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MANUFACTURING OF CITRIC ACID BONDED PARTICLE BOARD WITH VENNA (*RICINUS COMMUNIS*).

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DECLARATION

I am, Azad Hossain, declare that this thesis is the result of my own works and it has not been submitted or accepted for a degree in any other university. I also declare that this thesis or any information of this paper can not be used industrially or commercially without any prior permition of the author.

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Azad Hossain

Dedicated
To
My Beloved Parents

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July, 2018

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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 BSc. Degree in Forestry. I have approved the style and format of the project thesis.

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2.6 Chemicals

ABBREVIATION

Anon	Anonymous
ANOVA	Analysis of Variance
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

This study investigated the potentiality of citric acid and fibrous raw material for the production of particle board and its basic physical and mechanical properties. It appears that manufacturing of citric acid bonded particle board by using *Ricinus communis* particle is technically feasible for various structural purposes. The physical and mechanical properties were as:Density (0.8gm/cm^3); Moisture content (8.04%); water absorption (110.13%); thickness swelling (11.90%);Modulus of Rupture (MOR, 16.07 N/mm^2) and Modulus of Elasticity (MOE, 5249.33 N/mm^2).

The physical and the mechanical properties of 8 mm board were compared with different boards of various ratio . The study shows the using citric acid in 10%, 20% ,30% ,40%, and 50%.Among them using 30% citric acid showing the best result of physical and mechanical properties and 50% shows the lowest quality particle board.It has been observed that all the properties of the boards meet the requirements of the international standards like ANS, JIS, ANZS, andIS. Based on the experiment it has found that this board shows quite satisfactory results in case of physical and mechanical properties except thickness swelling and water absorptions.

CHAPTER ONE: INTRODUCTION

1.1 BACKGROUND

At present industries are now very much interested to produce eco-friendly product. This product will be economically viable, environment friendly and does have 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 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 any density composite board manufactured from venna tree .

Venna (*Ricinus Communis*) is a well-known fast growing fiber plant native to the Indian subcontinent and adjacent regions of Southeast Asia . Being an exotic species in our country venna 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. Venna 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. Venna is very much popular in our country. At present particle board is manufactured from low density wood like as venna tree. 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 venna tree (*Ricinus Communis*) 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 any density composite board.

1.2 Objectives of the study:

- ✓ To carry out comparative study of the quality of citric acid bonded composite board and its feasibility to use.
- ✓ To introduce a natural adhesive (citric acid) for manufacturing particleboard.

- ✓ To observe the optimum temperature and time for bonding the board with venna stem particles at different mixture ratio.
- ✓ To find out the physical and mechanical test value of these board bonded by citric acid.

CHAPTER TWO: LITERATURE REVIEW

2. General information about Compositeboard

2.1. Definition of Compositeboard

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. (Anon, 1970). 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 Composite board

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 Composite board

- 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 particle board is that it can be made in large dimensions (Salehuddin, 1992).

2.4 Considerations for the quality of Composite board

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 Composite board 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) Venna

c) Bagasse

d) Bamboo

e) Flax shaves

f) Cotton stalks

g) Cereal straw

h) Kenaf

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., 2004).

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, 1968). 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 (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 $C6H8O7$. 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 *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, Robert N, et al. 2002). 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 and it is also used as an odorless alternative to white vinegar for home dyeing with acid dyes.

- ❖ Citric acid is an alpha hydroxy acid and used as an active ingredient in chemical peels.

