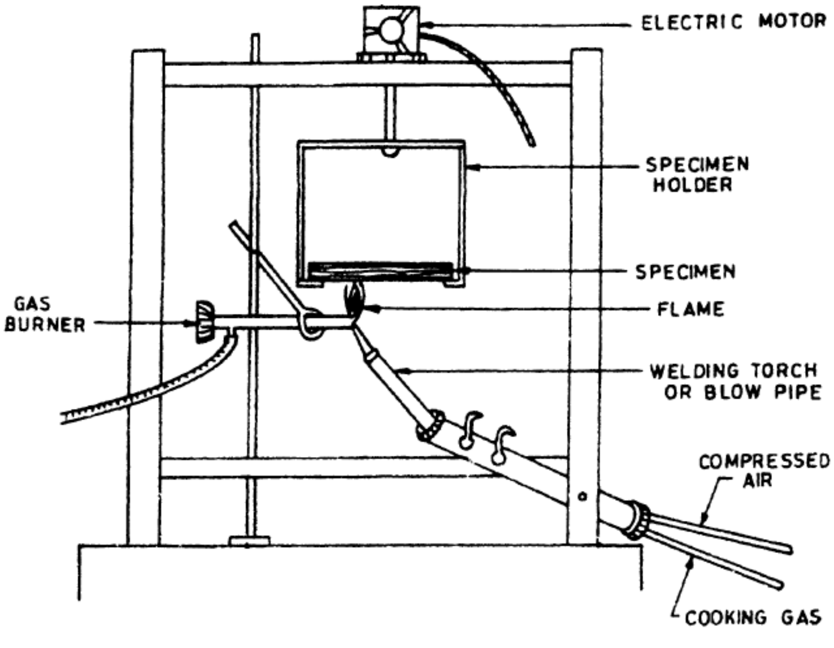
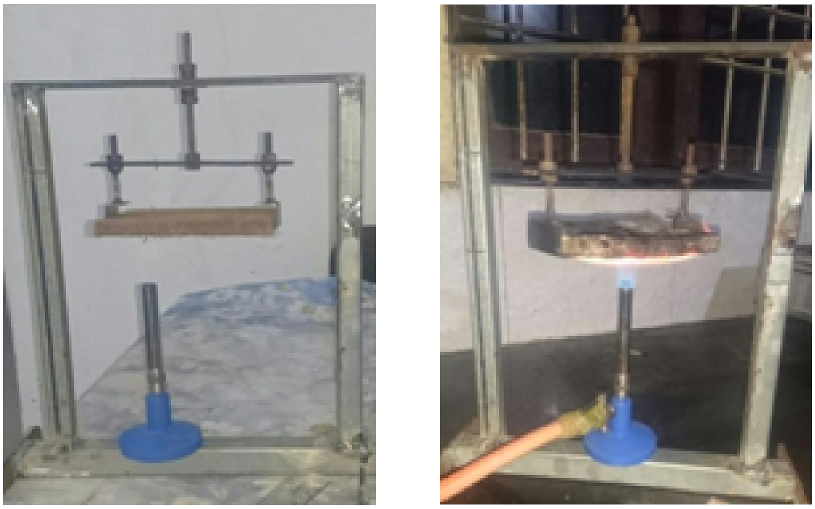


Figure 6.5: Schematic arrangement for flame penetration test

The test specimen was held 50 mm away from the nozzle of a Bunsen burner as shown in figure 6.6 and the time taken for the flame to penetrate the thickness of each sample was recorded as the flame penetration time (min).



(a) (b)

Figure 6.6: Flame penetration test apparatus

6.2.3 Rate of burning test

Rate of burning test as per IS 1734 was carried out on three chemically treated and three untreated samples measuring 100 x 12.5 x 12 mm. Figure 6.7 shows the arrangement for rate of burning test. Figure 6.8 shows a test specimen ignited by a blue flame during the rate of burning test.

The test specimen was suspended vertically and adjusted to a height of 30 mm from the nozzle of the burner. The weight of the sample is monitored throughout the test using a balance and the time taken for each 10 % loss in mass was recorded. The time taken for the loss of mass of the sample from 30% to 70% was recorded for the purpose of comparison.

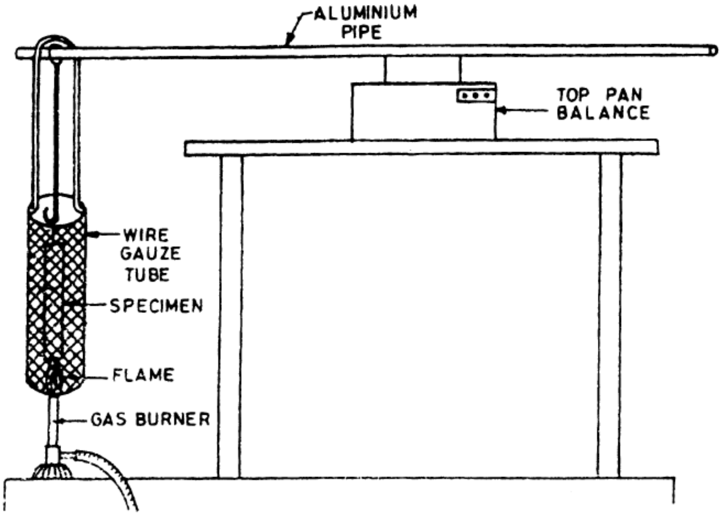


Figure 6.7: Schematic arrangement for rate of burning test

Figure 6.8: Rate of burning test apparatus

6.3 Resistance to Spread of Flame Test

The test for resistance to spread of flame was carried out as per IS 15491. A total of 6 test specimens measuring about 150mm x 25mm x 12mm were selected and 3 of them were chemically treated with the prepared solution containing borates. Figure 6.9 shows the set up for resistance to Spread of Flame.

