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DECLARATION

I, Md. Mahmud Reza, 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, hereby, give consent for my thesis, if accepted, to be available for photocopying and for inter-library loans, and for the title and summary to be made available to outside organizations only for research and educational purposes.

Signature

Md. Mahmud Reza

Dedicated

To

Samiya, Nusrat and Tanvi

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Abstract

This paper represents the physical and mechanical properties of kenaf (*Hibiscus cannabinus*) particleboard bonded with citric acid as natural binder. In this study, the effects of different citric acid content on physical and mechanical properties of particleboard were investigated. Citric acid contents were set at 0, 5, 15 and 30% based on air dried particles. Two types of particle (coarse and fine) were used. The particleboards were also made as a single layer and three layer boards with variation of layer composition. Fine particles provided higher internal bond strength of the particleboard than coarse particles, due to bigger contact area among fine particles. Single layer particleboard and three layer particleboard showed almost same internal bond strength. Again the results showed that addition of citric acid could significantly improve the dimensional stability and mechanical properties of the boards. The properties of citric acid-bonded kenaf particleboard could meet the requirements of the Japanese industrial standard for particleboard. The mechanism behind such type of adhesion is that the ester linkages resulting from the reaction between citric acid and wood and resulted in particleboard with excellent properties.

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Chapter One: Introduction

1.1 Background of the study:

Industries today are under tremendous pressure to design ecologically and environmentally friendly viable materials for their products. This is because of growing environmental awareness and new rules and regulations that are binding on industries. As a result researcher's choices are shifting from synthetic composite and plastics to natural composites. A substantial increase in the agricultural by products and wastes of different types has attracted many researchers to develop and characterize new and low cost wood based materials from renewable local resources. 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 particleboard manufactured from kenaf.

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 to the climate. Faster growth and higher yield gives us the opportunity of versatile uses of kenaf. Kenaf 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. This study was to investigate the influence of resin content to the mechanical and physical properties of low density kenaf core particleboard.

1.2 Objectives of the study:

- ✓ To introduce a natural adhesive (citric acid) for manufacturing particleboard.
- ✓ To assess the feasibility of citric acid as a binder for manufacturing particleboard.
- ✓ To increase economic value of citric acid bonded kenaf particleboard.
- ✓ To carryout comparative study of quality of citric acid bonded kenaf particleboard.
- ✓ To know the physical and mechanical properties of kenaf particleboard bonded with citric acid.

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, 1969). 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 particle 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 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
- ✓ Layered
- ✓ Graded structure
- ✓ 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) 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 (Source: Kenji Umemura and Shuichi Kawai, 2015)

a) Saccharide based
   - → Starch
   - → Cellulose etc.

b) Protein based
   - → Animal glue
   - → Casein
   - → Soy protein
   - → Blood albumin etc.

c) Aromatic based
   - → Lignin
   - → Tannin etc.

d) Oil based
   - → Castor oil
   - → Canola oil etc.

e) 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 pK_a 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. (*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 kenaf

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.)

Scientific Classification:

Kingdom: Plantae

(Unranked): Angiosperms

(Unranked): Eudicots

(Unranked): Rosids

Order: Malvales

Family: Malvaceae

Genus: *Hibiscus*

Species: *H. cannabinus*

Bionomial Name: *Hibiscus cannabinus* L (Sources: Wikipedia)

2.8.1 Structure and Chemical composition of kenaf:

Yield component research with five kenaf cultivars showed averaged 26% leaves and 74% stalks by weight (Webber 1993b). In the same research the kenaf stalk's average composition was 35% bark and 65% woody core by weight. The bark of the kenaf stalk contains the long fiber strands that are composed of many individual smaller fibers, normally called bast fibers. These individual bast 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. The individual bast fibers are longer and thinner than the individual shorter, thicker core fibers. Oil in seed and crude protein in kenaf leaves and stalks are also present.

2.8.2 Uses of Kenaf:

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. Kenaf is being used as food and fodder. Although kenaf is usually considered a fiber crop, the entire kenaf plant, stalk (core and bark) and leaves, can be used as a livestock feed. Research indicates that it has high protein content (Clark and Wolff 1969; Killinger 1969). Crude protein in kenaf leaves ranged from 14% to 34% (ranged from 6% to 23% (Killinger 1969; Swingle et al. 1978; Webber 1993a). Kenaf can be ensilaged effectively, and it has satisfactory digestibility with a high percentage of digestible protein (Wing 1967).