- 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 venna

venna (Ricinus communis L.) is a fiber plant native to the southeastern Mediterranean Basin,Eastern Africa ,and india ,but is widespread throughout tropical regions(and widely grown elsewhere as an ornamental plant). Scientific Classification:

Kingdom: Plantae
  (Unranked): Angiosperms
    (Unranked): Eudicots
      (Unranked): Rosids
        Order: Malpighiales
          Family: Euphorbiaceae
            Genus: Ricinus
              Species: R. communis
                Bionomial Name: Ricinus communis L (Sources: Wikipedia)

Figure 2.1: Image of Venna plant

2.8.1 Structure and Chemical composition of venna

Yield component research with five venna cultivars showed averaged 26% leaves and 74% stalks by weight (Webber 1993b). In the same research the venna stalk's average composition was 35% bark and 65% woody core by weight. The bark of the venna stalk contains the long fiber strands that are composed of many individual smaller fibers. These individual fibers, held together by lignin, are the building blocks of the bark fiber strands, which historically were used to make the cordage products. The woody core material of the stalk, the portion remaining when the bark is removed, contains core fibers. Oil in seed and crude protein in venna leaves and stalks are also present.

2.8.2 Uses of Venna:

It has been a source of textile fiber for such products as rope, twine, bagging and rugs. Venna is a promising source of raw material fiber for pulp, paper, particleboard and other fiber products. The common name "castor oil" probably comes from its use as a replacement for castoreum , a perfume base made from the dried perineal glands of the beaver (castor in Latin).It has another common name **palm of Christ** ,or palma Christi ,that derives from castor oils .Castor oil has many uses in medicine and other applications.An alcoholic extract of the leaf was shown ,in lab rats,to protect the liver from damage from certain poisons .Methanoic extracts of the leaves of Ricinus communis were used in antiomicrobial testing against eight pathogenic bacteria in rats and showed antimicrobial properties .It is also used extensively as a decorative plant in parks and other public areas, particularly as a "dot plant" in traditional bedding schemes.Castor oils is an effective motor lubricant and has been used in internal combustion enginees ,including those of World War 1 airplanes , some racing cars and other model airplanes .It has historically been popular for lubricating two-stroke engines due to high resistance to heat compared to petroleum based oils.

2.9Literature survey regarding the natural adhesive

Widyorini 2013 showed the development bio-based composites made from non-woody 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. 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 non-woody like as venna 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 non woody(venna) 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 5 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 non-woody materials. Their study investigated the physical and mechanical properties of particleboard made from non-woody (venna) using citric acid as natural binder. Citric acid contents were set at 10 , 20 and 30 wt. % based on air-dried particles. The properties of citric acid-bonded venna particleboard in this research could meet the requirements of the Japanese Industrial Standard for particleboard . 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 density fiberboard (DF) which was termite resistance, manufactured from agricultural fiber bonded with citric acid. In this study, citric acid application as natural adhesive was investigated for density fiberboard (DF) manufacture from pineapple leaf fiber. Using the ratio of citric acid 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 DF 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.

In this study are raw materials selected considering its production pattern and its chemical properties in order to show its feasibility to be used industrially and eco-friendly.

Chapter Three: Materials and Methods

3.1 Materials and Equipment

3.1.1 Materials:

Venna was used as raw material for composite board manufacturing and citric acid was used as an adhesive. After screening the grinded particles were separated as coarse and fine. All of the particles were then air-dried to moisture content around 12 %. Citric acid (anhydrous) was used without further purification.

3.1.2 Hot press

A digital hydraulic hot press was used to press the mat into particleboard. It has multi- layer plate . The both platens were movable up and down. Maximum temperature range within 400°C and pressure up to 5MPa commonly occur.

3.1.3 Universal Testing Machine (UTM)

Universal Testing Machine (UTM) was used to determine the mechanical properties of the particleboards.

3.1.4 Oven

A lab scale ventilated oven was used to determine the moisture content (%) of raw materials as well as the particle boards. A digital indicator outside the oven indicated the inside temperature.

3.1.5 Electric balance

A digital balance was used to measure the weight of the raw materials as well as particleboards and also used to measure the weight of different ingredients of the adhesive.

3.2 Manufacturing of particleboard

3.2.1Collection of Raw Materials

Venna was collected from Khulna University campus.

3.2.2 Processing & Screening of Raw Materials

Firstly venna 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 venna particle.

