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**MANUFACTURE OF CITRIC ACID BONDED MEDIUM
DENSITY PARTICLE BOARD.**



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LIFE SCIENCE SCHOOL
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THESIS WORK
COURSE NO: FWT- 5112

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DECLARATION

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A handwritten signature in black ink, appearing to read 'Ahmadul Hasan', written over a horizontal line.

Shah Ahmadul Hasan

*Dedicated
To
My beloved Parents*

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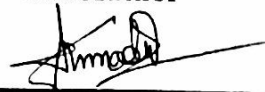
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september, 2017

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APPROVAL

This project thesis has been submitted to the Forestry and Wood Technology Discipline, Khulna University, Khulna, Bangladesh, for the partial fulfillment of professional MSc. degree in Forestry. I have approved the style and format of the project thesis.

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ABBREVIATION

Anon	Anonymous
ANOVA	Analysis of Variance
LSD	Least Significant Difference
BBS	Bangladesh Bureau of Statistics
FAO	Food and Agricultural Organization of United Nations
g/cm^3 or gm/cm^3	Gram per cubic centimeter
kg/m^3	Kilogram per cubic meter
KN	Kilo Newton
lb/ft^3	Pound per cubic feet
cm	Centimeter
m	Meter
mm	Millimeter
μm	Micro meter
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MPa	Mega Pascal
N/mm^2	Newton per square millimeter
UTM	Universal Testing Machine
ANSI	American National Standard Institute
IS	Indian Standard
ANZS	Australian Newzland Standard
JIS	Japan Standard
ASTM – D-1037partB	Test Methods for MDF

ABSTRACT

In this study citric acid bonded particle board has been developed successfully using agricultural residues. Here different concentration of citric acid were used among them 30% citric acid concentration showed good result in case of physical and mechanical properties of the board. Here, target board density was around 0.78gm/cm^3 and its properties were as- Moisture content (8.04%); water absorption (63.37%); thickness swelling (11.90%); Modulus of Rupture (MOR, 13.84 N/mm^2); Modulus of Elasticity (MOE, 4519.67 N/mm^2); Internal Bonding Strength (IB) 0.68 Mpa and Surface Soundness (SS) 1.0 Mpa. It was found that all the properties of the board were promising and satisfied the ANSI standard except water absorption properties. Hence, it appears that manufacturing of citric acid bonded medium density particleboard is technically feasible for various structural purposes.

CHAPTER ONE: INTRODUCTION

1.1 Background of the study:

At present industries are now very much interested to produce eco-friendly product. This product will be economically viable, environment friendly and does not have any hazardous material to health. Due to increasing global warming effect and raising environmental concern, present trends are shifting from synthetic composite to natural renewable one. For this reason industries are now in pressure to manufacture eco-friendly product to cope with the present market demand and to consider the environmental condition. So, researcher are now more interested in this regard. Agricultural residues may play a vital role as a renewable resource for this purpose due to its increasing production. As an alternative of wood, wood based materials like Particle Board plays a significant role in our daily life. Generally particle boards are used in housing construction and furniture manufacturing. It seems these wood based materials are frequently present in living environments (Sellers 2001; Zheng *et al.* 2007).

Wood based materials are commonly bonded with synthetic resins such as formaldehyde based, isocyanate based and vinyl acetate resins (Yang *et al.* 2006). These resins derived from fossil resources have satisfactory adhesion capability and superior working properties. As fossil resources are limited and in addition types of synthetic resin used in particle board manufacturing are toxic in nature, some are costly, and costing higher energy and some releases harmful chemical substances which cause environmental problems, health disorders. Considering the sustainability and global environmental effect and the potential scarcity of fossil resources, it is indispensable to reduce the consumption of synthetic adhesive and desirable to develop of natural adhesive derived from non-fossil resources to be safe adhesive without using harmful chemical substances.

To address these problems, natural adhesive derived from renewable resources such as protein, tannin, lignin, and starch are being investigated (Trosa and Pizzi 1997; Lei *et al.* 2011; Li *et al.* 2015). Many researchers have been reported ways of reducing the utilization of the synthetic adhesive such as development of natural adhesives (Xu J, Widyorini R, Kawai S, 2005). Researchers recently found that citric acid can be used as a natural adhesive for wood-based molding (Umemura *et al.* 2012a, 2012b). Citric acid (2-hydroxy-1, 2, 3-propanetricarboxylic acid) is an organic polycarboxylic acid containing three carboxyl groups. It is contained in citrus fruits such as lemons and limes and is commercially produced by fermenting glucose or glucose and sucrose-containing materials (Abou-Zeid and Ashy

1984, Tsao *et al.* 1999). It is widely used in food, beverages, and pharmaceuticals. In addition, citric acid has been researched as a cross-linking agent for wood (Vukusic *et al.* 2006, Hasan *et al.* 2007; Bogoslav *et al.* 2009), plant fiber (Ghosh *et al.* 1995), paper (Yang *et al.* 1996), starch (Yu *et al.* 2005; Reddy and Yang 2010), bioresource-based elastomers (Tran *et al.* 2009), and absorber for heavy metal ion (Thanh and Nhung 2009). Cross-linking chemicals reacting with hydroxyl groups reduced the hygroscopicity of wood and the tendency to swell or shrink (Rowell 1991; Vukusic *et al.* 2006). The possibility of using citric acid as a natural adhesive for wood was investigated by fabricating acacia wood and bark moldings (Umemura *et al.* 2011, 2012a, b). The results showed that citric acid-bonded molding had good mechanical properties and water resistance. The adhesion mechanism for citric acid is ester linkages between carboxyl groups from citric acid and hydroxyl groups from wood (Umemura *et al.* 2012a and Widyorini *et al.* 2014). In addition, citric acid-bonded composites made from non-wood materials are still limited. In this study, application of citric acid as a natural adhesive was investigated for medium density compositeboard manufactured from kenaf, jute and coir.

Kenaf (*Hibiscus cannabinus*) is a well-known fast growing fiber plant native to east-central Africa. . Being an exotic species in our country kenaf is well adapted in our climate. It has faster growth and higher yield which is very much vital in its utilization. It is a promising source of raw material for particleboard, fiber for pulp, paper, and other fiber products. Kenaf seems more considerable for these purposes because of its fibres, especially both the outer part and core part which is low cost, low density, high toughness, suitable for recycling, acceptable strength properties and biodegradability. Jute stick is very much popular in our country. At present particle board is manufactured from jute stick. So, this raw material plays a vital role as an alternative raw material of wood and can be used as a raw material of composite board. On the other hand coir fiber (*Cocos naucifera*) is very much popular and has good characteristics to be used. This study was to investigate the influence of resin content to the mechanical and physical properties of medium density composite board.

1.2 Objectives of the study:

- ✓ To assess the feasibility of using jute stick, kenaf stick and coir fiber to manufacture binderless particle board.
- ✓ To know the influence of citric acid and its optimum concentration on the properties of the board.

CHAPTER TWO: LITERATURE REVIEW

2. General information about particleboard

2.1. Definition of particleboard

A particleboard is a board (or sheet) constituted from fragments of wood and/or other lingo-cellulosic materials (chips, shavings, flakes, splinters, sawdust, etc.), bonded with organic binders with the help of one or more agents like heat, pressure, humidity, catalyst, etc. (Srivastava, 2012). It may be classified as a panel product manufactured under pressure and heat from particles of wood or other lingo-cellulosic materials bonded entirely with a binder, generally a synthetic resin, to which other chemicals (e.g., fire retardant, fungicide, water retardant etc.) may be added to improve certain properties (Salehuddin, 1992).

2.2. History and Development of particleboard

Particleboards are not more than a few decades old production. First unsuccessful efforts were made in the early 1920's for manufacturing as for the lack of suitable adhesives. New techniques introduced in the 1930's in resin applications paved the way for the industrial production of particleboard in the early 1940's (Moslemi, 1985). Today's particleboard provides industrial users the high, consistent quality and range of "design" flexibility needed for fast, efficient lines and high-quality products that consumers require.

It was thought up by German Luftwaffe fighter pilot and inventor Max Himmelheber, in first made in a factory in Bremen, Germany during the Second World War. Those first boards were made up of wood scraps, shavings, sawdust, and offcuts, all glued together using a specialist resin. Before the pieces were glued together, they would be smashed and pounded in order to create small enough pieces that would pass through a specific sized wire mesh.

When particle board first came onto the market, it was only made available to the wealthy because the price was higher than some real timbers. Over time, the manufacturing process matured, the supply became steadier, and the material became cheaper. Some of the biggest furniture manufacturers on the planet have grown because of the wide availability and low cost of particle board. In some areas, the low cost and increasing durability of particle board has overtaken and often displaced solid wood in the manufacture of low cost and medium cost furniture.

2.3 Advantages of particleboard

- Particleboards overcome some inherent weakness of solid wood and make useful products out of wastes, small pieces of wood and inferior species thus ensuring complete utilization of raw materials, make products with unique properties and can tailor products for particular end-use.
- The characteristic defects of wood such as knots, spiral grain, etc., may either be eliminated or scattered throughout the particleboard during manufacturing. Thus ensure not occurring defects during service condition.
- The variation in strength and stiffness due to anisotropy in wood is largely overcome as also the differential change in dimension due to absorption and desorption of moisture along or across the grain of wood.
- During the manufacture of particleboard, various treatments, such as heating, incorporation of chemical additives, etc. may be carried out to improve many physical and mechanical properties including the dimensional stability.
- By using different species and adhesives, or particles of different size and geometry, particleboard may be manufactured suitable for exposure to weather, for interior use, for interior paneling, for exterior sideboards, for load bearing flooring purposes and so on.
- Perhaps the most important advantage of particleboard is that it can be made in large dimensions (Salehuddin, 1992).