Figure 6.9: Resistance to flame spread test apparatus

The specimens were held at an angle of 45^0 to the horizontal and subjected to a blue flame, supplied by the nozzle of a Bunsen burner. The specimens were exposed to fire intermittently for 30 seconds and the cycle was repeated until ignition. An ignited sample subsequently burning for more than 20 seconds suggests flame spread [67]. The time for which coir fibre board samples were able to withstand ignition and flame spread were recorded to compare the resistance to flame spread. The ignited specimens which continued to burn for not more than 20 seconds after the flame was removed were taken as specimens which passed the test [67].

6.4 Results and Discussion

The consolidated results of the fire resistance measurement values are shown in Table 6.2. Even though the untreated samples exhibit a value of 34 minutes when tested for flammability and conformed to the acceptance parameter of 30 minutes, their performance is enhanced after chemical treatment and found to have a flammability time of 54 minutes.

Table 6.2: Consolidated results for determination of Fire Resistance

Sl. No.	**Test as per IS 1734 (part 3) 1983**	**Results (min)**		**Prescribed value as per IS 5509:2000**
		Untreated samples	**Treated samples**	
1	**Flammability** (time taken for second specimen to ignite after the first lower specimen ignites)	34	54	Not less than 30 min
2	**Flame penetration** (time taken for flame penetration from bottom to top surface)	27	38	Not less than 15t/6 = 30 min (where, thickness of sample, t = 12 mm)
3	**Rate of burning** (time taken to lose weight from 70% to30%)	12	44	Not less than 20 min

The untreated samples when tested for flame penetration exhibits a value of 27 minutes and failed to meet the minimum requirement of 30 minutes, while treated samples show a value of 38 minutes which is more than the minimum requirement to qualify as a fire resistant material in this test. Treated samples when tested for rate of burning, show significant improvements with a value of 44 minutes when compared to untreated samples which gave a low value of 12 minutes against a minimum requirement of 20 minutes to qualify as a fire resistant material.

The flammability, flame penetration and rate of burning values for treated samples considerably improve over untreated samples and exhibited relatively higher values than the prescribed minimum requirements for all the three tests.

The results of resistance to flame spread test has been tabulated in table 6.3. It is observed that the boron treated samples gain significant resistance to ignition and flame spread when compared to the untreated samples. FR boron treated medium-density coir fibre boards offer greater resistance to fire and flame propagation when compared to natural or engineered wood products which contains combustible organic carbon and hydrogen compounds [79, 80].

Table 6.3: Results of resistance to flame spread test

Sl. No.	**Test as per IS 15491: 2004**	**Results (min)**		**Prescribed value as per IS 15491:2004**
		Untreated samples	**Treated samples**	
1	**Resistance to Spread of Flame** (time taken for the specimen to get ignited)	6	62	If the specimen gets ignited, it should not continue to burn for more than 20 s after flame removal

Any specimen showing LOI values from 24 to 28 are classified as limited fire-resistant materials [115]. LOI values of FR treated samples are found to be 100 which indicates its high self-extinguishing nature, whereas the untreated samples show an LOI of 28 which can be classified under the group of materials with limited fire resistance. The LOI value of FR treated samples is approximated as the maximum possible value of 100 as the flame could not sustain for 180 s as stipulated by ASTM D 2863. Even though the treated samples get ignited, there is no flame spread after ignition.

Table 6.4 gives the values of Limiting oxygen index test conducted for FR treated and untreated medium density coir fibre board samples.

Table 6.4: LOI Test results

Sl. No.	**Sample**	**LOI**
1	Untreated Samples	28
2	FR Treated Samples	100

6.5 Conclusion

Even though a large variety of fire-retardant chemicals and their treatment methods have been studied in the past, the combination of BX and BA has been found to be effective in many cases. Coir fibre–based medium-density panel boards were subjected to treatment with specially prepared solution of BX and BA for enhancing their fire-retardant properties. The results encourage using medium-density coir boards for more varied applications involving stringent fire safety norms. The study also suggests the possibility of developing a new class of fire-proof coir fibre–based panel boards that can cater to a wide range of applications.

CHAPTER 7

FRUGAL COMBINATIONS OF BORON FOR FLAME-RESISTANT COIR FIBRE BOARDS

This chapter details the investigations on determining an optimum concentration of boron-based fire-retardant (FR) solution for the chemical treatment of coir composite panel boards to have a positive effect on fire resistance. The relation between the quantity of boron present in the FR solution for treatment and the performance of composite board materials when exposed to fire is also investigated.

7.1 Chemical treatment

Different weight percentage levels of boron, starting from 10%, were added to water to form FR solutions until maximum solubility was achieved. Addition of boron compounds to form the FR solution have been achieved through a mixture of Boric Acid (BA) and Borax (BX) in water and the combination for maximum solubility without precipitation was observed when a mixture of 15% BA and 20% BX, is added to 65% water, yielding approximately 35% boron in the FR solution. Addition of boron beyond this combination was found to be not fruitful as it seldom dissolved completely into solution and the remaining existed as precipitate in the solution.

The samples were cleaned and air dried before treating it with aqueous solutions containing different weight percentage of BX and BA combinations in a controlled environment. A modified hot and cold bath method was applied for chemical treatment without affecting the laminate. During the initial phase of treatment, air molecules present in the sample expanded as a result of heating and are driven out, whereas the residual air in the sample contracted at a later stage during the cooling of the sample, thus creating a partial vacuum which allowed the preservative boron to penetrate [86].