3.2.3 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 solution was used as an adhesive and sprayed over the particle. The particles were blended manually.

Figure 2: Solution preparation

Citric acid contents were set at 10 ,20 ,30 ,40 and 50% based on air dried particles. The sprayed particles were then oven dried for 24 hours at 80°c to reduce the moisture. The moisture content of the oven dried particle was 12-19%.

3.2.4 Mat Formation

After drying the particles the mat was formed by using a wooden forming box, followed by hot pressing to make the board. Single layered board, mixed board and layered board were prepared using different citric acid concentration as treatment like 10, 20, 30, 40, and 50%. The average mat thickness was 50 mm. Compositions of the layered board was 1:2:1

(fine:coarse:fine). The dimension of the prepared mat was 300 mm in length, 200 mm in width.

Figure 3: Mat formation

3.2.5 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%,40%,50% citric acid used), 10 minutes (when 10%, 20%, 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.

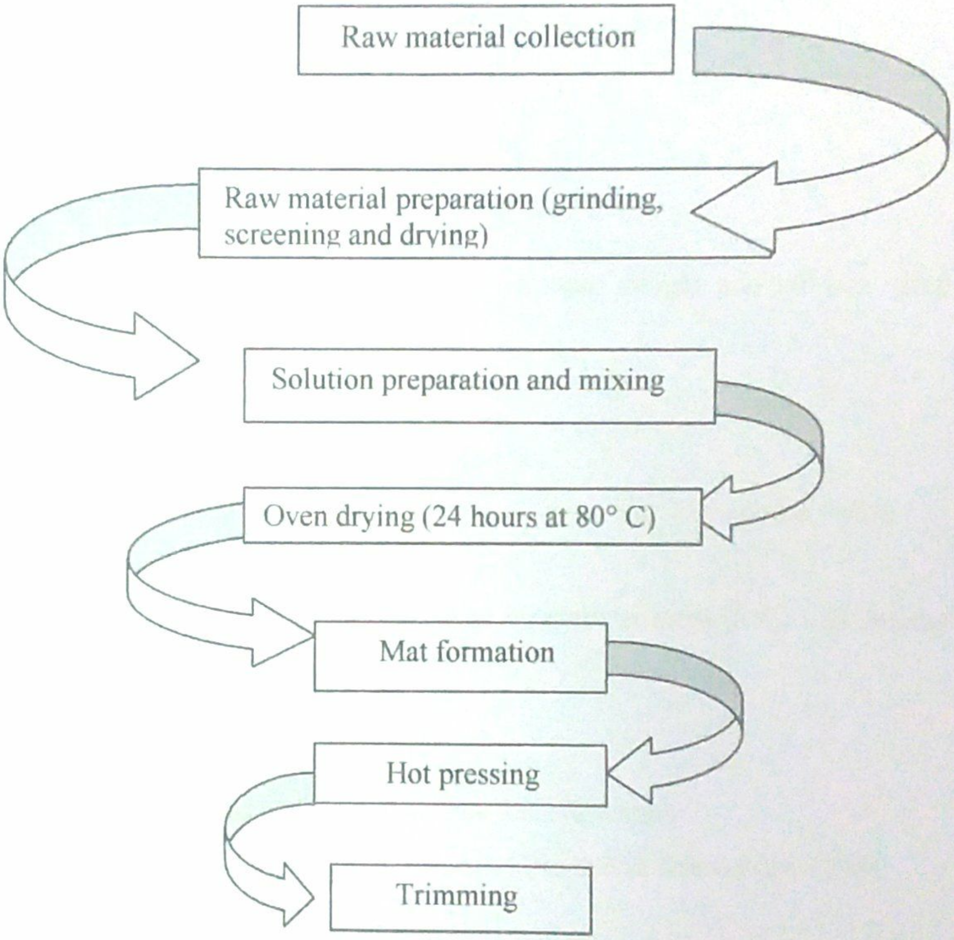
Figure 4: Hot pressing & board preparation

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

3.2.6 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).