2.4 Considerations for the quality of particleboard

Quality of particleboard largely depends on the following factors:

- Density
- Layering
- Types of adhesive
- Geometry of the particles i.e. length, width, thickness, diameter, etc.
- Species from which the raw materials are collected.
- Slenderness ratio. Surface quality and internal bond strength are higher with small particles, i.e. with lower slenderness ratio (Salehuddin, 1992).

2.5 Raw materials for particleboard manufacturing

2.5.1 Woody materials

- a) Planer savings
- b) Sawmill residues, such as slabs, edging, trimmings, etc.
- c) Residues from timber cutting in furniture and cabinet manufacturing plants.
- d) Residues from match factories (Kadam, Chatian)
- e) Veneer and plywood plant residues
- f) Saw dusts
- g) Logging residues, such as short logs, broken logs, crooked logs, small tree tops and branches, forest thinning , etc, and
- h) Bark

2.5.2 Non-woody materials

- a) Jute sticks
- b) Kenaf
- c) Bagasse
- d) Bamboo
- e) Flax shaves
- f) Cotton stalks
- g) Cereal straw
- h) Coir fiber
- I) Almost any agricultural residue after suitable treatment (Salehuddin, 1992)

2.6 Chemicals

2.6.1 Binder or Adhesive

Adhesives are substances capable of holding materials together in a useful manner by surface attachment. The principle attribute of adhesives is their ability to form strong bonds with surfaces of a wide range of materials and to retain bond strength under expected use conditions (Lehman, R. L., 1988).

These adhesives have been chosen based upon their suitability for the particular product under consideration. Factors taken into account include the materials to be bonded together, moisture content at time of bonding, mechanical property and durability requirements of the resultant composite products, and of course resin system costs.

2.6.2 Types of adhesive/ binder

There are mainly two types of adhesive. One originated from natural sources known as natural adhesive and another is synthetic adhesive.

Synthetic adhesive

Adhesives of synthetic origin are called synthetic adhesives. These are man made polymers which resemble natural resins in physical characteristics but which can be tailored to meet specific wood working requirements.

a) Thermosetting adhesives

These types of adhesives are usually based on formaldehyde. Thermosetting adhesives undergo a chemical change during application and curing. The bonds formed by thermosetting adhesives are generally moisture resistant and support loads under normal use. During the polymerization, or chain – building step, thermoset polymers form links, or chemical bonds, between adjacent chains. The results are a three – dimensional network that is much more rigid than the linear thermoplastic structure. The interlinked chains are not free to move when heat is applied, and the thermoset as the name implies, is “set” into a permanent shape after polymerization. The level of cross linking can be varied. Materials with high cross – linking densities are hard, rigid and somewhat brittle substances. Thermosets with low cross – linking densities can be softened by heating to high temperatures, but they do not melt and their original shape is retained (Gilleo, K. et al.). Some characteristics and uses of some thermosetting adhesives are listed below–

- i. Phenol formaldehyde:
- ii. Polymeric diphenylmethane diisocyanates (PMDI)
- iii. Urea-formaldehyde (UF)
- iv. Melamine-formaldehyde (MF)
- v. Phenol Resorcinol formaldehyde (PRF)

b) Thermoplastic adhesives

Thermoplastic adhesives are especially useful because they can be used in a dry form and are already fully polymerized as received. The bonding process basically involves softening or melting the polymer while in contact with their adherents, and allowing the joint structure to cool. The structure can be easily disassembled or repositioned by reheating while applying force. These materials have been used for some time under such terms as heat-activated, hot bond and hot melt adhesives. Thermoplastic adhesives are convenient, safe and highly

reliable. These are based on poly-vinyl acetate (PVAC). They generally have less resistance to heat, moisture, and long-term static loading to do thermosetting polymers. Common wood adhesives that are based on thermoplastic polymers include polyvinyl acetate emulsions, contacts, hot-melts etc. (Vick, 1999).

Natural adhesive

Before synthetic adhesives were introduced in the 1930s, adhesives made from natural polymers found in plants and animals were used for bonding wood. These adhesives were made from animal blood, hide, casein, starch, soybean, dextrin and cellulose. Some natural options may someday replace or supplement synthetic resins. Tannins, which are natural phenols, can be modified and reacted with formaldehyde to produce a satisfactory resin. Resins have also been developed by acidifying spent sulfite liquor, which is generated when wood is pulped for paper. In the manufacture of wet-process fiberboard, lignin, which is inherent in lingo-cellulosic material, is frequently used as the resin (Suchland and Woodson, 1985). Considering the sustainable global environmental and the potential scarcity of fossil resources, it is essential to reduce the consumption of synthetic adhesive. Development of natural adhesive derived from non fossil resources is desirable as a safe adhesive without using harmful chemical substances.

Sources of Natural Wood Adhesives (Source: Kenji Umemura and Shuichi Kawai, 2015)

- a) Saccharide based
 - Starch
 - Cellulose etc.
- b) Protein based
 - Animal glue
 - Casein
 - Soy protein
 - Blood albumin etc.
- b) Aromatic based
 - Lignin
 - Tannin etc.
- c) Oil based
 - Castor oil
 - Canola oil etc.
- d) Others

→ Natural rubber, Liquefied wood etc

2.7 Citric acid as natural adhesive

Citric acid is a weak organic tricarboxylic acid having the chemical formula $C_6H_8O_7$. It occurs naturally in citrus fruits. Citric acid is an organic polycarboxylic acid containing three carboxyl groups, which is commercially, produced by fermenting glucose or glucose and sucrose containing materials. Lemons and limes have particularly high concentrations of the acid; it can constitute as much as 8% of the dry weight of these fruits about 47 g/L in the juices (Penniston KL et al. 2008).

2.7.1 Chemical characteristics

Citric acid exists in greater than trace amounts in a variety of fruits and vegetables, most notably citrus fruits. Citric acid was first isolated in 1784 by the chemist Carl Wilhelm Scheele, who crystallized it from lemon juice (Frank H. Verhoff, 2005). It can exist either in an anhydrous (water-free) form or as a monohydrate. The monohydrate can be converted to the anhydrous form at 78 °C.

Citric acid is normally considered to be a tribasic acid, with pKa values, extrapolated to zero ionic strength, of 5.21, 4.28 and 2.92 at 25 °C (Goldberg R.N., et al. 1986). The pH of a 1mM solution of citric acid will be about 3.2. The pH of fruit juices from citrus fruits like oranges and lemons depends on the citric acid concentration, being lower for higher acid concentration and vice versa.

2.7.2 Applications of citric acid

It is used widely as an acidifier, as a flavoring and chelating agent (Apleblat, Alexander., 2014). The other applications of citric acids are:

- ❖ It is one of the stronger edible acids, the dominant use of citric acid is as a flavoring and preservative in food and a beverage, especially soft drinks Citric acid is an excellent chelating agent, binding metals. It is used to remove lime scale from boilers and evaporators. (Frank H. Verhoff, 2005).
- ❖ Citric acid is widely used as an acidulate in creams, gels, and liquids of all kinds.
- ❖ Citric acid is commonly used as a buffer to increase the solubility of brown heroin.
- ❖ Citric acid is used as one of the active ingredients in the production of antiviral tissues. (Tissues that fight germs". CNN. July 14, 2004).
- ❖ Citric acid is an alpha hydroxy acid and used as an active ingredient in chemical peels.

- ❖ Citric acid is used as an odorless alternative to white vinegar for home dyeing with acid dyes.
- ❖ Sodium citrate is a component of Benedict's reagent, used for identification both qualitatively and quantitatively, of reducing sugars.
- ❖ Citric acid can be used as an alternative to nitric acid in passivation of stainless steel ("Pickling and Passivating Stainless Steel". PDF)
- ❖ Citric acid can be used as a lower-odor stop bath as part of the process for developing photographic film. Photographic developers are alkaline, so a mild acid is used to neutralize and stop their action quickly, but commonly used acetic acid leaves a strong vinegar odor in the darkroom (Anchell, Steve. 2013)
- ❖ Citric acid/potassium-sodium citrate can be used as a blood acid regulator.

2.7.3 Potentiality of citric acid as an adhesive

More than a million tons of citric acid is manufactured every year. Researchers recently found that citric acid can be used as a natural adhesive for wood-based molding (Umemura et al. 2012a, 2012b). Citric acid has been researched as a cross-linking agent for wood (Hasan et al. 2007; Vukusic et al. 2006), plant fiber (Ghosh et al. 1995), paper (Yang et al. 1996), starch (Reddy and Yang 2010; Yu et al. 2005), and absorber for heavy metal ion (Thanh and Nhung 2009). Cross-linking chemicals reacting with hydroxyl groups reduced the hygroscopicity of wood and the tendency to swell or shrink (Rowell 1991; Vukusic et al. 2006).

2.8 General information about raw material (Jute stick, kenaf stick and coir fiber)

Kenaf Stick

Kenaf (*Hibiscus cannabinus* L.) is a fiber plant native to east-central Africa where it has been grown for several thousand years for food and fiber. It is a common wild plant of tropical and subtropical Africa and Asia. (LeMahieu et al. 2003) It has been a source of textile fiber for such products as rope, twine, bagging and rugs. Kenaf is a promising source of raw material fiber for pulp, paper, particleboard and other fiber products. (Sources: Wikipedia)



Figure 2.1: Image of Kenaf plant

Coir fiber

The English word "coir" comes from the Malayalam and Tamil word 'kayar' (കയർ in Malayalam and கயிறு in Tamil). Coir or coconut fibre, is a natural fibre extracted from the husk of coconut. Indian. Red coir is used in floor mats and doormats, brushes, mattresses, floor tiles and sacking. A small amount is also made into twine. Rubberized coir is used as upholstery padding for the automobile industry in Europe.