Table 7.1 gives the weight percentage of boron compounds used for preparation of fire retardant solutions and its subsequent treatment with coir fibre boards. The investigation was made to assess the possibilities to find the optimum combination of chemicals to be used in fire resistance treatment and seven different solutions having varying weight percentage of BA and BX were prepared for treatment.

Table 7.1: Composition of FR solution

Sample	**Water (%)**	**BX (%)**	**BA (%)**
FRS 1 (40% boron)	60	23	17
FRS 2 (35% boron)	65	20	15
FRS 3 (30% boron)	70	18	12
FRS 4 (25% boron)	75	15	10
FRS 5 (20% boron)	80	12	8
FRS 6 (15% boron)	85	9	6
FRS 7 (10% boron)	90	6	4

7.2 Determination of Fire Resistance

The amount of time required for any material to be classified as fire resistant, varies for different tests. In order to endorse a material to be as fire resistant, it has to pass each of the several tests. A sample is expected to withstand the flame subjected on it for a stipulated minimum time to be classified as fire resistant.

Chemically treated samples were subjected to four different fire retardation tests for determination of flame resistance and fire proofing abilities and to assess their flame spread characteristics. To investigate the degree of fire resistance imparted to the samples after FR treatment, a combination of fire resistance tests were carried out as per IS:1734 (BIS 1983), (1) flammability test, (2) Flame Penetration test, and (3) Rate of Burning test along with (4) Resistance to Spread of Flame Test as per IS:15491.

Table 7.2 shows the prescribed minimum values of time in minutes, the sample has to withstand an active flame supplied by a Bunsen burner as per BIS standards. It gives a fair idea about how good the values exhibited in tables 7.3 and 7.5 are in resisting fire. Longer the time a sample withstands against fire, better the flame resistance of coir fibre board samples. It also helps in explaining if the presence or differences in weight percentage of boron can contribute to flame resistance of the material.

7.2.1 Resistance to Flammability

The flammability test was conducted as per IS 1734 standards. A total of 48 specimens were subjected to flammability test in three trials performed

Table 7.2: Prescribed minimum time to pass the test

Sl. No.	**Test as per IS 1734 (Part 3) 1983**	**Prescribed minimum time (minutes) as per IS 5509:2000**
1	**Flammability** (Time taken for 2nd specimen to ignite after the first lower specimen ignites)	Not less than 30 minutes.
2	**Flame penetration** (Time taken for Flame penetration from bottom to top surface)	Not less than 15t/6 = 30 minutes (where, thickness of sample, t = 12 mm)
3	**Rate of burning** (Time taken to lose weight from 70% to 30%)	Not less than 20 minutes.
Test as per IS 15491: 2004		**Prescribed minimum time (minutes) as per IS 15491: 2004**
4	**Resistance to Spread of Flame** (Time taken for the specimen to get ignited and sustained burning)	No minimum time prescribed for ignition, but after ignition, no burning for a period of more than 20 seconds.

for every set of samples. Two test specimens measuring about 125mm x 125mm x 12 mm were prepared for each set of tests. Flammability (in minutes) of fibre board samples were recorded as the response variable and it indicates the speed at which the specimen catches fire when placed in the immediate vicinity of a flame.

7.2.2 Resistance to Flame penetration

The flame penetration test was conducted as per IS 1734 standards. A total of three trials were performed for each of the eight set of samples that added to a total of 24 specimens which underwent flame penetration tests. They were conducted on specimens that measured about 125mm x 125mm x 12 mm. Flame penetration time (in minutes) for fibre board samples were recorded as the response variable and it indicates the speed at which a continuous flame penetrates the thickness of sample.

7.2.3 Reduction in Rate of Burning

The rate of burning test was carried out as per IS 1734 standards. Three trials each of the test was performed on eight set of samples, a total of 24 specimens that measured 100 mm x 12.5 mm x 12 mm. Rate of burning (in minutes) of fibre board samples were recorded as the response variable and it indicates the speed at which the sample turns to char when continuously subjected to an active flame.

7.3 Resistance to Flame Spread

The resistance to flame spread test was carried out as per IS 15491 standards. Three trials of the test were performed for eight set of samples, a total of 24 samples that measured about 150mm x 25mm x 12 mm. Resistance to flame spread (in minutes) of fibre board samples were recorded as the response variable and it indicated the speed at which flame propagates through the sample surface.

7.4 Results and Discussion

Coir fibre board samples are fire retardant treated with seven different aqueous solutions containing varying weight percentage of FR boron in it, starting from 10 to 40% boron present in the solution. These FRT coir fibre board samples are tested for flammability (min), flame penetration time (min), rate of burning (min) and resistance to flame spread (min) throughout the range of fire resistance treatment and the results are tabulated in Table 7.3.

Table 7.3: Data set of test results for fire resistance

Samples	**FR Boron (Wt. %)**	**Flammability (min)**	**Flame penetration time (min)**	**Rate of burning (min)**	**Resistance to flame spread (min)**
		as per IS 1734 (Part 3) 1983			as per IS 15491
FRS 1	**40**	49.00	38.50	41.50	56.00
		51.00	42.00	40.00	57.75
		48.00	40.00	42.00	59.00
FRS 2	**35**	55.00	38.00	42.25	65.00
		56.00	35.50	44.00	62.00
		50.00	39.25	46.50	59.00
FRS 3	**30**	52.00	40.00	47.50	65.00
		54.00	38.00	49.00	67.00
		50.00	46.00	60.00	64.00
FRS 4	**25**	44.00	35.00	44.50	62.00
		43.50	35.50	51.00	64.00
		40.00	39.00	46.50	57.00
FRS 5	**20**	38.00	33.00	28.00	57.50
		38.50	32.50	31.00	63.50
		35.50	36.00	27.00	55.00
FRS 6	**15**	30.25	30.00	26.00	57.00
		26.50	30.00	26.15	55.00
		29.00	29.50	27.25	60.00
FRS 7	**10**	26.75	27.50	21.00	52.25
		25.00	29.00	19.00	52.50
		26.00	25.75	20.00	53.50
Untreated	**0**	34.00	25.00	12.00	6.00
		32.50	27.00	13.00	6.00
		35.50	29.50	11.00	6.00