3.3 Flow Diagram Particle Board Preparation



3.4 Manufacturing place & Laboratory Test

The particle 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 there, and MOE, MOR were tested from our Bio–nano technology Lab. The properties were tested according to the procedures defined in the Japanese standard for particleboards.

3.5 Evaluation of Board properties

The properties of the particleboard were evaluated according to the Japanese industrial standard .Mechanical tests like modulus of elasticity (MOE), modulus of rupture (MOR) and physical tests like water absorption (WA), thickness swelling(TS) 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, and thickness swelling were determined after 24 hours of soaking under water. The dimension of samples for testing the physical properties strength was approximately 50 mm x 50 mm x 8 mm.

3.5.1 Physical Properties

3.5.1.1 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.

(Desch and Dinwoodie, 1996)

3.5.1.2 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.

(Desch and Dinwoodie, 1996)

3.5.1.3 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-

$$A_w(\%) = \frac{m_2 - m_1}{m_1} \times 100$$

(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.

3.5.1.4 Thickness Swelling

Thickness Swelling was determined by using the following equation-

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

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

3.5.2Mechanical Properties

3.5.2.1 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-

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

$$MOR = \frac{3PL}{2bd^2} \qquad \text{(Desch and Dinwoodie, 1996)}$$

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).

3.6 Experimental Design

For data analysis 3 treatments of different citric concentration (10%, 20% and 30%) and 4 replications of each treatment were taken and CRD (Completely Randomized Block Design) was applied.

3.7 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 SAS (Statistical Analysis Difference) statistical software, Microsoft office excels.

Chapter Four: Results & Discussions

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 venna particle was used in face and back portion and in the core portion and fine mixture was used at 50:50 percentages.On the other hand 10% citric acid content is denoted as C-10 and rest are denoted as C-20 (20% citric acid), C-30 (30% citric acid), C-40 (40% citric acid), C-50 (50% citric acid) respectively.

4.1.1 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 three boards were analyzed statistically and significant differences were found among them. ($\alpha = 0.05$, $df = 5$, $p < 0.01$; See Apendix, Data table – 1, page no- 33). Due to significant differences , it was observed that C-30 (30% citric acid) treatment hold the highest value . Graphical presentation is given here-

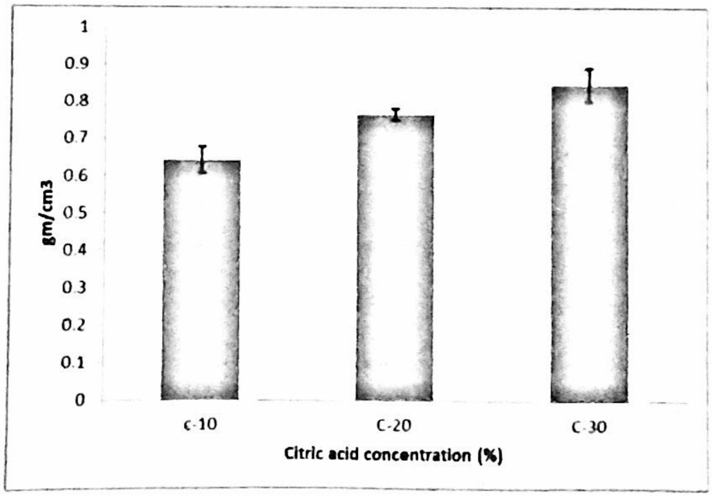


Figure 4.1: Density of particleboard at different citric acid content

Discussion

In this study density of the boards were ranges from 0.63-0.86 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 MOR and MOE increased linearly with increasing densities. Physical properties also affected by density variation. So, it is an important parameter and it virtually affects all the properties of board. 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; Volasqueze *et al.*, 2003). 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. Density also 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).