Figure 2.2: Coconut and coir fiber image

Jute stick

The word 'jute' is probably coined from the word jhuta or jota, an Oriya word. Jute is one of the most affordable natural fibers and it is second only to cotton in amount produced and variety of uses of vegetable fibers. Jute fibers are composed primarily of the plant materials cellulose and lignin. Moreover, jute can be grown in 4–6 months with a huge amount of cellulose being produced from the jute hurd (inner woody core or parenchyma of the jute stem) that can meet most of the wood needs of the world. *Jute is the major crop among others that is able to protect deforestation by industrialization.* In the 21st century, jute again rose to be an important crop for export around the world in contrast to synthetic fiber, mainly from Bangladesh.



Figure 2.3: Image of Jute plant

2.9 Worldwide production status of jute, kenaf and coir fiber

Global production of JACKS (Jute, abaca, coir, kenaf and sisal) fibres increased by 1.6 percent to 4.37 million tonnes in 2014, compared to output levels in 2013.

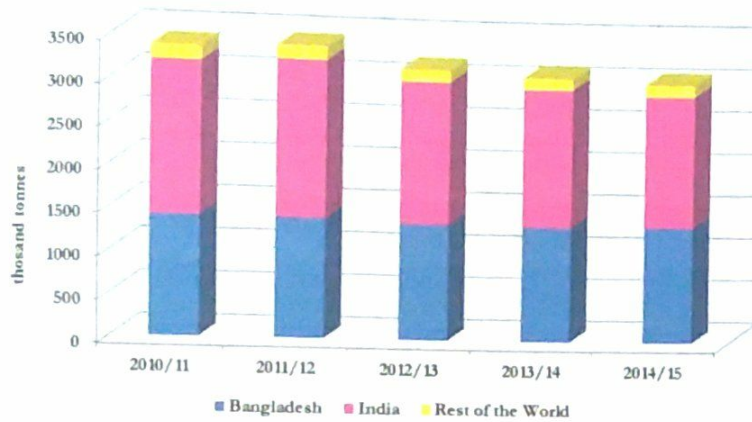


Figure 2.4 – Jute and Kenaf Production (thousand tonnes) [Source: Secretariat FAO IGGHF/JU]

Global production of brown coir fibre increased by 11 percent to reach 918 300 tonnes in 2014. India accounted for nearly 50% of the total.

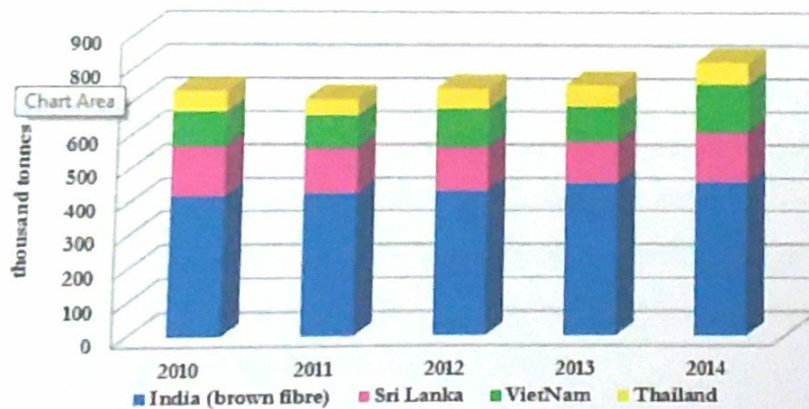


Figure 2.5 – Brown coir fiber Production (thousand tonnes) [Source: Secretariat FAO IGGHF/JU]

This above stated statistics shows that the increasing production of jute, kenaf and coir has promising possibility to be used industrially in near future.

2.10 Chemical characteristics of jute, kenaf and coir fiber

Table 2.1: Comparative chemical characteristics of jute, kenaf and coir fiber are-

Raw material	Cellulose %	Hemicellulose	Lignin	Fiber length microns	Density (g/cm ³)	Elongation (%)	Tensile strength (MPa)	Young's modulus (GPa)	Ash	Pectin
Jute stick	41-48	23-23.6	22.2-23.5		1.3-1.49	1.16-1.5	393-800	13-26.5	0.8	0.2
Kenaf stick	37-49	21.5-23	15-21	400-1100	1.4	1.6	930	53	2-4	3-5
Coir fiber	32-43	0.15-0.25	40-45	Length-0.3 -0.1 (mm), width 100 - 450 (µm)	1.2	15.0-40.0	175-220	4.0-6.0	2	3-4

Source: Frederick TW, Norman W (2004)

Here, the amount of the production of the residues and its chemical properties shows that these residues have the huge possibility in future to be used industrially. For this reason I have selected these raw materials for my study to show its feasibility to be used industrially and eco-friendly.

2.11 Literature survey regarding the natural adhesive

Widyorini 2013 showed the development bio-based composites made from bamboo biomass waste materials. This report focused on the physical and mechanical properties of the particleboards. The author also tried to investigate the possibility of using citric acid to improve the properties of the boards. Petung bamboo particles (*Dendrocalamus asper* Backer) were used in this research. The contents of citric acid were set in 0% (binderless board), 10%, and 20% based on dried particles, were hot pressed at 200°C and 220°C for 10 and 15 min. The research showed bamboo particleboards bonded with citric acid had high performance on mechanical properties and good dimensional stability.

Umemura *et al.* 2011 discussed the application of citric acid as natural adhesive for wood. They showed the effects of Molding Temperature on the Physical Properties of Wood-Based Molding Bonded with Citric Acid. In their study they use Citric acid powder as an adhesive and bark powder obtained from *Acacia mangium* was used as raw materials. Citric acid powder was mixed with the bark powder, and the resulting powder mixture was poured into a metal mold, hot pressed at a pressing temperature of 180°C, at 4 MPa for 10 minute. In addition they also clarify the effect of tannin on the adhesiveness of molding. Widyorini *et al.* 2015, describes the manufacturing and properties of citric acid-bonded particleboard made

from bamboo materials. Their study investigated the physical and mechanical properties of particleboard made from bamboo using citric acid as natural binder. Petung bamboo (*Dendrocalamus asper*), wulung bamboo (*Gigantochloa atrovioleacea*), and apus bamboo (*Gigantochloa apus*) were used as raw materials in this research. Citric acid contents were set at 0, 15 and 30 wt. % based on air-dried particles. The properties of citric acid-bonded bamboo particleboard in this research could meet the requirements of the Japanese Industrial Standard for particleboard (A 5908). They also showed that addition of citric acid could significantly improve the dimensional stability and mechanical properties of the boards.

Umemura *et al.* 2014, focused on Investigating of a new natural adhesive composed of citric acid and sucrose for particleboard; they also discuss the effects of board density and pressing temperature. Recycled wood particles consisting mainly of softwood obtained from waste wood were given by a particleboard company in Japan. The relationship between board density and board physical and mechanical properties were observed under a press temperature at 200°C.

Indrayani Y. *et al.* 2015, Evaluated medium density fiberboard (MDF) which was termite resistance, manufactured from agricultural fiber bonded with citric acid. In this study, citric acid application as natural adhesive was investigated for medium density fiberboard (MDF) manufacture from pineapple leaf fiber. Using the ratio of citric acid and sucrose (25–75 the board was hot pressed at 200°C and 4.5 MPa for 10 min. Additional boards with same structure were prepared using citric acid only. The biological properties of the boards such as their resistance against subterranean termite attack have been examined. The results indicate that, there was a significant effect of impregnation with mixture of citric acid and sucrose and citric acid only on the susceptibility of the MDF board specimens.

Kenji Umemura and Shuichi Kawai (2015) investigate the development of Wood-Based Materials Bonded with Citric Acid. They showed the bonding properties of citric acid in wood-based moldings and particleboards. In wood-based molding, wood and bark powders were used as elements. Citric acid powder was mixed with wood or bark powders, and the mixture was hot pressed at 200°C for 10 minutes resulting good mechanical properties and excellent water resistance. For particleboard, sucrose was used in addition to citric acid, dissolving them in a water solution (25/75 ratio) and the solution used as an adhesive.

Particleboard was manufactured at 200°C for 10 minutes yields good mechanical properties and water resistance particleboard. They tried to establish citric acid as a bio-adhesive for wood. Widyorini *et al.* 2016, investigated the bonding ability of a new adhesive composed of citric acid-sucrose for teak particleboard. The physical and mechanical properties of the particleboards showed that increasing the pressing temperature affected the dimensional stability. However, increasing of citric acid in adhesive composition improved the dimensional stability and mechanical properties of the particleboards. The optimum properties of the board were achieved at a pressing temperature of 200 °C and addition of only 10% citric acid. Zhao *et al.* 2016, showed the effect of the addition of citric acid on the curing properties tannin-sucrose adhesive and on the physical propertied of the particleboard. This paper also showed that, the addition of citric acid promoted the reaction between tannin and sucrose at a lower temperature, mechanical properties and water resistance of the board were also improved.

CHAPTER THREE: MATERIALS AND METHODS

3.1 Collection of Raw Materials

Kenaf was obtained from Agrotechnology Discipline and jute stick was collected from wood technology lab of Forestry Discipline of Khulna University. Coir fiber was collected from Joragate Khulna.

3.2 Processing & Screening of Raw Materials

Firstly jute stick and kenaf stick was grounded into particle by using mesh opening at 2mm. Then grinded particles were screened through a mesh for classifying the coarse and fine particles. Particles between 1-0.5 mm were classified as coarse and the particles size less than 0.5 mm was classified as fine particles. The coarse and fine particles were used individually for single layer composite board. Mixture of coarse and fine particles also used for layered board.