It is observed from the results that chemically treated medium density coir fibre board samples gained substantial resistance to ignition and the flame spread when compared to the untreated samples. The results suggest that maximum fire resistance to coir fibreboard samples are achieved when they are treated with an FR solution containing 30% to 35% boron compounds in it. A combination of BA and BX in the ratio of 1:1.5 is used to prepare the FR solution. The FR solution will contain 30% boron compounds if 18% BA and 12% BX are added to 70% water and there will be 35% boron compounds, if 15% BA and 20% BX are added to 65% water which gives maximum solubility without precipitation.

When boron added to water is beyond 35% of the total weight percentage, instead of forming a clear solution, boron precipitates in the solution and adversely affects the treatment. However at elevated temperatures the amount of precipitate is lesser than when the solution is at room temperature.

The experimental investigation proceeded with four distinctive fire performance tests which collectively helped to evaluate the various attributes of fire retardation imparted in FR treated coir fibre boards. Flammability values indicated the easiness by which a sample gets ignited and chances of burning rapidly. The presence of boron particles in FR treated samples contributed to a decrease in flammability proportional to their weight percentage. Maximum solubility of boron compounds in water yielded around 35% boron compounds in a FR solution and the values for flammability is found to be most favourable when the weight percentage is around 30 -35% in the chemical solution.

FR treated coir fibre boards also exhibited better resistance to flame penetration and took more time to penetrate the sample. Similarly a reduction in rapid burning due to the presence of boron particles had led to a reduced rate of burning for FR treated samples. It was observed that the presence of boron hugely influenced the resistance to flame spread of coir fibre boards and even the weakest solution concentration would impart considerably improved values of flame spread resistance.

The relationship between FR solutions having different weight percentages of boron compounds present in it to the effect it has on the fire resistance properties of coir composite boards was plotted and is shown in figure 7.1.

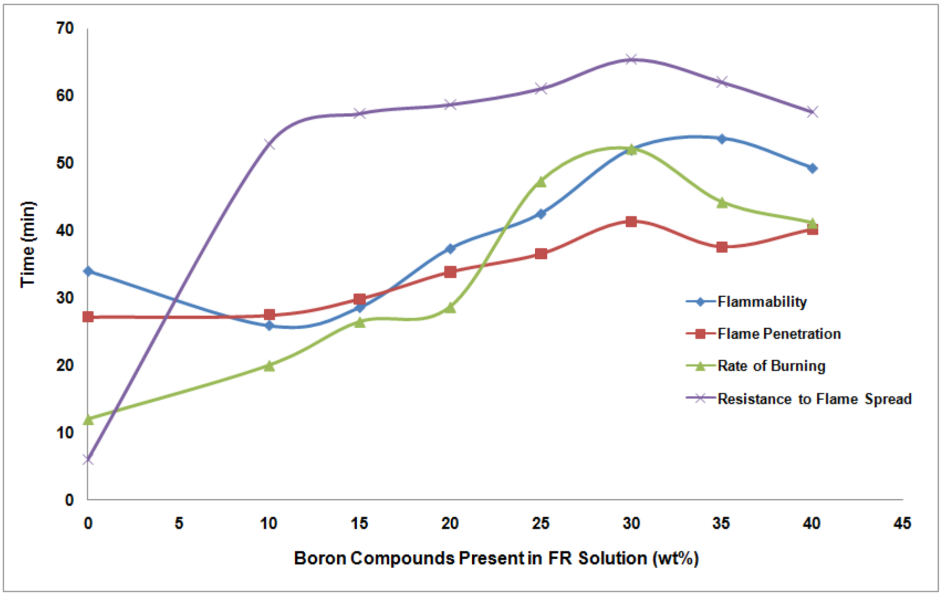


Figure 7.1: Fire Resistance of FRT coir composite boards

A close examination of the graph suggests that the FR solutions having a concentration of boron from 25 to 35% of the total weight of the solution imparted highest flame resistance to coir fibre boards. The peak values for flame penetration, rate of burning and flame spread in the graph were exhibited by coir fibre board samples treated with a solution designated as FRS3 which has a weight percentage of 30% boron in the solution. Whereas the samples treated with a solution having 35% boron designated as FRS2 indicated the maximum value for flammability. The plot also depicts a subsequent decline after adding more boron to it.

Self extinguishing nature of the FR treated samples were observed when they were subjected to fire in all these four tests since boron treated samples where often charred than burning. The solution concentration is the strongest when maximum solubility of a combination of borax and boric acid were reached and is experimentally found to be when containing 35% boron and beyond that it seldom dissolves. Even though this solution is stronger, the agglomeration of boron particles makes it less permeable whereas the solution containing 30% boron finds it easy to penetrate the fibre board and gives a better result for flame penetration, rate of burning and flame spread.

Regression analysis is used as a tool to investigate these data and to establish a relationship between weight percentage of boron compounds and different fire resistance tests.