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 ($\alpha = 0.05$, $df = 5$, $p < 0.01$; See Apendix, Data table – 2, page no: 33-34). In this case significant treatments were as C-30. Figure-4.2 represents the graphical presentation of the treatment of moisture content.

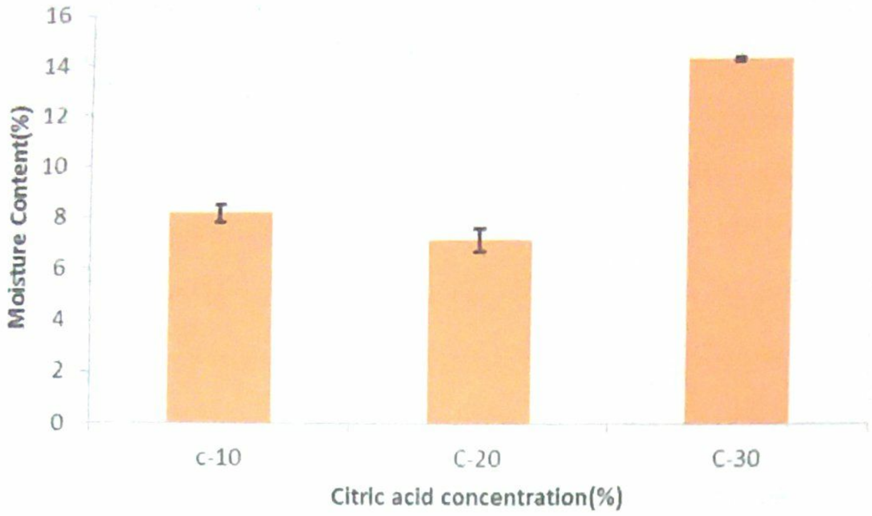


Figure 4.2: Moisture content of citric acid bonded board.

Discussion:

This variation may be due to the density variation and its binding material content and spraying condition. Among the three variations i.e. C-30 density was higher than the rest two for this reason.

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 ($\alpha = 0.05$, $df = 5$, $p < 0.01$; See Apendix, Data table – 3, page no-34) and significant differences were observed among the data 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. Following figure (4.3) represents the data

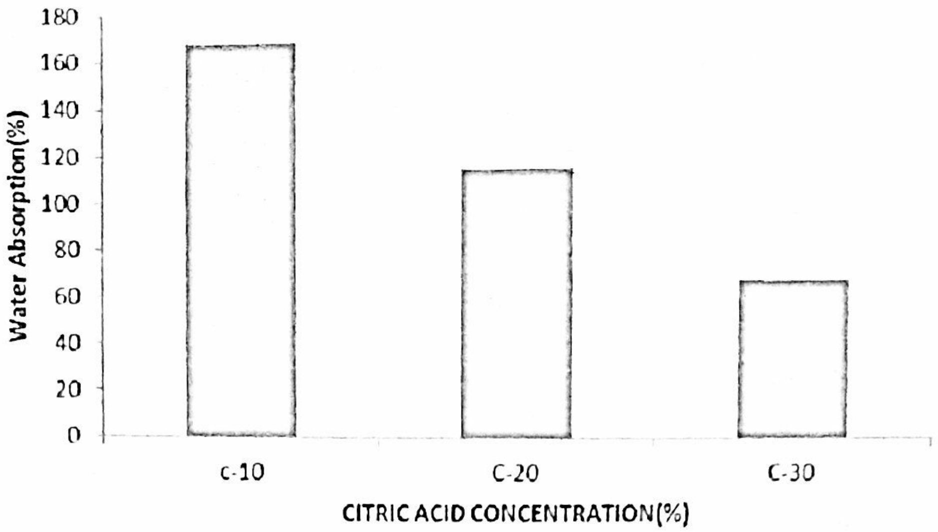


Figure 4.3: Water absorption at different citric acid content

Discussion

Here the probable cause of the variation may be due to the utilization of citric acid. In control treat 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). It was shown that the water absorption of particleboards bonded using citric acid was significantly decreased with increasing resin content. But venna particle absorb high amount of water. A study from Paridah *et al.* (2007), showed venna particleboard when bonded with formaldehyde-based adhesives absorb high amount of water. Light and highly porous venna could be a reason of high amount of water absorption. The highly porous structure of venna particle allowed water to penetrate through and to result in high water uptake hence causing thickness swelling at the same time. (Akim *et al*, 2011).