Figure 3.1: Processing and screening of kenaf particle

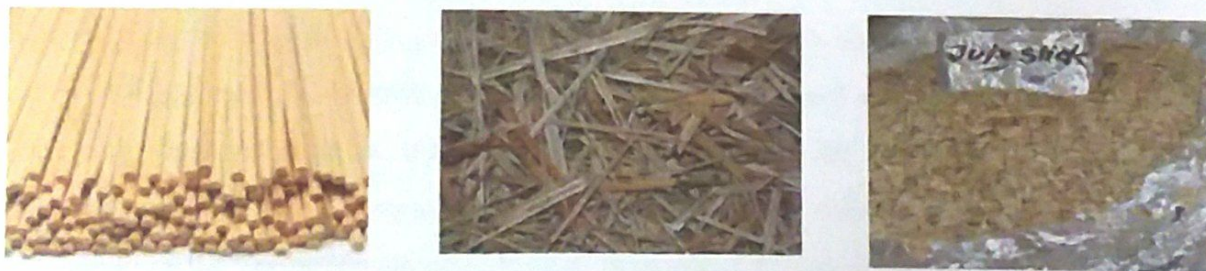


Figure 3.2: Processing and screening of Jute stick particle



Figure 3.3: Processing of coir fiber

3.3 Solution preparation & Drying of Raw Materials

The air dried particles were then mixed with citric acid. Anhydrous citric acid and water was used to make the solution. Concentration of citric acid in the solution was 59-60%. The raw materials were oven dried at 80°C for 24 hours. Then solution was used as an adhesive and sprayed over the particle. The particles were blended manually.

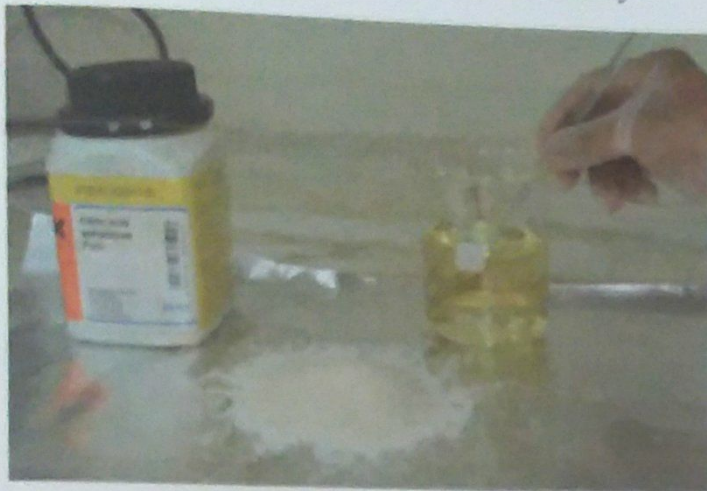


Figure 3.4: Solution preparation

Citric acid contents were set at 0, 10, 15, 20, 25 and 30% based on air dried particles. The sprayed particles were further oven dried for 24 hours at 80°C to reduce the moisture. The moisture content of the oven dried particle was 12-19%. Due to excessive moisture content dangerous explosion may be occurred. For this reason this procedure was maintained strictly.

3.4 Mat Formation

After drying the particles the mat was formed by using a wooden forming box, followed by hot pressing to make the board. Mixed board and layered board were prepared using different citric acid concentration as treatment like 0, 10, 15, 20, 25, and 30%. The average mat thickness was 50 mm. Compositions of the layered board was 1:2:1 (face:core:back). The dimension of the prepared mat was 300 mm in length, 200 mm in width.

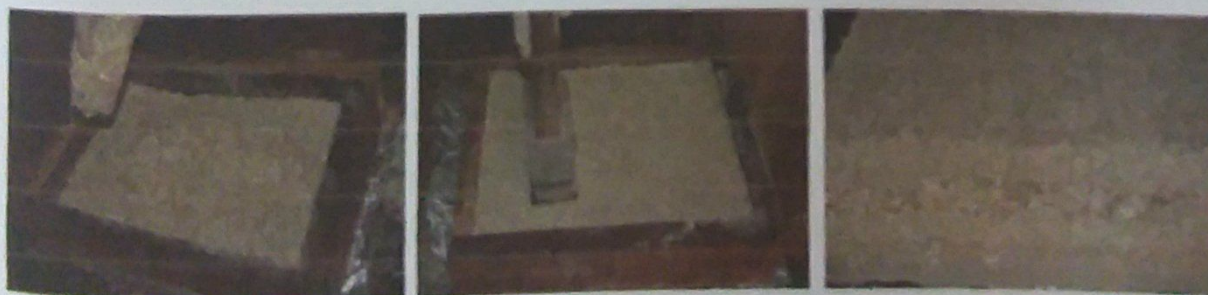


Figure 3.5: Mat formation

Here fine kenaf particle was used in face and back portion and in the core portion jute stick and coir fiber mixture was used at different ratios. For 0, 10, 15, 20, 25, and 30% citric acid concentration coir fiber was used at the ratio of 10, 20, 30, 40 and 50 respectively. In mixed board it was used in ratio of 50 with jute stick. Thus the mat was produced for final pressing.

3.5 Hot Pressing

A steel sheet was placed on the mat after finishing mat formation. Then, mats were pressed on a computer controlled hot press under temperature at 180°C and 5MPa. The temperature switch was switched off after 8 and 10 minutes. The mat were pressed at 180°C at a pressure of 5 MPa for 8 minutes (when 30% citric acid used), 10 minutes (when 25%, 20%, 15%, 5% citric acid used). Binderless particle board was also prepared at pressing temperature 190°C, at 5MPa for 10 minutes pressing. Density of the board was ranged from 0.72- 0.78 g/cm³. During hot pressing eight millimeter distance bar used to control the board thickness.



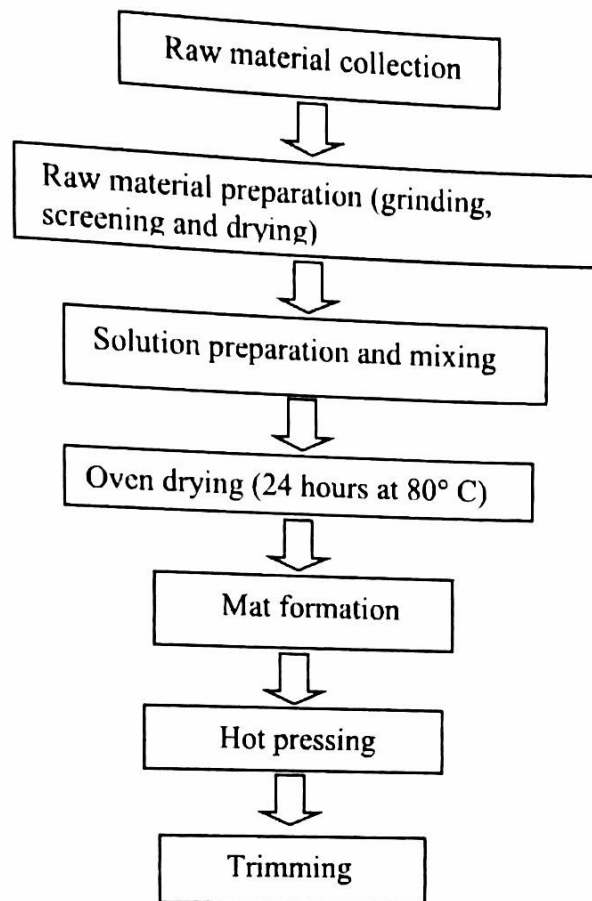
Figure 3.6: Hot pressing & board preparation

After stopping temperature the board was remained fixed for cooling or conditioning. The hot boards were removed from the press and further conditioned to equilibrate board moisture content and to stabilize and fully cure the adhesives (AWPA, 2001).

3.6 Trimming

After the boards of each type were produced separately, these were trimmed at edges with the fixed type circular saw. The board is trimmed to obtain the desired length and width and to square the edges. Trim losses usually amount to 0.5% to 8%, depending on the size of the board, the process employed, and the control exercised (Youngquist, 1999).

3.7 Flow Diagram of Board Preparation



3.8 Manufacturing place & Laboratory Test

The composite board was manufactured at Pulp and paper technology laboratory and wood lab that are controlled by Forestry and wood technology discipline, Khulna University, Khulna. All physical tests for its quality were also done in bio-nano technology lab, and MOE, MOR, IB were tested from Akij Particleboard industry Ltd. The properties were tested according to ASTM D 1037 (American standard). and properties were tested according to

3.9 Board properties Evaluation

The properties of the board were evaluated according to the American standard (ANSI A208.2-2002). Mechanical tests like modulus of elasticity (MOE), modulus of rupture (MOR), internal bonding strength (IB) and physical tests like water absorption (WA), thickness swelling (TS), linear expansion (LE) were tested. Every sample was prepared at a dimension of 300mm x 200mm x 8 mm for static bending test. For testing physical properties, four samples were collected from each board of each type for testing physical properties. Moisture content, water absorption, thickness swelling and linear expansion were determined after 24 hours of soaking under water. The dimension of samples for testing the physical properties and IB strength was approximately 50 mm x 50 mm x 8 mm.

3.10.1 Determination of physical properties

3.10.1.1 Density

Density (D) of each board was calculated after measuring weight and volume using the following equation-

$$D = \frac{m}{v}$$

Where m is the mass and v is the volume of each sample.

(Deschand Dinwoodie, 1996)

3.10.1.2 Moisture content

After measuring the initial mass and oven-dry mass moisture content (MC) was determined by following equation-

$$mc (\%) = \frac{m_{int} - m_{od}}{m_{od}} \times 100$$

Where m_{int} is the initial mass and m_{od} is oven-dry mass of the sample.

(Deschand Dinwoodie, 1996)

3.10.1.3 Water Absorption

The water absorption (A_w) and thickness swelling (G_t) were determined by soaked in water for 24 hours. The water absorption and thickness swelling rate were increased with the time passed. After 24 hours the water absorption and thickness swelling were calculated by an electric balance and a digital slide caliper as a percentage.

Water absorption was calculated by the following formula-

$$A_w(\%) = \frac{m_2 - m_1}{m_1} \times 100 \quad (\text{ASTM, 1997})$$

Where m_1 is the weight of the sample before immersion and m_2 is the weight of the sample after immersion in water.