Digestibility of dry matter and crude proteins in kenaf feeds ranged from 53% to 58% and 59% to 71%, respectively (Wing 1967; Killinger 1969; Suriyajantratong et al. 1973; Swingle et al. 1978; Webber 1993a), stalk crude protein ranged from 2% to 12% (Swingle et al. 1978; Webber 1993a), and whole-plant crude protein Whole stalk kenaf (bast and core fibers) has been identified as a promising fiber source for paper pulp (Nieschlag et al. 1960; White et al. 1970). The kenaf fibers, bast and core, can be pulped together or separated and pulped individually depending on the pulping process and the paper pulp to be produced (Kaldor et al. 1990).

2.9 Literature survey regarding the natural adhesive

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 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.* 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.

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 atroviolacea*), 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.

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 particleboard were also improved.

Chapter Three: Materials and Methods

3.1 Materials and Equipment

3.1.1 Materials:

Kenaf was used as a raw material for particleboard 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), owned by Akij Particle 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 particleboards. 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 Manufacture of particleboard

3.2.1Collection of Raw Materials

Kenaf was obtained from the kenaf field of Agrotechnology Discipline of Khulna University.

3.2.2 Processing & Screening of Raw Materials

The outer skin layer was scraped out with the help of scissor without damaging the surface and chipped individually by using traditional hand tools. Subsequently, the chips were converted separately into particle with a grinder by using a mesh opening at 1 mm. The grinded particles of kenaf 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 particle board. Mixture of coarse and fine particles also used for layered particle board.

Figure 1: Processing and screening of raw material

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.

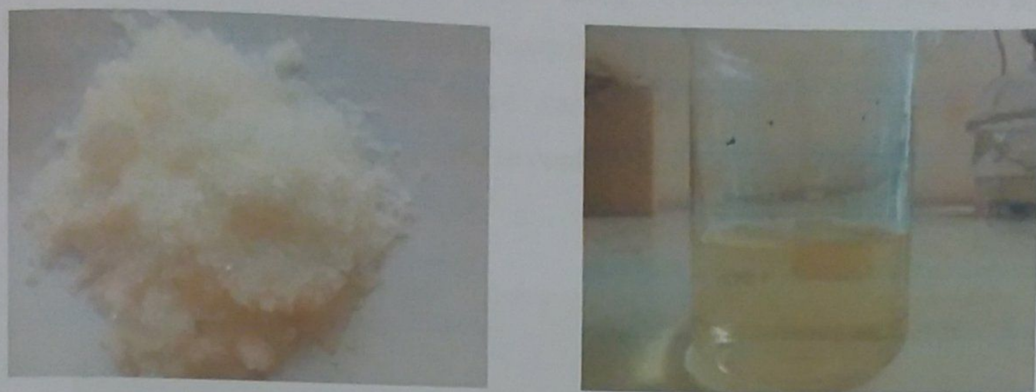


Figure 2: Solution preparation

Citric acid contents were set at 0, 5, 15, and 30% 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 were formed into a mat by using a wooden forming box, followed by hot pressing into particleboard. Single layered board of coarse particle using 0, 5, 15, 30% citric acid, single layered board of fine particles using 30% citric acid and a three layered board using 30% citric acid were prepared. The average mat thickness was 42 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: Mat formation

3.2.5Hot 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 4MPa. The temperature switch was switched off after 8 and 10 minutes. The mat were pressed at 180°c at a pressure of 4 MPa for 8 minutes (when 30% citric acid used), 10 minutes (when 15%, 5% citric acid used). Binderless particle board was also prepared at pressing temperature 190°c, at 4MPa for 10 minutes pressing. Density of the board was ranged from 0.3- 0.7 g/cm3. During hot pressing eight millimeter distance bar used to control the board thickness.