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 Apendix, Data table – 4, page no- 35). In control treatment highest value was found than the other value because no binding agent was used here and its density was low (0.61 gm/cm3) 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, here probable reason may be the utilization of citric acid at different concentration. Increasing citric acid concentration reduces the thickness swelling. Following figure (4.4) represents the data set-

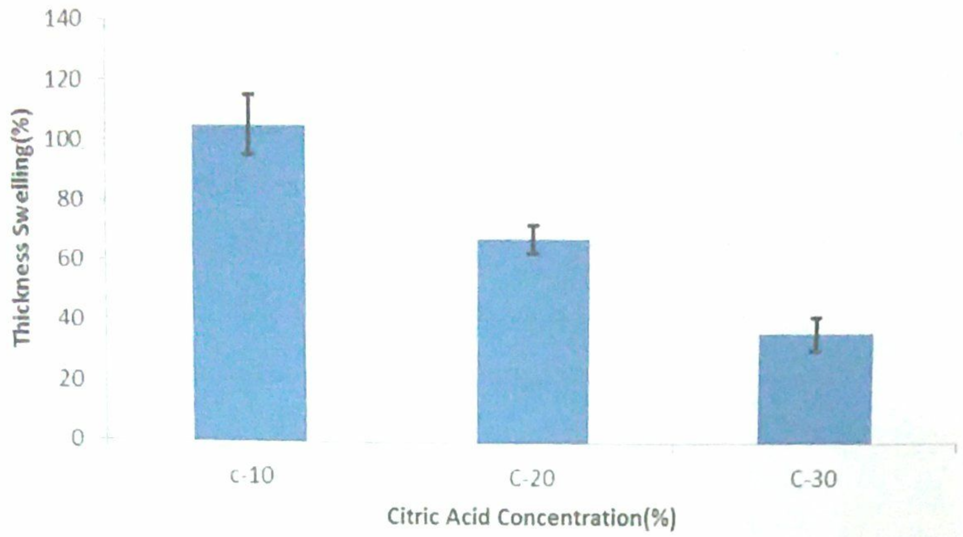


Figure 4.4: Thickness swelling at different citric acid content

Discussion

It is known that TS tends to increase with increasing board density because of the swelling of the wood itself and the release of the compression stress from the pressing operation (Halligan AF, 1970). According to Moslemi (1985) 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).

Board density had an effect on TS; however it was influenced by the citric acid content. A study from Widyorini *et al.* (2015) showed thickness swelling of citric acid bonded venna 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.

4.1.2 Mechanical properties

4.1.2.1 Modulus of elasticity (MOE)

Modulus of elasticity of the boards were tested and analyzed statistically as ($\alpha = 0.05$, $df = 5$, $p < 0.01$; See Apendix, Data table – 5, page no:35-36) and significant differences were found. Further LSD 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 MOE value (2194.742 Mpa, Specific MOE 5249.33 Mpa) and satisfied ANS, IS and ANZS standard.