3.10.1.4 Thickness Swelling

Thickness Swelling was determined by using the following equation-

$$G_t = \frac{t_2 - t_1}{t_1} \times 100 \quad (\text{ASTM, 1997})$$

Where t_1 is the sample thickness before immersion and t_2 is the sample thickness after immersion into water.

3.10.2 Determination of mechanical properties

3.10.2.1 Modulus of elasticity (MOE) and Modulus of rupture (MOR)

By using Universal Testing Machine followed by three points bending test modulus of elasticity (MOE) and modulus of rupture (MOR) were determined for each board. MOR and MOE were calculated by following formulas-

$$MOE = \frac{P/L^3}{4\Delta/bd^3} \quad (\text{Deschand Dinwoodie, 1996})$$

$$MOR = \frac{3PL}{2bd^2} \quad (\text{Deschand Dinwoodie, 1996})$$

Here,

P represents load in the limit of proportionality (N);

L is the length of the span (mm);

b is the width (mm);

d is the thickness (mm) and

Δ represents the deflection at the limit of proportionality (mm)

3.10.2.2 Internal bonding test

Adhere a test piece to steel or aluminum blocks, apply a tension load vertically to the board face, measure the maximum load (P') at the time of failing force (breaking load of perpendicular tensile strength to the board), and calculate the internal bond from the formula below.

In this test, the tension loading speed shall be approximately 2 mm/min.

$$\text{Internal bond (N/mm}^2\text{)} = P'/2bL$$

Where,

P': maximum load (N) at the time of failing force

b: width (mm) of sample

L: length (mm) of sample

(JIS A 5908, 2003)

3.10.2.3 Surface Soundness test

Strength or quality of bonding between the particles or fibres at the surface of a board and the layer below (unfaced panels) or between the coating material and the underlying board (overlaid panels). Measurement of the tensile load required to pull off a defined surface area of overlaid or unfaced panel.

To carry out the test in accordance with EN 326-1, each sample is cut into 50 mm × 50 mm.

The surface soundness SS for each test piece in newton's per square millimeter shall be calculated from the equation

$$SS = F/A$$

Where,

F, is the maximum force in Newtons;

A, is the surface area given in mm².

Express the result to the nearest 0,01 N/mm².

3.11 Experimental Design

For data analysis 6 treatments of different citric concentration (0%, 10%, 15%, 20%, 25%, and 30%) and 4 replications of each treatment were taken and CRD (Completely Randomized Block Design) was applied.

3.12 Statistical Analysis

All the data, produced during the laboratory tests for characterization of physical and mechanical properties of each type of particleboards, were analyzed by Excel and Minitab-17 statistical software.

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CHAPTER FOUR: RESULT & DISCUSSION

4.1 Board properties in different citric acid content:

The relationship between board physical and mechanical properties with different citric acid content was compared under a pressing temperature 180°C. Six types of board were prepared on different citric acid concentration. Each board was three layer at the ratio of 1:2:1 (fine: coarse: fine). Here fine kenaf particle was used in face and back portion and in the core portion jute stick and coir fiber mixture was used. The board were named as control which is binder less i.e. no citric acid (0%) is used. On the other hand 10% citric acid content is denoted as C-10 and rest are denoted as C-15 (15% citric acid), C-20 (20% citric acid), C-25 (25% citric acid), C-30 (30% citric acid) respectively.

4.1.1 Effect of Citric acid on different Physical properties

4.1.1.1 Density

The relationship between board densities with different citric acid content was observed under a press temperature at 180°C. Here six boards were analyzed statistically and significant differences were not found among them. ($\alpha = 0.05$, $df = 5$, $p > 0.05$; See Appendix, Data table – 1, page no- 43) because board density were around 0.78 gm/cm³. Graphical presentation is given here-

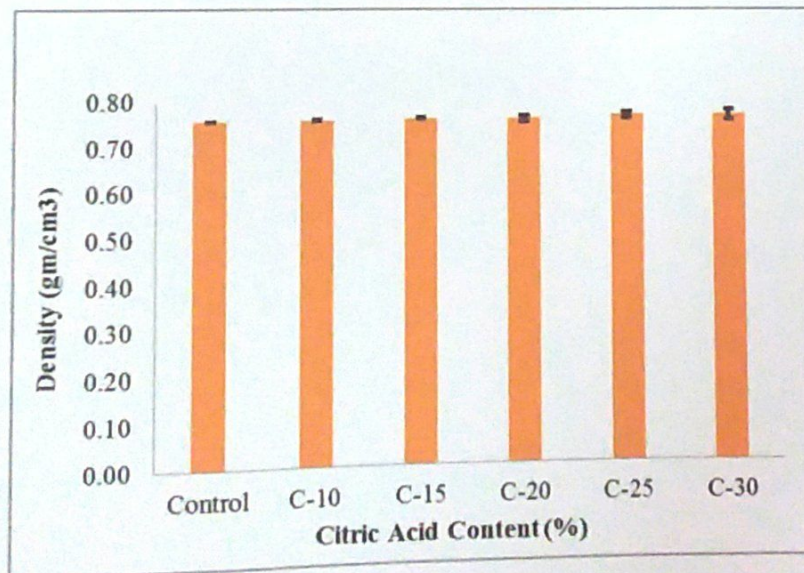


Figure 4.1: Density of particleboard at different citric acid content

4.1.1.2 Moisture content

To determine the moisture content all boards were kept at 103°C for 24 hours and their weight were taken as green and dry condition. By using formula their moisture content were determined and analyzed statistically. Significant differences were observed among the data which is as ($\alpha = 0.05$, $df = 5$, $p < 0.05$; See Appendix, Data table – 2, page no-44). Further HSD (Least Significant Difference) was done to find out the significant treatment by lettering. In this case significant treatment was C-30 in which moisture content found 8.04 that satisfied the ANSI standard. Figure-4.2 represents the graphical presentation of the treatment of moisture content.

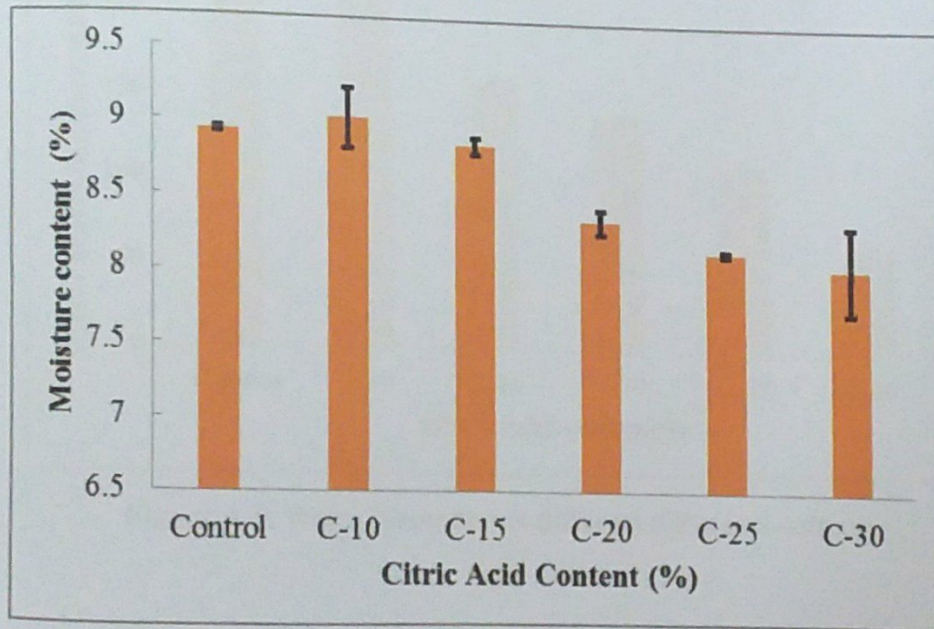


Figure 4.2: Moisture content of citric acid bonded board.

4.1.1.3 Water absorption

Prepared boards were soaked under cold water for 24 hours for this test. Obtained data were analyzed statistically as ($\alpha = 0.05$, $df = 5$, $p < 0.05$; See Appendix, Data table – 3, page no-45) and significant differences were observed among the data and further HSD was done to determine the highest treatment value. Significant (A) differences were observed in control treatment i.e. binderless board and less value was observed in citric acid concentration where C-30 showed the lowest value- 63.37% in comparison with the other but it did not satisfied the standard. Following figure (4.3) represents the data

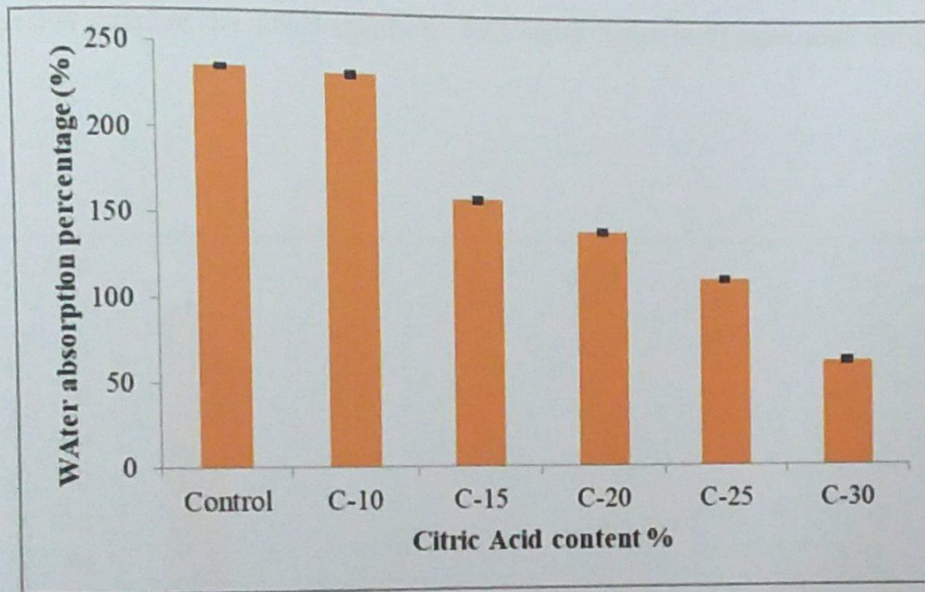


Figure 4.3: Water absorption at different citric acid content

4.1.1.4 Thickness swelling (TS)

This test indicates how the boards will behave when used under conditions of severe humidity and are especially important regarding boards that are to be used externally (Mancera *et al.*, 2011). Significant differences were observed among the data which is as ($\alpha = 0.05$, $df = 5$, $p < 0.01$; See Appendix, Data table – 4, page no- 46). Further HSD (Least Significant Difference) was done to find out the significant treatment by lettering. In control treatment highest value was found than the other value because no binding agent was used here than the other one. But in citric acid treatment this value was less than the control one and more or less same to each other and in C-30 treatment the least value (11.90) was observed that satisfied the ANSI standard. Following figure (4.4) represents the data -