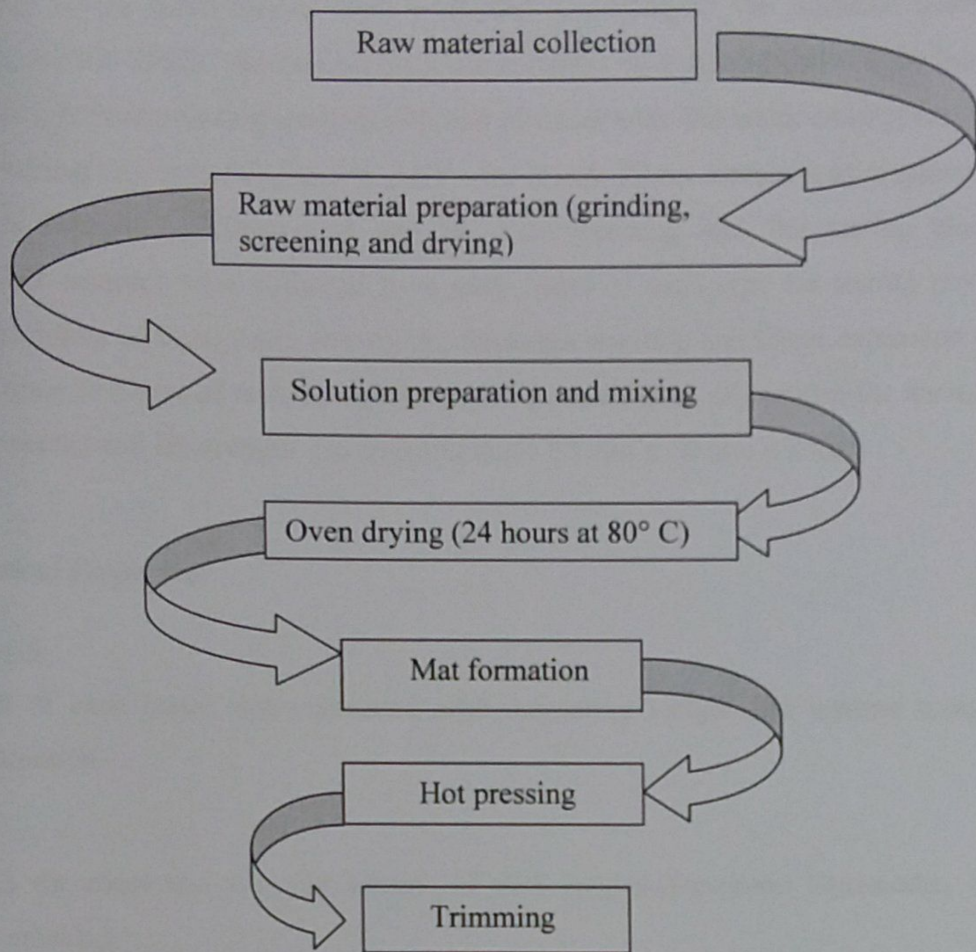
Figure 4: Hot pressing & board preparation

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 (AWPA, 2001).

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 of Kenaf 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, IB were tested from Akij Particle. 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 (JIS A 5908 2003). 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.

3.5.1 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.(Deschand Dinwoodie, 1996, http://www.imal.it.)

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.

(Deschand Dinwoodie, 1996, http://www.imal.it.))

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-

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

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

3.5.1.4 Thickness Swelling

Thickness Swelling was determined by using the following equation-

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

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

3.5.1.5 Linear Expansion

The Linear Expansion was calculated by the following formula-

$$LX(\%) = \frac{L_A - L_B}{L_B} \times 100 \text{ (ASTM, 1997)}$$

Where ,L_A = Length of sample after immersion (24 hr.) in water (mm),L_B = Length of sample before immersion in water (mm).

3.5.2Mechanical Properties

3.4.2.1 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-

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

$MOR = \frac{3PL}{2bd^2}$ (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)

3.5.2.2 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.

Internal bond *(N/mm2) = 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)

3.6 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 on 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 particleboard were prepared on different citric acid concentration. The board were named binderless (0% citric acid), C-5 (5% citric acid with coarse particle), C-15 (15% citric acid with coarse particle), C-30 (30% citric acid with coarse particle), F-30 (30% citric acid with fine particle) and Mix-30 (three layered board with 30% citric acid. The three layer board was in a ratio of 1:2:1 (fine: coarse: fine).

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. Density is an important parameter and it virtually affects all the properties of particleboard. The variation of density between particleboard was due to the variation of the raw materials itself. Density depends on the density of raw materials used, hot pressing conditions and other factors (Hsu *et al.*, 1988; Sekino, 1999; Volasqueze *et al.*, 2003). Pressing temperatures or press pressures have an important effect on board density. 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).