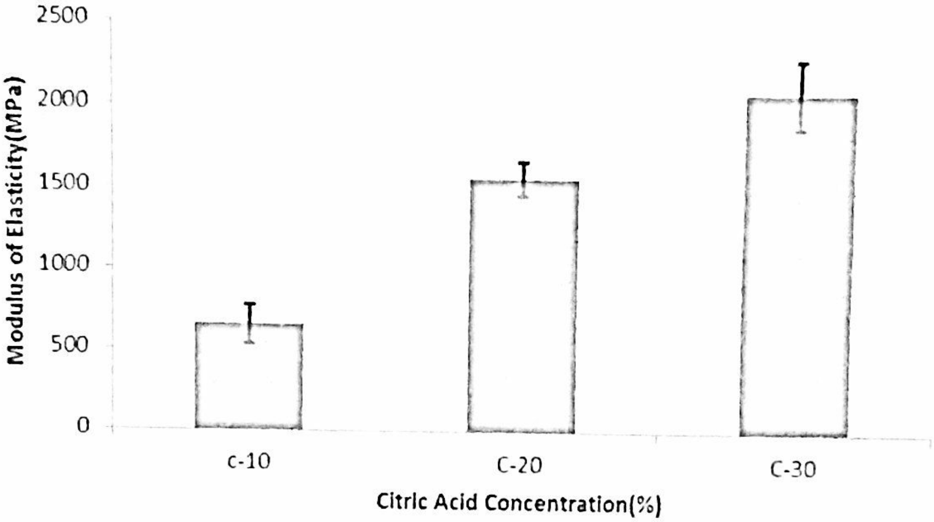


Figure 4.6: Effect of citric acid on modulus of elasticity

Discussion

A study on venna particleboard bonded with formaldehyde based adhesive of Akim .*et al.* (2011) suggests that the mechanical properties of citric acid bonded particleboard at 30% concentration exhibits good MOE and MOR value. Without it 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. 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 venna; their inherent chemical properties and their nature of compaction under heat and pressure may influence the value.

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, $p < 0.01$; See Apendix, Data table – 6, page no-36) and significant differences were found. Further LSD was done to find out the best treatment. Among the treatments citric acid bonded C-30 board showed satisfactory result. Following figure – 4.7 presents the statistical data. Here, 30% citric acid content showed the satisfactory MOR value (12.5725 Mpa, Specific MOR 16.07 Mpa) and satisfied ANS, IS standard.

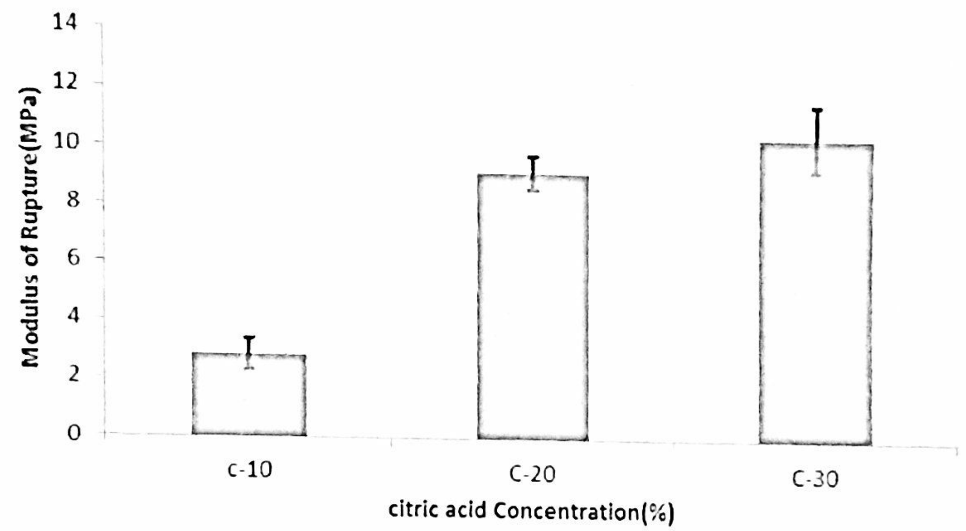


Figure 4.7: Effect of citric acid on modulus of rupture

Discussion

The property of binderless board was not satisfactory, where citric acid bonded board showed higher value. Comparing binderless with citric acid bonded composite board indicates binderless layered [Fine: Coarse:fine] particle developed poor bonding. 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.