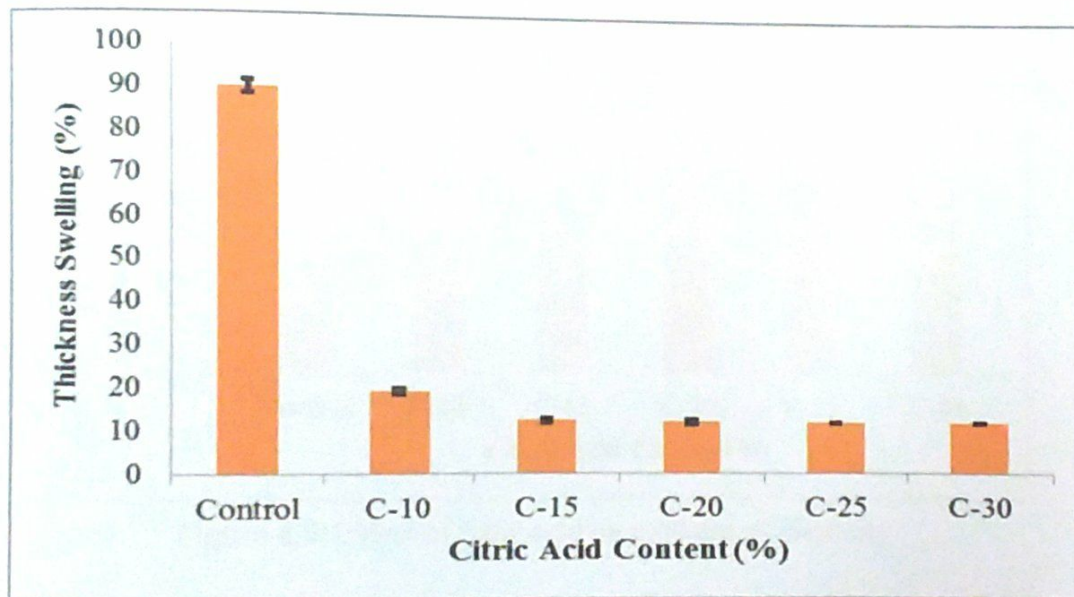


Figure 4.4: Thickness swelling at different citric acid content

4.1.2 Effect of Citric acid on different Mechanical properties

4.1.2.1 Modulus of elasticity (MOE)

Modulus of elasticity of the boards were tested and analyzed statistically as ($\alpha = 0.05$, $df = 5$, $p < 0.05$; See Appendix, Data table – 5, page no-47) and significant differences were found. Further HSD was done to find out the best treatment. Among the treatments citric acid bonded C-30 board showed satisfactory result. Following figure – 4.5 presents the statistical data. Here, 30% citric acid content showed the satisfactory MOE value (4519.67 Mpa, Specific MOE 5249.33 Mpa) and satisfied ANSI standard.

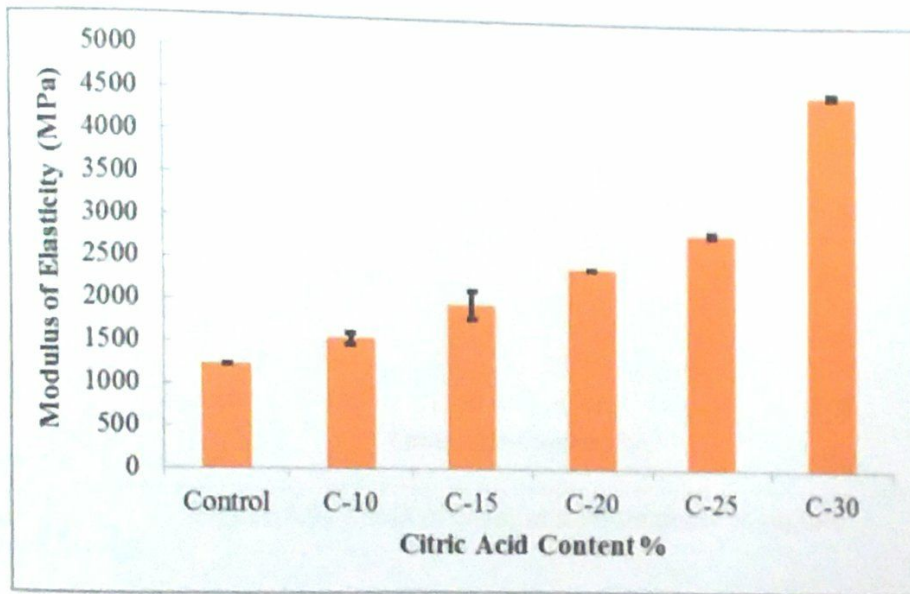


Figure 4.5: Effect of citric acid on modulus of elasticity

4.1.2.2 Modulus of rupture (MOR)

Modulus of rupture of the boards were tested and analyzed statistically as ($\alpha = 0.05$, $df = 5$, $p < 0.05$; See Appendix, Data table – 6, page no-48) and significant differences were found. Further HSD was done to find out the best treatment. Among the treatments citric acid bonded C-30 board showed satisfactory result. Following figure – 4.6 presents the statistical data. Here, 30% citric acid content showed the satisfactory MOR value (13.84 Mpa, Specific MOR 16.07 Mpa) and satisfied ANSI standard.

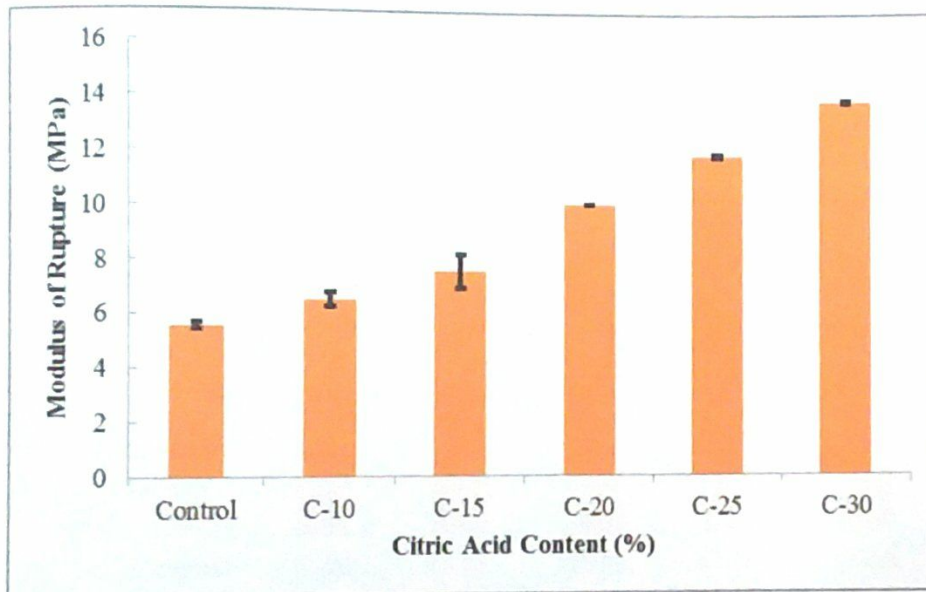


Figure 4.6: Effect of citric acid on modulus of rupture

4.1.2.3 Internal Bonding Strength (IB)

The IB value of the boards increased with increasing citric acid content. Comparing with binderless board it was higher and citric acid bonded board met the requirement of ANSI standard and which value was much higher than the standard (0.40 MPa). Treatment value were analyzed statistically as ($\alpha = 0.05$, $df = 5$, $p < 0.05$; See Appendix, Data table – 7, page no- 49) and significant differences were found. Further HSD was done to find out the best treatment. Among the treatments citric acid bonded C-30 board showed satisfactory result. Here, 30% citric acid content showed the satisfactory IB value (0.68 Mpa) and satisfied ANSI, standard. On the other hand binderless one did not satisfy any standard. Following figure – 4.7 presents the statistical data.