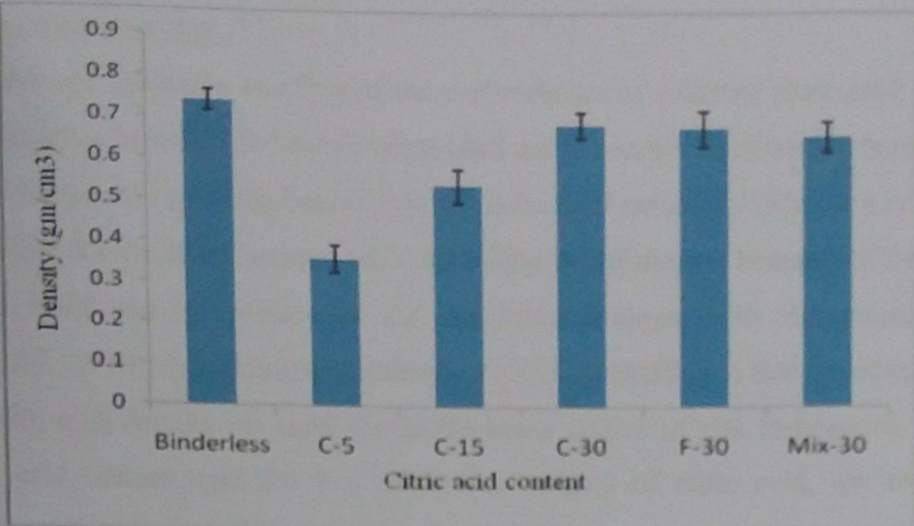


Figure 5: Density of particleboard at different citric acid content

In this study density of the boards were ranges from 0.35-0.7 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. Bending properties of the board were greatly influenced by density. The MOR and MOE increased linearly with increasing densities. Physical properties also affected by density variation.

4.1.1.2 Thickness swelling (TS)

Figure 6 showed thickness swelling of the particleboard at different citric acid content. This 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). 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 (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).

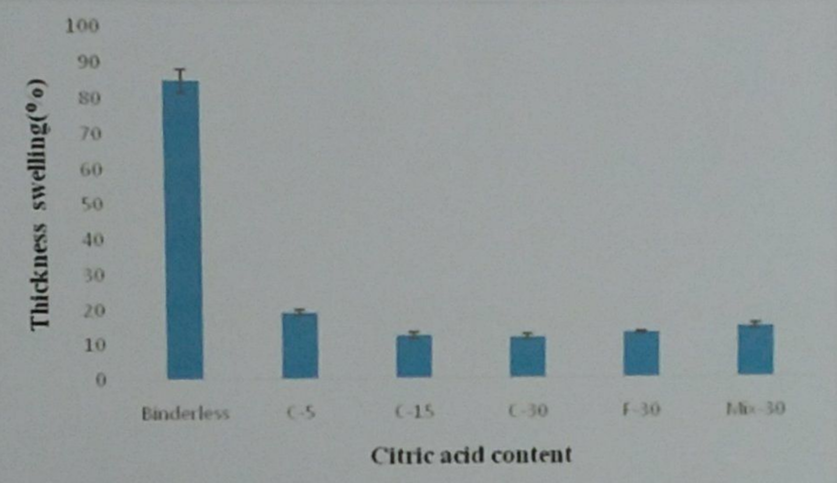


Figure 6: Thickness swelling at different citric acid content

The results showed that thickness swelling of the particleboard satisfy the requirement of type 8, type 13 and type 18 standard of JIS A 5908 except the binderless particleboard. The thickness swelling of binderless particleboard after half an hour soaking was 51.88% and after two hours thickness swelling of the board was 85.12% and after 24 hours the board was delaminated. Board density had an effect on TS; however it was influenced by the citric acid content. When single layered fine particleboard, three layered boards and single layered coarse particleboard bonded with 30% citric acid it showed thickness swelling of 13.13, 14.8 and 11.90% respectively. When 15% and 5% citric acid was used the TS value was 12.52 and 19.24%. Thickness swelling of particleboards decreases with increasing citric acid content. Similar was observed by Schneider et al (1996). A study from Widyorini et al. (2015) showed thickness swelling of citric acid bonded bamboo particleboard was very low compare to the binderless particleboard. In their study, the TS values were 35, 28, and 45 % for petung, wulung, and apus bamboo binderless particleboards, whereas it decreased to 9, 7, and 7 %, respectively, by addition of 15 wt.% of citric acid. 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 regaining of the particle original shape.