7.5 Regression analysis

A regression analysis was performed to investigate the extent to which the weight percentage of boron present in the FR solutions predicted the fire resistance of the medium density coir fibre boards using Minitab 17 (Minitab, Inc., State College, PA, USA). Linear, quadratic and cubic regression models were considered for analysis. It was found that the cubic model gave the best fit and most accurate predictions. Therefore, a third degree polynomial (cubic) regression model was used with the weight percentage of boron compounds in FR solution as the predictor (X) and the results of the various fire resistance tests (in min) as the responses (Y).

The relationships between the predictor (X), i.e., weight percentage of boron compounds present in the FR solutions and the different responses (Y), i.e., the values for flammability, flame penetration, rate of burning, and resistance to flame spread, were investigated. The significance threshold was set at a level of significance, $\alpha < .05$, for all four of the fire performance tests. Since fire resistance is a collective measure of different fire resistance tests, each test was analysed separately for regression.

7.5.1 Regression analysis for Flammability

A regression analysis was performed to predict the flammability of coir fibreboards after treatment with a solution of varying concentrations of FR boron compounds.

A regression equation was generated with (F (3,20) = 187.16, $p < .001$), with an R^2 value of 96.6%. The regression equation used to predict flammability of coir fibre boards are described in the equation (7.1).

$$\boxed{Y_1 = 34.19 - 2.543\,(X) + 0.1928\,(X)^2 - 0.002989\,(X)^3} \tag{7.1}$$

Figure 7.2 shows a fitted curve plot with flammability (Y_1) in minutes as the ordinate and weight percentage of boron compounds in FR solution (X) as the abscissa.

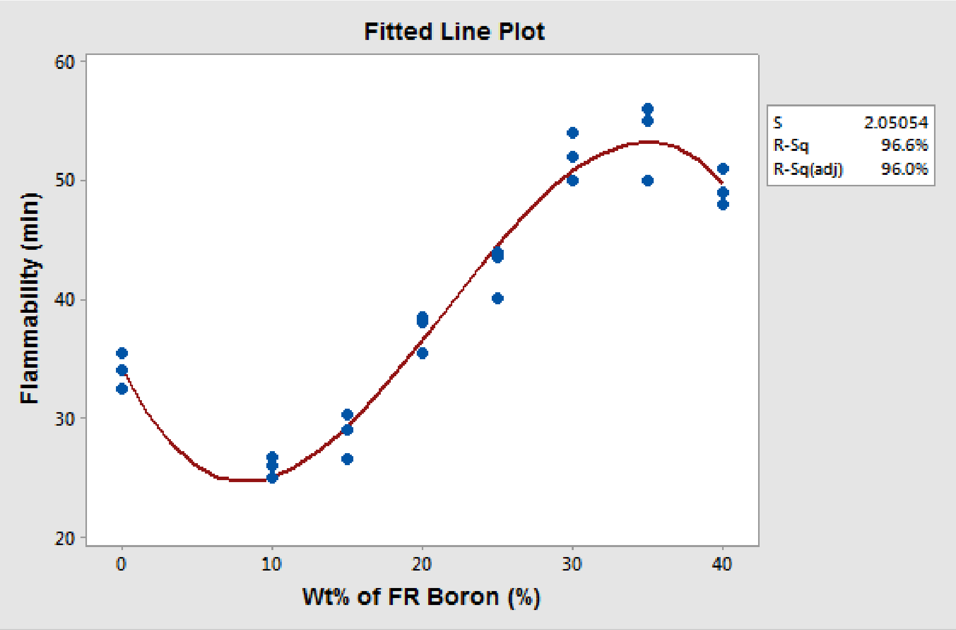


Figure 7.2: Fitted line: Flammability versus Wt. % of FR boron

7.5.2 Regression analysis for Flame penetration

A regression equation was generated with (F (3,20) = 34.31, $p < .001$), with an R^2 value = 83.7%. The regression equation used to predict the time for flame penetration in coir fibre board samples are described in the equation (7.2).

$$\boxed{Y_2 = 27.05 - 0.4014\,(X) + 0.05477\,(X)^2 - 0.000929\,(X)^3} \tag{7.2}$$

Figure 7.3 shows a fitted line plot with flame penetration time (Y_2) in minutes as the ordinate and weight percentage of boron compounds in FR solution (X) as the abscissa.