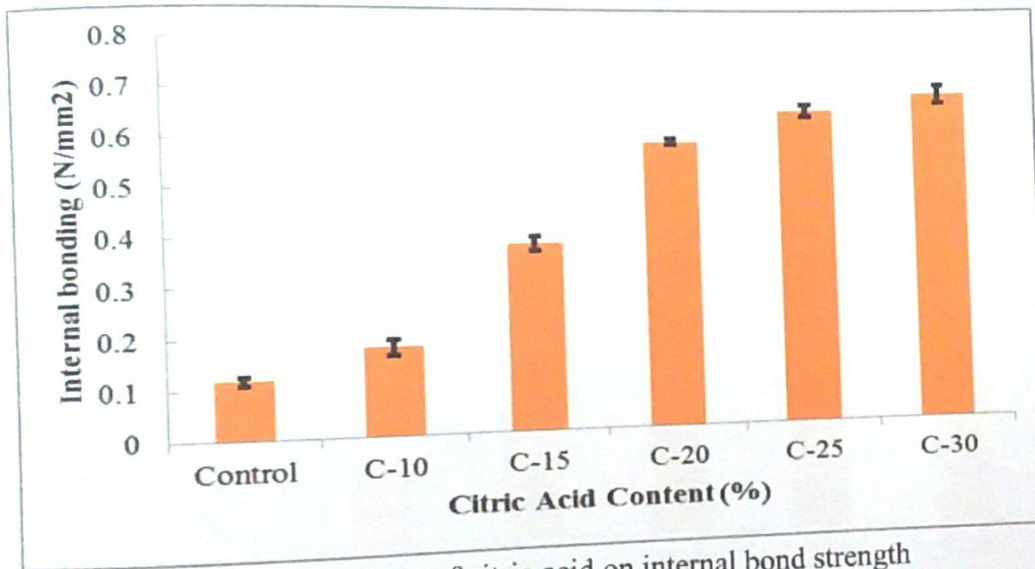


Figure 4.7: Effect of citric acid on internal bond strength

4.1.2.4 Surface Soundness (SS)

Strength or quality of bonding between the particles or fibres at the surface of a board and the layer below (unfaced panels) or between the coating material and the underlying board (overlaid panels). Measurement of the tensile load required to pull off a defined surface area of overlaid or unfaced panel. Treatment value were analyzed statistically as ($\alpha = 0.05$, $df = 5$, $p < 0.05$; See Appendix, Data table – 8, page no- 50) and significant differences were found. Further HSD was done to find out the best treatment. Among the treatments citric acid bonded C-30 board showed satisfactory result and satisfied the ANSI standard. On the other hand binderless board showed very poor value. Following figure – 4.8 presents the statistical data.

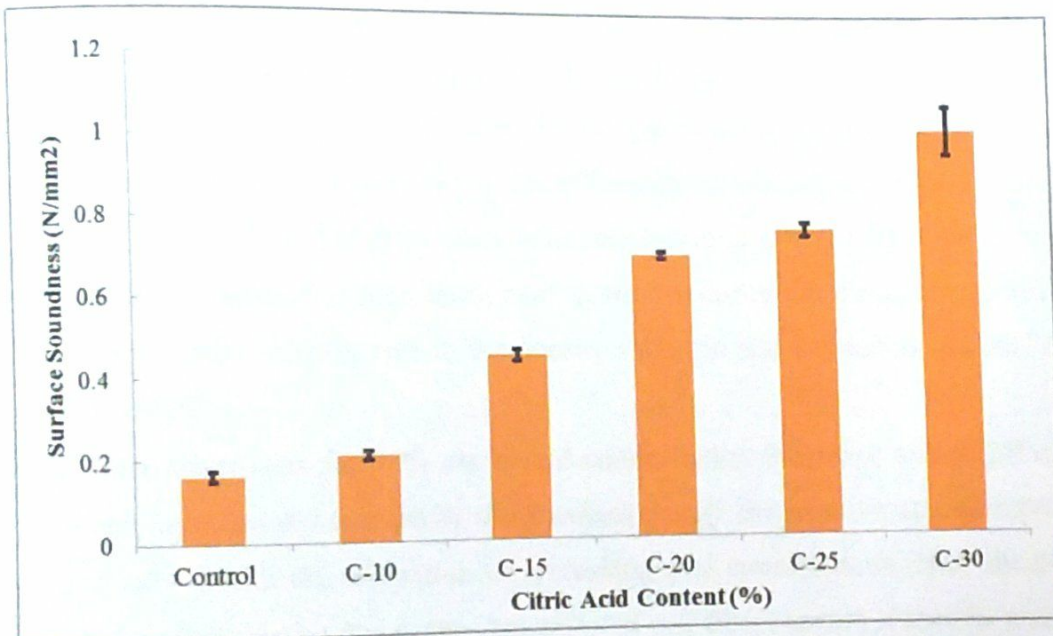


Figure 4.8: Effect of citric acid on internal bond strength

4.2 Discussion

In this study density of the boards were ranges from 0.61-0.86 gm/cm³. The variation of densities was due to mat thickness and pressing time variation and it was done intentionally to observe different physical and mechanical properties of board at different densities. Different citric acid concentration, temperature and pressure may affect the board density. Bending properties of the board are greatly influenced by density. The variation of density between particleboard was due to the variation of the raw materials itself. It depends on the density of raw materials used, hot pressing conditions and other factors (Hsu *et al.*, 1988; Sekino, 1999; Velasquez *et al.*, 2009). Arias (2008) emphasized four factors that are significantly important for the density and these factors are pretreatment temperature, pretreatment time, pressing temperature and initial pressing pressure. In optimum citric acid concentration it was found highest density 0.8 gm/cm³. It may depend on the proper distribution of lignin between the particles during pressing process. To allow a good distribution of lignin between the particles during the pressing process, it is necessary to apply enough heat and pressure to melt the lignin through the whole board (Arias, 2008).

In case of moisture content, for 30% citric acid concentration it was 8.04% which was very less than the other value. So, increasing acid content reduces the moisture content value. Without it this variation may be due to the density variation and its binding material content and spraying condition.

In case of water absorption for 30% citric acid concentration the value was 63.37%, which was very much high and did not satisfy the standard though the rate of water absorption was reduced gradually due to the utilization of increasing acid concentration. Here the probable cause of the variation may be due to the utilization of coir fiber because it absorbs more water than the other particle used in this board. In control treatment i.e in binderless board no citric acid was used so particle compaction was less in comparison with citric acid bonded board and it soaks more water than the rest one. Water absorption decreased with increasing board density indicating that the penetration of water into the board was prevented by higher density. Low-density board had high water absorption compared to medium density particle board (Widyorini *et al.*, 2005). Widyorini *et al.* (2015) showed water absorption of different bamboo species bonded with citric acid was comparatively lower. It was shown that the water absorption of particleboards bonded using citric acid was significantly decreased with increasing resin content. The WA values were 98, 70, and 85 % for petung, wulung, and apus bamboo binderless particleboards, and decreased to 35, 17, and 26 %, respectively, after only 15 wt. % addition of citric acid. But kenaf particle absorb high amount of water. A study

from Paridah *et al.* (2007), showed kenaf particleboard when bonded with formaldehyde-based adhesives absorb high amount of water. Light and highly porous kenaf could be a reason of high amount of water absorption. The highly porous structure of kenaf particle allowed water to penetrate through and to result in high water uptake hence causing thickness swelling at the same time. (Akil H. L. *et al.*, 2011).

In case of thickness swelling (TS) it was observed that for 30% citric acid concentration the value was 11.90% and the values were gradually decreased due to increasing acid content gradually. According to Moslemi (1974) less swelling is also expected when wood species with high density are used due to the lower degree of mat compaction. Considering the high acid content and the low molecular weights of citric acid, the reason the TS remained constant irrespective of the density (Kenji Umemura *et al.* 2014). A study from Widyorini *et al.* (2015) showed thickness swelling of citric acid bonded bamboo particleboard was very low compare to the binderless particleboard. The same trend was also found by Umemura *et al.* (2011, 2012a, b) that the molding bonded with citric acid had high water resistance. More adhesive added to particle means that greater area of particles is covered with adhesive. If greater area is covered by adhesive that also means that particles are more bonded together, hence less water can penetrate between particles and also less chance to regain its (particles) original shape.

In this study it was found that for optimum citric acid concentration (30%) the Modulus of elasticity (MOE) value was 4519.67 N/mm² (Specific MOE 5249.33 N/mm²) higher than the other one. Here citric acid works as a binding agent and hold the particles together with strong bond, for this reason citric acid bonded boards showed good mechanical properties like MOE and MOR. A study on kenaf particleboard bonded with formaldehyde based adhesive of Akil H.L.*et al.*, (2011) suggests that the mechanical properties of citric acid bonded particleboard at 30% concentration exhibits good MOE and MOR value. On the other hand binderless composite board showed very poor value in comparison with citric acid bonded board. In this case probable reason may be the utilization of different raw material like kenaf, jute stick and coir fiber; their inherent chemical properties and their nature of compaction under heat and pressure may influence the value. Comparing binderless with citric acid bonded compositeboard indicates binderless layered [Face (Kenaf): Core (Jute stick + Coir fiber): Back (Kenaf)] particle developed poor bonding. Widyorini *et al.* (2015) showed the MOR values of binderless particleboard (0 wt.%) made from petung, wulung and apus bamboos were very low, i.e. 1.9, 3.3, and 2.9 MPa, respectively, indicating that the bamboo particles developed almost no bonding strength under this condition. However, the

value drastically increased in the range of 4–6 times as citric acid content increased to 15 wt.%. The main mechanism of bonding in citric acid application was hydroxyl groups of cellulose react with carboxyl groups from citric acid and forming ester linked groups, supports superior bonding (Umemura *et al.*, 2012a and Widyorini *et al.* 2014). For this reason citric acid bonded board showed satisfactory result than the other.

In this study it was observed that increasing citric acid concentration causes increased Internal Bonding Strength (IB). For this reason in optimum citric acid concentration it had showed 0.68 Mpa IB strength. So, this result suggests that the mechanical properties of the boards bonded with citric acid were greatly affected by the resin content. A study of Widyorini *et al.* (2015) also suggest that the IB value of bamboo particleboard increased with increasing citric acid, and a range value of 0.34–0.40 MPa was recorded in the resin content of 15 wt.% and these values were eight times higher than that of binderless board. The adhesion mechanism was hydroxyl groups of cellulose react with carboxyl groups from citric acid and forming ester linked groups, supports superior bonding (Umemura *et al.*, 2012a and Widyorini *et al.* 2014). Generally, it is known that IB strength is influenced by the adhesiveness of the inner layer of the board, and the bending properties are affected by the adhesiveness of the surface layer of the board (Umemura *et al.* 2014)

Here, Surface Soundness (SS) value was found higher due to the application of increasing citric acid concentration. For this reason in this study highest result was observed for 30% citric acid concentration and that was 1.0 Mpa. Probable reason in this case may be the utilization of citric acid and its adhesiveness. Increasing acid content increases adhesiveness and causes increased MOE and MOR value. Surface soundness increases with increasing MOE and MOR value (Widyorini *et al.* 2014).