4.1.1.3 Water absorption

Figure 7 showed the water absorption of kenaf particleboard at different citric acid content. When single layered fine particleboard, three layered board and single layered of coarse particleboard bonded with 30% citric acid showed water absorption of 118.18%, 113.63% and 131.99% respectively, and for 15% and 5% citric acid the WA value was 139.66% and 146.42% after 24 hours of immersion. On the other hand binderless particle boards after 2 hours of immersion absorb water more than 154.61%.

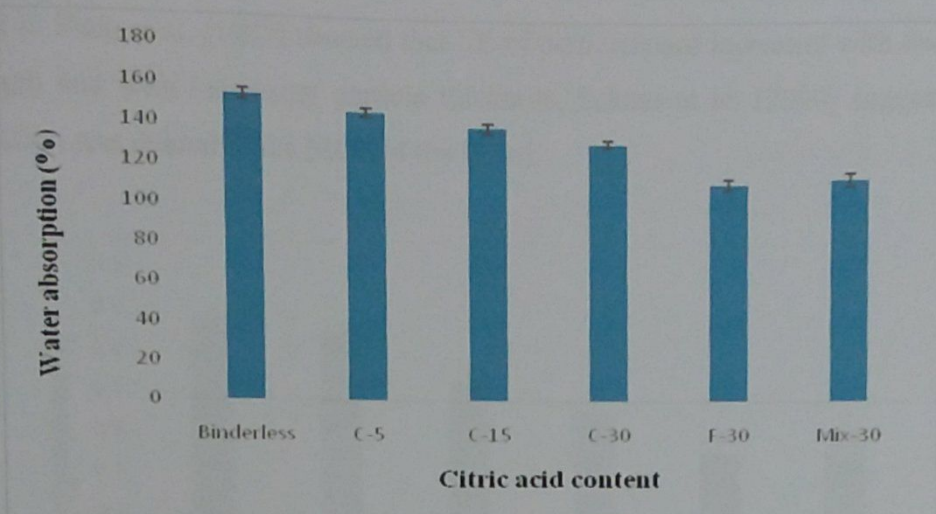


Figure 7: Water absorption at different citric acid content

The figure suggests that the board with high citric acid content absorb low amount of water compare to the binderless particleboard. Water absorption decreased with increasing board density in the case of kenaf core binderless board, 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. (Akim H. L. et al, 2011).

4.1.1.4 Linear expansion (LE)

Figure 8 showed different linear expansion at different citric acid content. The board composed of small particles showed a larger linear expansion at the same density level (Kohta Miyamoto et al. 2000). Generally, The planar or linear expansion of particleboards is 0.2 to 1.0% (Brochmann, J. et al. 2004). In this study single layer fine particleboard showed low LE compared to the coarse particleboard which was 0.53% for fine particleboard and 0.85, 0.73, 0.65 for C-5, C-15 and C-30 particleboard. Binderless and Mix-30 showed 0.88% and 0.61% LE. Suda et al. (1987) showed that LE of particleboard increased with decreasing particle length and with increasing particle thickness. Sekino et al. (2000) suggested that linear expansion was related to the MOE of the board.