CHAPTER FIVE: CONCLUSION

5.1 Conclusion

The effects of citric acid on the physical and mechanical properties of composite board were investigated. When citric acid added with raw material (Venna) used in composite board, it could significantly improve the dimensional stability and mechanical properties of the boards. The properties of citric acid-bonded composite board met the requirements of different standard like Japanese industrial standard for particleboard (JIS A 5908), American National Standard (ANS) and Indian Standard (IS).

In this study the optimum properties of Citric Acid bonded particle board were - modulus of elasticity 2194.742MPa, modulus of rupture 12.5725MPa, water absorption 67.28075%, thickness swelling 38.1870% . The result indicated that all properties were satisfactory except water absorption and thickness swelling. So, utilization of citric acid as a natural binder has a great potentiality as eco-friendly binder industrially in the field of particle board manufacturing in near future.

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Density

Data Table – 1

Treatment	c-10	C-20	C-30
A	0.61	0.79	0.78
B	0.57	0.72	0.76
C	0.73	0.78	0.93
D	0.66	0.78	0.92

SUMMARY - 1

Groups	*Count*	*Sum*	*Average*	*Variance*
c-10	4	2.57	0.6425	0.004758333
C-20	4	3.07	0.7675	0.001025
C-30	4	3.39	0.8475	0.008091667

ANOVA - 1

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.0854	2	0.0427	9.232432432	0.006600741	4.256495
Within Groups	0.041625	9	0.004625			
Total	0.127025	11				

Moisture content

Data Table – 2

Treatment	c-10	C-20	C-30
A	8.33	6.25	14.03
B	7.69	7.14	14.05
C	9.1	6.66	14.28
D	7.69	8.33	14.3

DATA SUMMARY – 2

Treatment	c-10	C-20	C-30
A	8.33	6.25	14.03
B	7.69	7.14	14.05
C	9.1	6.66	14.28
D	7.69	8.33	14.3

ANOVA - 2

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	67.10195	2	33.550975	1.568999686	0.260273	4.256495
Within Groups	192.45305	9	21.38367222			
Total	259.555	11				

Water Absorption

Data Table – 3

Treatment	c-10	C-20	C-30
A	184.8214	149.519	40.033
B	167.0329	100	70
C	161.3075	103.335	71.04
D	164.2854	107.142	88.05

DATA SUMMARY- 3

Groups	*Count*	*Sum*	*Average*	*Variance*
c-10	4	677.4472	169.3618	111.6881976
c-20	4	459.996	114.999	538.1272487
c-30	4	269.123	67.28075	398.4426356

ANOVA - 3

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	20870.5149	2	10435.25745	29.8645657	0.000106404	4.256494729
Within Groups	3144.774246	9	349.4193606			
Total	24015.28915	11				

Thickness swelling

Data Table – 4

Treatment	c-10	C-20	C-30
A	113.292	70.7905	31.514
B	89.275	53.821	28.7012
C	130.10075	74.9209	33.181395
D	89.085	71.82561	52.6785

DATA SUMMARY – 4

Groups	*Count*	*Sum*	*Average*	*Variance*
113.292	3	308.46075	102.82025	558.1782852
70.7905	3	200.56751	66.85583667	129.8254302
31.514	3	114.561095	38.18703167	162.5200277

ANOVA - 4

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	6292.792319	2	3146.396159	11.09808933	0.009635694	5.14325285
Within Groups	1701.047486	6	283.5079144			
Total	7993.839805	8				

Modulus of elasticity (MOE)

Data Table-5

Treatment	c-10	C-20	C-30
A	720.599	1264.75	1745.25
B	588.602	1704.69	2585.136
C	375.132	1621.68	1754.08
D	953.025	1683.25	2245.01

DATA SUMMARY – 5

Groups	*Count*	*Sum*	*Average*	*Variance*
720.599	3	1916.759	638.9196667	85388.98055
1264.75	3	5009.62	1669.873333	1856.866433
1745.25	3	6584.226	2194.742	174558.6727