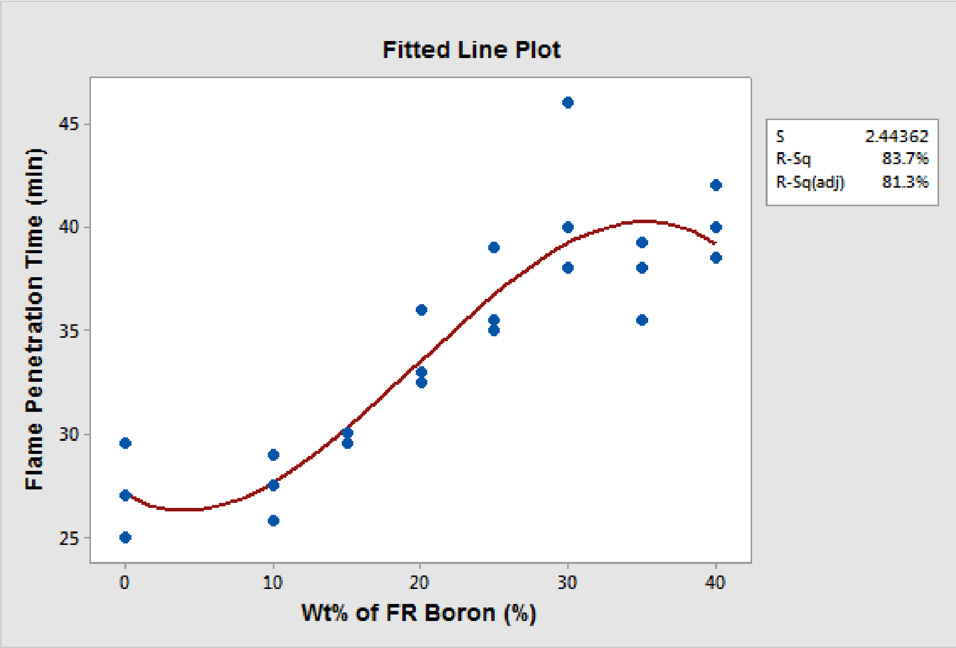


Figure 7.3: Fitted line: Flame penetration time versus Wt. % of FR boron

7.5.3 Regression analysis for Rate of burning

A regression equation was generated with (F (3,20) = 63.33, $p < .001$), with an R^2 value = 90.5 %. The regression equation used to predict the rate of burning in minutes for coir fibre board samples are given in the equation (7.3).

$$\boxed{Y_3 = 12.49 - 0.6698\,(X) + 0.1314\,(X)^2 - 0.002544\,(X)^3} \tag{7.3}$$

A fitted line plot relating rate of burning (Y_3) in minutes as the ordinate to weight percentage of boron compounds in FR solution (X) as the abscissa is shown in Figure 7.4.

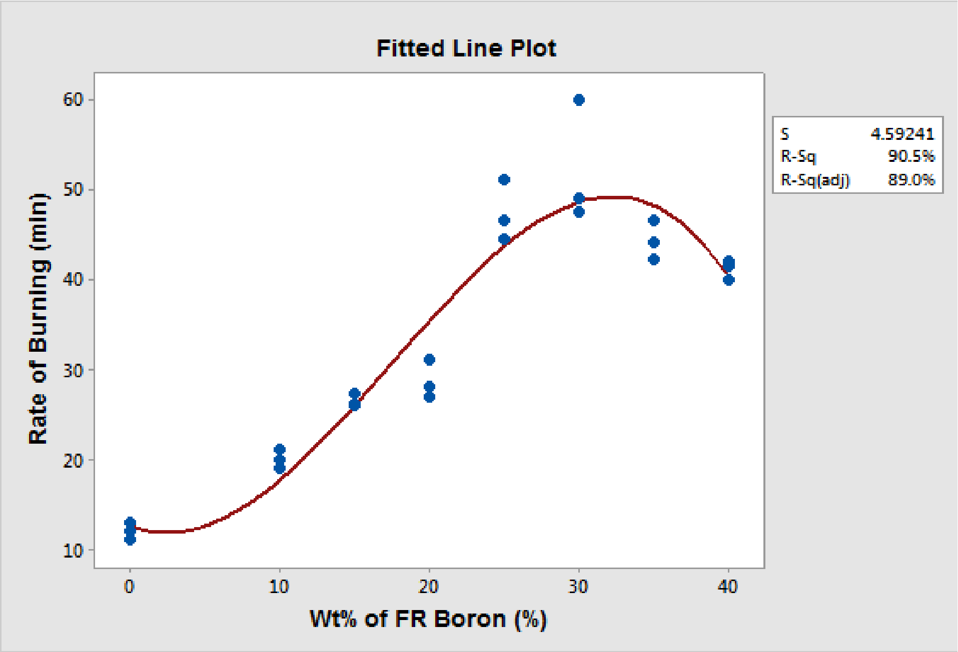


Figure 7.4: Fitted line: Rate of burning versus Wt. % of FR boron

7.5.4 Regression analysis for Resistance to flame spread

A regression equation was generated with (F (3,20) = 179.62, $p < .001$), with an R^2 value = 96.4%. The weight percentage of boron compounds in FR solution present in the FRT coir fibre board samples appears to have an affect on the flame spread.

The regression equation used to predict the resistance to flame spread of coir fibre boards in minutes are described in the equation (7.4).

$$\boxed{Y_4 = 7.071 + 5.936\,(X) - 0.2007\,(X)^2 + 0.002125\,(X)^3} \tag{7.4}$$

Figure 7.5 shows a fitted line plot with resistance to flame spread (Y_4) in minutes as the ordinate and weight percentage of boron compounds in FR solution (X) as the abscissa.