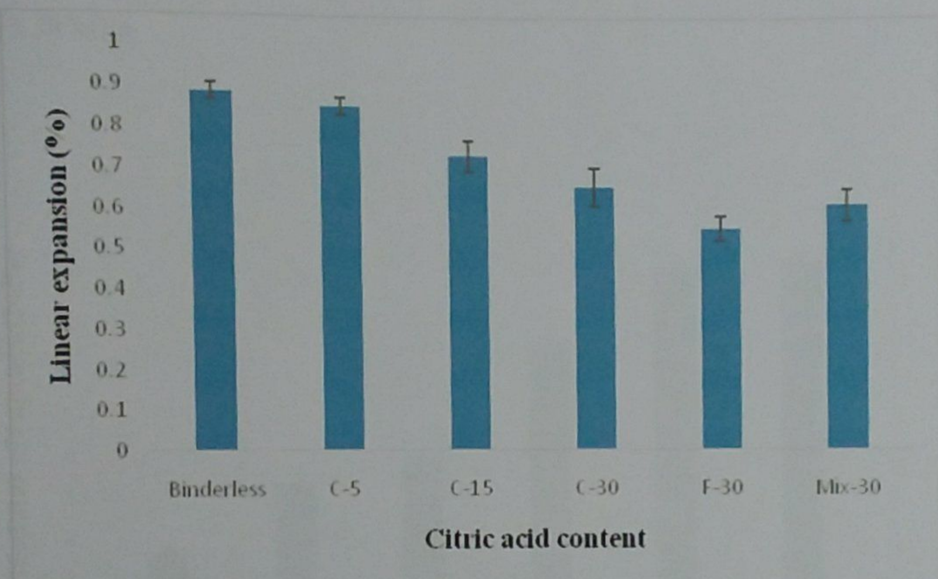


Figure 8: Linear Expansion at different citric acid content

4.1.2 Mechanical properties

4.1.2.1 Modulus of rupture (MOR)

Figure 9 showed the MOR value of the experimented board. The MOR value of binderless board made from kenaf was very low, i.e. 1.63 MPa, when citric acid was used at a range of 5%, 15%, 30% showed MOR value i.e. 3.32, 3.8, 6.68 MPa, and MOR value for fine and three layered board were 8.38 and 8.83 MPa. Comparing binderless with citric acid bonded particleboard indicates binderless kenaf particle developed poor bonding. The property of binderless board was not satisfactory, where citric acid bonded board showed higher value. Single layered fine particleboard and three layered boards met the standard of type 8 of JIS A 5908, according to the standard MOR value was 8.0 MPa or more. The specific MOR value for binderless, C-5, C-15, C-30, F-30 and Mix-30 particleboard was 2.2, 10.38, 7.04, 9.68, 12.32 and 13.38 MPa.

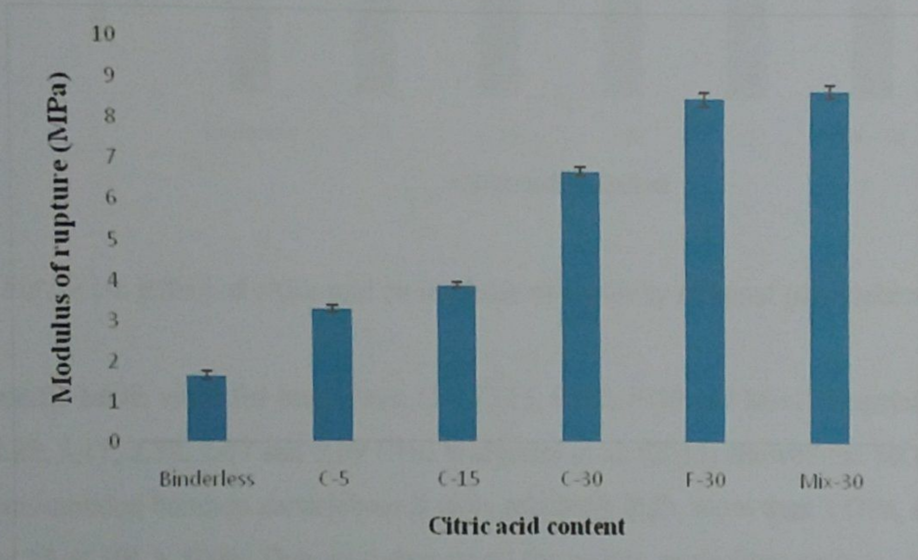


Figure 9: Effect of citric acid on modulus of rupture of kenaf particleboard

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.%.

4.1.2.2 Modulus of elasticity (MOE)

The MOE value of the particleboard also higher for citric acid bonded particleboard; it satisfies the standard, that was more than 3 GPa according to the standard of type 8 of JIS A 5908. A study on kenaf particleboard bonded with formaldehyde based adhesive of Akim H.L.et al, (2011) suggests that the mechanical properties of citric acid bonded particleboard at 30% concentration exhibits good MOE and MOR value.