CHAPTER FIVE: CONCLUSION

Conclusion

In this study citric acid bonded board has been developed successfully. The effects of citric acid concentration on the physical and mechanical properties of particleboard were assessed. When citric acid was added with raw material (Kenaf, Jute and coir) for particle board production then it could significantly improve the dimensional stability and mechanical properties of the boards. All the properties of the board satisfied the ANSI standard except water absorption. So, utilization of citric acid as a natural binder has a great potentiality as environment-friendly binder in the field of particleboard manufacturing in near future.

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Density

Data Table - 1

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	0.75	0.76	0.76	0.74	0.74	0.75
Replication 2	0.76	0.76	0.76	0.76	0.75	0.77
Replication 3	0.76	0.76	0.76	0.76	0.78	0.78
Replication 4	0.76	0.76	0.76	0.77	0.79	0.80

SUMMARY - 1

Groups	Count	Sum	Average	Variance
Control	4	3.022	0.7555	1.7E-06
C-10	4	3.026	0.7565	1.7E-06
C-15	4	3.037	0.75925	1.1E-05
C-20	4	3.029	0.75725	0.00016
C-25	4	3.06	0.765	0.00057
C-30	4	3.1	0.775	0.00043

ANOVA - 1

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	0.00111	5	0.000223	1.14177	0.37468	2.77285315
Within Groups	0.00351	18	0.000195			
Total	0.00463	23				

Moisture content

Data Table - 2

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	8.9	8.55	8.7	8.14	8.1	7.5
Replication 2	8.91	8.98	8.85	8.32	8.14	7.55
Replication 3	8.95	9.05	8.92	8.4	8.16	8.42
Replication 4	8.98	9.55	8.96	8.55	8.18	8.69

DATA SUMMARY - 2

Groups	Count	Sum	Average	Variance
Control	4	35.74	8.935	0.001367
C-10	4	36.13	9.0325	0.167892
C-15	4	35.43	8.8575	0.013092
C-20	4	33.41	8.3525	0.029158
C-25	4	32.58	8.145	0.001167
C-30	4	32.16	8.04	0.3662

ANOVA - 2

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	3.75227	5	0.750454	7.778406	0.00047	2.77285
Within Groups	1.73663	18	0.096479			
Total	5.4889	23				

Table for Lettering - 2

Treatment	Average	No. of Replication	Letter
C-10	9.0325	4	A
Control	8.935	4	A
C-15	8.8575	4	A
C-20	8.3525	4	AB
C-25	8.145	4	B
C-30	8.04	4	B

Water Absorption

Data Table – 3

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	234.35	229.85	157.4	138.95	110.58	60.87
Replication 2	235.01	231.25	158.52	139.7	111.88	62.82
Replication 3	235.20	232.45	160.20	140.82	112.63	63.92
Replication 4	236.35	234.55	161.58	142.55	114.44	65.88

DATA SUMMARY- 3

Groups	Count	Sum	Average	Variance
Control	4	940.91	235.2275	0.692691667
C-10	4	929.2	232.3	7.583333333
C-15	4	637.7	159.425	3.3881
C-20	4	562.02	140.505	2.4491
C-25	4	450.53	112.6325	4.220358333
C-30	4	253.49	63.3725	4.385025

ANOVA - 3

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	91207.29894	5	18241.45979	4817.582007	1.7784E-27	2.772853
Within Groups	68.155825	18	3.786434722			
Total	91275.45476	23				

Table for Lettering – 3

Treatment	Average	No. of Replication	Letter
Control	235.2275	4	A
C-10	232.3	4	B
C-15	159.425	4	C
C-20	140.505	4	D
C-25	112.6325	4	E
C-30	63.3725	4	F

Thickness swelling

Data Table – 4

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	85.88	18.05	11.54	11.15	11.8	11.76
Replication 2	89.99	19.15	12.2	12.18	12.04	11.87
Replication 3	91.88	19.49	12.88	12.51	12.1	11.97
Replication 4	92.63	20.28	13.44	12.8	12.13	12.01

DATA SUMMARY - 4

Groups	Count	Sum	Average	Variance
Control	4	360.38	90.095	9.1299
C-10	4	76.97	19.2425	0.85609167
C-15	4	50.06	12.515	0.67956667
C-20	4	48.64	12.16	0.51753333
C-25	4	48.07	12.0175	0.022425
C-30	4	47.61	11.9025	0.01249167

ANOVA - 4

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	19683.4	5	3936.681	2105.55067	3.026E-24	2.772853
Within Groups	33.65403	18	1.869668			
Total	19717.06	23				

Table for Lettering - 4

Treatment	Average	No. of Replication	Letter
Control	90.095	4	A
C-10	19.2425	4	B
C-15	12.515	4	C
C-20	12.16	4	C
C-25	12.0175	4	C
C-30	11.9025	4	C

Modulus of elasticity (MOE)

Data Table – 5

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	1222.12	1420.27	1565.27	2396.9	2797.57	4518.15
Replication 2	1231.08	1458.27	1966.3	2400.96	2799.36	4522.1
Replication 3	1244.56	1622.89	1966.31	2402.95	2853.48	4456.17
Replication 4	1248.32	1691.75	2367.36	2403.9	2896.67	4582.26

DATA SUMMARY - 5

Groups	Count	Sum	Average	Variance
Control	4	4946.08	1236.52	146.945067
C-10	4	6193.18	1548.295	16879.5508
C-15	4	7865.24	1966.31	107224.728
C-20	4	9604.71	2401.1775	9.63269167
C-25	4	11347.08	2836.77	2267.79407
C-30	4	18078.68	4519.67	2652.6578

ANOVA - 5

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	27795895	5	5559178.91	258.203558	4.25556E-16	2.77285315
Within Groups	387543.9	18	21530.2181			
Total	28183438	23				

Table for Lettering - 5

Treatment	Average	No. of Replication	Letter
C-30	4519.67	4	A
C-25	2836.77	4	B
C-20	2401.1775	4	C
C-15	1966.31	4	D
C-10	1548.295	4	E
Control	1236.52	4	E

Modulus of rupture (MOR)

Data Table -6

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	5.25	5.86	6.06	10	11.75	13.73
Replication 2	5.57	6.35	7.5	10.01	11.8	13.84
Replication 3	5.63	6.89	7.6	10.03	11.89	13.85
Replication 4	5.78	6.99	9.04	10.04	11.96	13.94

DATA SUMMARY - 6

Groups	Count	Sum	Average	Variance
Control	4	22.23	5.5575	0.049825
C-10	4	26.09	6.5225	0.27409167
C-15	4	30.2	7.55	1.48173333
C-20	4	40.08	10.02	0.00033333
C-25	4	47.4	11.85	0.00873333
C-30	4	55.36	13.84	0.0074

ANOVA - 6

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	209.52	5	41.90443667	137.986016	1.0582E-13	2.77285315
Within Groups	5.4664	18	0.303686111			
Total	214.99	23				

Table for Lettering - 6

Treatment	Average	No. of Replication	Letter
C-30	13.84	4	A
C-25	11.85	4	B
C-20	10.02	4	C
C-15	7.55	4	D
C-10	6.5225	4	DE
Control	5.5575	4	E

Internal bonding

Data Table -7

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	0.1	0.14	0.34	0.57	0.62	0.64
Replication 2	0.11	0.17	0.38	0.58	0.64	0.66
Replication 3	0.12	0.18	0.39	0.59	0.65	0.7
Replication 4	0.14	0.22	0.41	0.6	0.68	0.72

DATA SUMMARY - 7

Groups	Count	Sum	Average	Variance
Control	4	0.47	0.1175	0.000291667
C-10	4	0.71	0.1775	0.001091667
C-15	4	1.52	0.38	0.000866667
C-20	4	2.34	0.585	0.000166667
C-25	4	2.59	0.6475	0.000625
C-30	4	2.72	0.68	0.001333333

ANOVA- 7

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.1909375	5	0.2381875	326.6571429	5.2815E-17	2.77285315
Within Groups	0.013125	18	0.00072916	7		3
Total	1.2040625	23				

Table for Lettering - 7

Treatment	Average	No. of Replication	Letter
Control	0.1175	4	D
C-10	0.1775	4	D
C-15	0.38	4	C
C-20	0.585	4	B
C-25	0.6475	4	A
C-30	0.68	4	A

Surface Soundness

Data Table -8

Treatment	Control	C-10	C-15	C-20	C-25	C-30
Replication 1	0.13	0.18	0.42	0.68	0.73	0.98
Replication 2	0.15	0.21	0.45	0.7	0.75	0.99
Replication 3	0.18	0.22	0.47	0.71	0.78	1
Replication 4	0.19	0.23	0.48	0.72	0.8	1.09

DATA SUMMARY - 8

Groups	Count	Sum	Average	Variance
Control	4	0.65	0.1625	0.00075833
C-10	4	0.84	0.21	0.00046667
C-15	4	1.82	0.455	0.0007
C-20	4	2.81	0.7025	0.00029167
C-25	4	3.06	0.765	0.00096667
C-30	4	3.52	1.015	0.0152

ANOVA - 8

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.802233333	5	0.360446667	117.643518	4.249E-13	2.7728532
Within Groups	0.05515	18	0.003063889			
Total	1.857383333	23				

Table for Lettering - 8

Treatment	Average	No. of Replication	Letter
Control	0.1625	4	A
C-10	0.21	4	B
C-15	0.455	4	B
C-20	0.7025	4	B
C-25	0.765	4	B
C-30	1.015	4	B