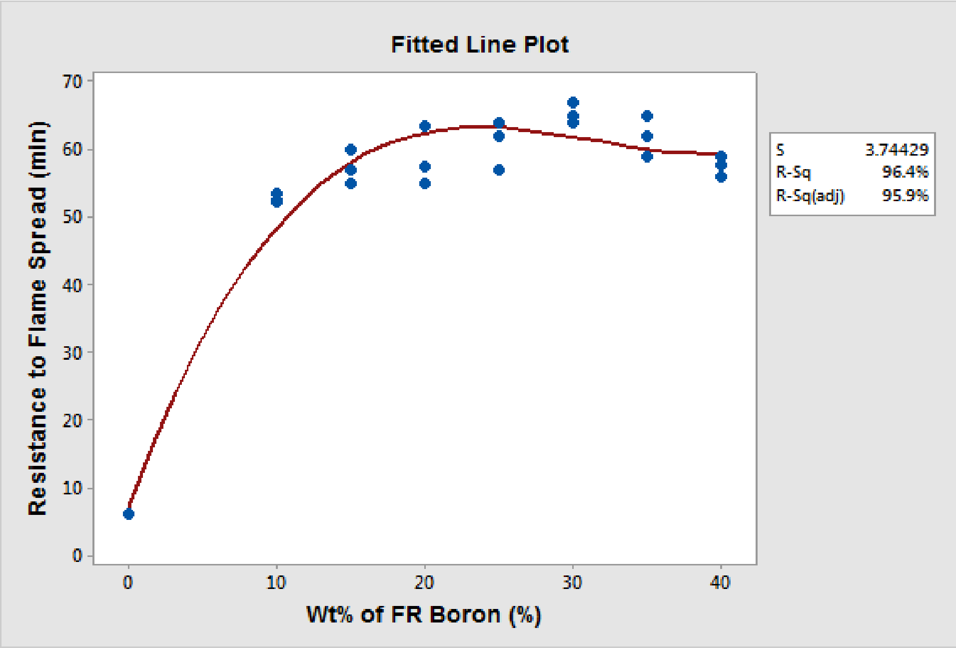


Figure 7.5: Fitted Line: Resistance to flame spread versus Wt.% of FR boron

7.5.5 Predictions of regression analysis

The predicted values for flammability, flame penetration time, rate of burning and flame spread in minutes after regression analysis are tabulated in Table 7.4. The values correspond to the presence of boron compounds within 25 to 35% of the total weight in a solution for fire resistant treatment. The regression analysis predicted maximum values of flammability and flame penetration for samples treated with FR solution having 35% boron in it. Rate of burning values exhibited a maximum for samples treated with 33% boron solution and flame spread was largely unaltered by variations in boron for FR treatment for solutions having 30 - 35% borates in it and exhibited only slight variations in their values.

Table 7.4: Predictions of regression analysis

FR Boron (Wt.%)	**Flammability (min)**	**Flame Penetration Time (min)**	**Rate of Burning (min)**	**Resistance to flame spread (min)**
36	53.5	40.2	46.3	59.8
35	53.6	40.2	47.1	60.0
34	53.5	40.2	47.6	60.3
33	53.2	40.0	47.8	60.6
32	52.6	39.8	47.8	60.9
31	51.8	39.5	47.6	61.3
30	50.9	39.1	47.2	61.6
29	49.8	38.7	46.6	61.9
28	48.6	38.3	45.7	62.2
27	47.3	37.7	44.8	62.5
26	45.9	37.2	43.7	62.7

These predictions indicated that there was no compromise in their fire resistant properties by reducing the amount of FR boron within the range of study. It also suggested that the most frugal combination of FR boron in a solution to treat coir fibre boards was between 30 - 35% borates. However the actual measured values of all the four fire resistance tests corresponding to treatment with a solution having 35% FR boron were accurate, with variations less than ± 3% of the predicted values. Further investigations were done by testing the samples treated with 33% FR boron to confirm the validity of predictions.

7.5.6 Confirmation of regression analysis

Coir fibre board samples were chemically treated with a solution that contained 33% boron compounds (19% BX and 14% BA) and 67% water. A confirmation experiment was performed with all four of the fire performance tests to assess the validity of the regression analysis. Table 7.5 shows both measured values and regression predicted values of all the four fire resistant characteristics of coir fibre board samples treated with FR solution containing 33% boron compounds (19% BX and 14% BA) and 67% water.

Table 7.5: Results of confirmation test with 33% boron

	Flammability (min)	**Flame Penetration Time (min)**	**Rate of Burning (min)**	**Resistance to flame spread (min)**
Measured values	52	40	49	62
Predicted values	53.2	40.0	47.8	60.6

It was found that the predictions were accurate with variations less than ± 3% with in the prescribed range between 10% to 40% boron compounds present in FR solution. Therefore the results suggested that the regression analysis could successfully predict the values for flammability (min), flame penetration time (min), rate of burning (min) and resistance to flame spread (min) with an accuracy of ± 3% when coir fibre boards are fire retardant treated.

7.6 Conclusion

Increasing popularity and use of wood substitutes for building and construction sector makes coir fibre board a possible construction material to substitute engineered wood products like plywood. Since the decreased ability of a material to catch fire accounts to better safety, especially in residential buildings, it is a preferable choice when the materials are treated with fire-retardant chemicals. In places where severe degradation conditions are prevailing, it is also desirable to have better resistance to biodegradation along with fire resistance; boron is an extensively used chemical for wood preservation.

This study evaluated the flame resistance of chemically treated coir composite panel boards for different weight percentage of borates in the solution. The investigation found that the presence of 30% - 35% boron compounds in the FR solution gave better results out of the selected samples under investigation and it was identified as the optimum composition of boron compounds for fire retardant treatment of coir fibre boards. Regression analysis could successfully predict the values for flammability (min), flame penetration time (min), rate of burning (min) and resistance to flame spread (min) with respect to the weight percentage of FR boron present in the solution for chemical treatment with an accuracy of ± 3% and the equations fit were found to be significant.

CHAPTER 8

CONCLUSION

There are numerous design possibilities for products manufactured from composite materials due to the wide variety of available manufacturing processes. Consequently, these products and its manufacturing process can be chosen to best fit the developing rural societies in which the products are made and used. Besides the technical feasibility, manufacturing of composites becomes economically feasible when domestically grown natural fibres are used in combination with simple manufacturing processes. Coir fibre is a natural fibre extracted from the husk of coconut and coir fibre based composite boards are being investigated worldwide for their ability to substitute wood.

Increasing popularity and use of wood substitutes for building and construction sector makes coir fibre board a possible construction material to substitute engineered wood products like plywood. Since resistance to fire accounts to better safety, especially in residential buildings, the materials treated with fire-retardant chemicals are always preferred. In places where severe degradation conditions are prevailing, it is also desirable to have better resistance to biodegradation along with fire resistance. Boron is an extensively used chemical for wood preservation. A large variety of fire-retardant chemicals and their treatment methods are being studied and the combination of BX and BA has been found to be effective in many cases. Coir fibre–based medium-density panel boards were subjected to treatment with a specially prepared solution of BX and BA for enhancing their fire-retardant properties.