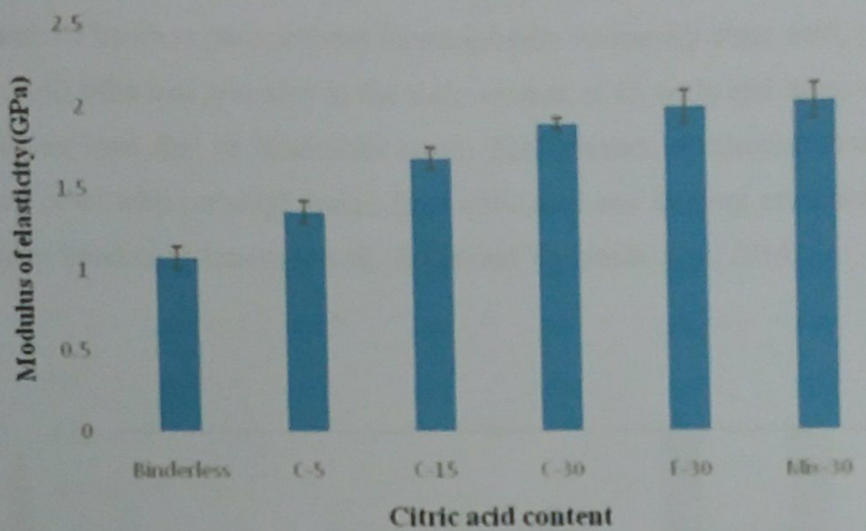


Figure 10: Effect of citric acid on modulus of elasticity of kenaf particleboard

The specific MOE value for binderless, C-5, C-15, C-30, F-30 and Mix-30 particleboard was 1.46, 4.28, 3.17, 2.78, 2.97 and 3.09 GPa. Widyorini et al. (2015) showed the MOE values of citric acid-bonded bamboo particleboards were relatively high, more than 3 GPa, the standard of Type 18 of JIS A 5908. They also showed all the boards made from particles bonded with 30 wt.% resin content had average MOR and MOE that exceeded the minimum requirements for grade 8 of JIS A 5908 particleboards, i.e. 12.3 MPa and 4.1 GPa for fine particles and 9.7 MPa and 4.3 GPa for coarse particles.

4.1.2.3 Internal Bonding Strength (IB)

Figure 10 showed the internal bond (IB) strength of the particleboard. The IB value of the particleboard increased with increasing citric acid content. Comparing with binderless particleboard it was higher and citric acid bonded particleboard met the requirement of JIS A 5908. The strength of the board bonded using citric acid was much higher than the requirement (0.3 MPa) for Type 18 of JIS A 5908. The result suggests that the mechanical properties of the boards bonded with citric acid were greatly affected by the resin content. 30% Citric acid content was the optimum. 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 kenaf react with carboxyl groups from citric acid and forming ester linked groups, supports superior bonding (Umemura et al., 2012a and Widyorini et al. 2014).

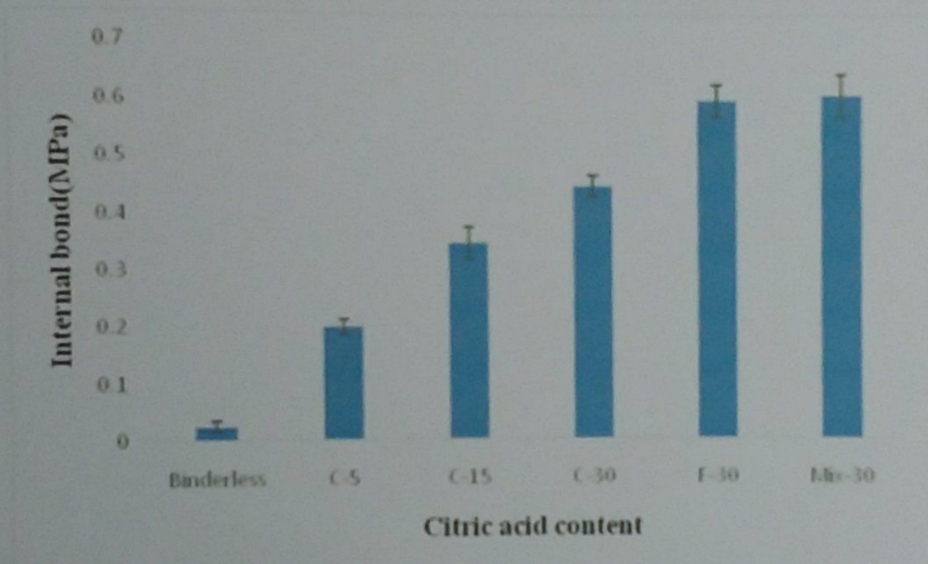


Figure 11: Effect of citric acid on internal bond strength of kenaf particleboard