A thorough review on the role of coir fibres in sustainable manufacturing of bio-composite boards were done. The annual production and global market potential for coir fibre is carefully reviewed. The market potential of coir fibre based composite boards has been ascertained and compared with similar bio composite boards. The different types of coir composite panel boards, which are already established as per BIS standards and that are available in the market were reviewed and it was found that the fire resistant properties were not adequate. It was seen that the fire retardant properties of the composite panel boards made from coir fibre has to be enhanced for its wider acceptance.

The various types of available fire retardants and their mechanisms inhibiting fire, were reviewed for their suitability to be used for coir fibre boards also to enhance flame resistant behaviour. A mixture of Borax and Boric Acid in the ratio of 1.1.5 were found to impart fire resistance to coir fibre boards without adversely affecting its properties. The common methods adopted for Fire Retardant Treatment were also reviewed to find a suitable chemical treatment process for coir fibre boards. A modified hot and cold bath method was found to be effective for fire retardant treatment of coir fibre boards.

Chemically treated coir fibre board samples were found to be thermally more stable over untreated samples throughout the range of temperatures. Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) analysis revealed that the first decomposition temperature and crystallization enthalpy increased after treatment. The derivative weight loss was decreased after treating the sample with boron. The research gave substantial results to claim that the thermal properties of coir board samples have greatly improved by the chemical treatment with a preservative solution containing boric acid and borax. The research also suggested that the method of chemical treatment used in coir board samples is effective and could increase the thermal stability.

The samples after FR treatment conformed to the required values for density and moisture content as put up by BIS standards with considerable improvement in values. The treated samples exhibited reduced values for thermal conductivity and improved thermal resistance when compared to untreated samples, which means there is an overall improvement in thermal insulation characteristics of FRT coir fibre boards.

The study also estimated the mechanical behaviour of FR treated coir fibre boards using a combination of tensile test, bending test and compression test. The obtained values showed substantial improvements in tensile and bending properties but a slight decrease in compression strength. However, the compression strength of FRT coir fibre boards still conform to the values prescribed in BIS standards. The results suggested that there is no significant loss of strength to coir fibre boards after FR treatment and the performance of FR treated coir fibre board samples were found to be satisfactory against untreated samples.

In this research, coir fibre boards were subjected to chemical treatment with boron compounds and the samples after treatment showed very significant improvement in resistance to flammability, flame penetration, reduced rate of burning and resistance to flame spread. The test for limiting oxygen index showed reduced values of ignitability, suggesting the use of the earlier methods to enhance the fire-retardant properties of coir fibre–based panel boards and use of the panels as substitute for wood-based applications.

In an attempt to reduce the amount of chemicals used in the process, this study also evaluated the flame resistance of chemically treated coir composite panel boards for different weight percentage of borates in the solution. An optimum composition of water-borne preservative solution was identified which gave better results out of the selected samples under investigation. Regression analysis could successfully predict the values of flammability, flame penetration time, the rate of burning and resistance to flame spread with respect to the weight percentage of FR boron present in the solution for chemical treatment and the equations fit were found to be significant.

The present work explored the potential of coir fibre boards and studied their thermal, mechanical and fire resistant properties. Based on the above study chemical modification of coir fibre-based fibre boards were made to successfully develop a fire retardant board which can find a variety of applications as a substitute to wood. The results incorporated in this research are encouraging and suggests the use of FR treatment on coir fibre boards for applications involving stringent fire safety norms. The study also proposes the possibility of developing a new class of fire proof coir fibre boards that can cater to a wide range of applications.

The major outcomes and contributions of this research are listed as below.

- ⋆ A new class of fire retardant fibre boards were developed from coir fibre with enhanced fire resistance properties without compromising its structural abilities.

⋆ The study established that a combination of Borax and Boric acid in the ratio 1:1.5, imparted the best fire resistance parameters to coir fibre boards.

⋆ A modified hot and cold bath method was adopted for FR treatment with boron compounds and the method was found best suitable to impart fire resistance in coir fibre boards.

⋆ The study investigated the most economical combination of FR boron (BA+BX) in a chemical solution to improve fire retardation properties of coir fibre boards and found that 35% boron compounds in an aqueous solution, (i.e., 15% BA, 20% BX and 65% water), imparted the best results in a combination of fire performance tests.

⋆ The effect of fire retardant treatment on the physical and mechanical properties of coir fibre boards were evaluated and it was found that the FRT boards were exhibiting superior properties in all major parameters.

⋆ The thermal stability and fire resistance properties of FRT coir fibre boards were assessed and it was found that the values of FRT coir fibre boards were much superior compared to untreated boards in every aspect.

Apart from the above mentioned contributions, several physical and mechanical properties of coir fibre based composite boards were found during the study which were otherwise not available in the public knowledge domain.

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LIST OF PAPERS

Part of the thesis has been published/accepted for publication in the following internationally refereed and Scopus/SCI indexed journals.

Papers in Refereed Journals

(i) Relative improvements in flame resistance of coir fiber boards treated with fire-retardant solution.
Rejeesh, C. R., Saju, K. K.
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(ii) Effect of fire retardant treatment on mechanical properties of medium density coir composite boards.
Rejeesh, C. R., Saju, K. K.
Wood and Fiber Science, **50(1)**, 113-118 (2018)

(iii) Effect of chemical treatment on fire-retardant properties of medium density coir fiber boards
Rejeesh, C. R., Saju, K. K.
Wood and Fiber Science, **49(3)**, 332-337 (2017)

(iv) Methods and materials for reducing flammability behaviour of coir fibre based Composite Boards: A Review
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