The IB strength of particleboard made from fine particles was higher than that of boards made from coarse particles. It was probably due to the larger surface contact area of fine particles than that of coarse particles (Okuda and Sato 2004). 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)

Chapter Five: Conclusion

The effects of citric acid on the physical and mechanical properties of particleboard were investigated. When citric acid added with kenaf particles it could significantly improve the dimensional stability and mechanical properties of the boards. Single layered and three layered particleboard showed higher internal bond strength with smoother surface compared to the single layer coarse particleboard. The properties of citric acid-bonded kenaf particleboard met the requirements of the Japanese industrial standard for particleboard (JIS A 5908). In this study the optimum properties of kenaf particleboard were internal bond strength 0.61 MPa, modulus of rupture 8.83 MPa, modulus of elasticity 1.93 GPa, thickness swelling 14.8%, linear expansion 0.73% and water absorption 113.63%. The result indicated that carboxyl groups from citric acid were ester linked with hydroxyl groups from kenaf to produce better properties of particleboard. So, utilization of citric acid as a natural binder has a great potentiality as eco-friendly binder in the field of particleboard manufacturing.

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Appendix:

ANOVA for Density

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.311578	5	0.062316	68.39512	2.13E-08	3.105875
Within Groups	0.010933	12	0.000911			
Total	0.322511	17				

LSD for Density

Treatments	N	Mean	Grouping
C5	4	0.7400	A
F30	4	0.6900	A B
Mix30	4	0.6800	A B
BL	4	0.6600	B
C30	4	0.5400	C
C15	4	0.3625	D

ANOVA for Thickness Swelling (TS)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	12026.68	5	2405.337	276.9863	5.83E-12	3.105875
Within Groups	104.2075	12	8.683956			
Total	12130.89	17				

LSD for TS

Treatments	N	Mean	Grouping
BL	4	85.12	A
C5	4	19.240	B
Mix30	4	14.798	B C
F30	4	13.188	B C
C15	4	12.518	C
C30	4	11.995	C

ANOVA for Water Absorption (WA)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	4547.479	5	909.4958	26.22845	4.55E-06	3.105875
Within Groups	416.1111	12	34.67592			
Total	4963.59	17				

LSD for WA

Treatments	N	Mean	Grouping
BL	4	154.61	A
C5	4	146.41	A B
C15	4	139.66	B C
C30	4	131.99	C
Mix30	4	113.63	D
F30	4	111.36	D

ANOVA for Linear Expansion (LE)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.286867	5	0.057373	9.414038	0.000774	3.105875
Within Groups	0.073133	12	0.006094			
Total	0.36	17				

LSD for LE

Treatments	N	Mean	Grouping
BL	4	0.9200	A
F30	4	0.7900	B
C5	4	0.7600	B
Mix30	4	0.7300	B
C30	4	0.7100	B
C15	4	0.7000	B

ANOVA for Modulus of Rupture (MOR)

Source of Variation	*SS*	*Df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	132.481	5	26.49619	939.2112	4.01E-15	3.105875
Within Groups	0.338533	12	0.028211			
Total	132.8195	17				

LSD for MOR

Treatments	N	Mean	Grouping
Mix30	4	13.3800	A
F30	4	12.3200	B
C5	4	10.3800	C
C30	4	9.6800	D
C15	4	7.0400	E
BL	4	3.0400	F

ANOVA for Modulus of Elasticity (MOE)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	2.360644	5	0.472129	19.72226	2.07E-05	3.105875
Within Groups	0.287267	12	0.023939			
Total	2.647911	17				

LSD for MOE

Treatments	N	Mean	Grouping
C5	4	3.5925	A
C15	4	3.0000	B
Mix30	4	2.9300	B C
F30	4	2.7800	C D
C30	4	2.6800	D
BL	4	1.6200	E

ANOVA for Internal Bonding (IB)

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.737828	5	0.147566	65.10245	2.83E-08	3.105875
Within Groups	0.0272	12	0.002267			
Total	0.765028	17				

LSD for IB

Treatments	N	Mean	Grouping
Mix30	4	0.6100	A
F30	4	0.6000	A
C30	4	0.4500	B
C5	4	0.2000	C
BL	4	0.1800	C
C15	4	0.1700	C