

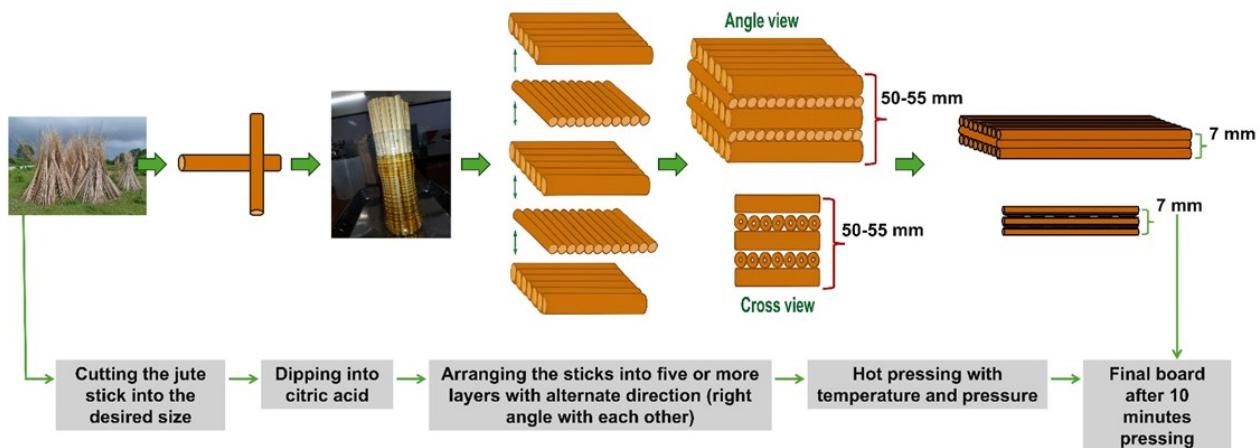
# A Simplified Manufacturing Technique of Fabricating Jute Stick Cross-banded Board Reinforced with Citric Acid as a Natural Binder: Process Optimization and Characterization

Summia Rahman,<sup>a,\*</sup> Moushumi Akter,<sup>b</sup> Aynun Nishat Farhabi ,<sup>c</sup> Sukma S. Kusumah,<sup>d</sup> Kenji Umemura,<sup>e</sup> and Md. Iftekhar Shams <sup>b</sup>

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## GRAPHICAL ABSTRACT



# A Simplified Manufacturing Technique of Fabricating Jute Stick Cross-banded Board Reinforced with Citric Acid as a Natural Binder: Process Optimization and Characterization

Summia Rahman,<sup>a,\*</sup> Moushumi Akter,<sup>b</sup> Aynun Nishat Farhabi ,<sup>c</sup> Sukma S. Kusumah,<sup>d</sup> Kenji Umemura,<sup>e</sup> and Md. Iftekhar Shams  <sup>b</sup>

A simple manufacturing strategy was developed to fabricate cross-banded jute stick boards using citric acid (CA). The effects of CA concentrations and pressing temperatures on the physical and mechanical properties of the boards were systematically investigated. Jute sticks were impregnated with CA concentration ranging from 20 to 60 wt% and hot-pressed at 160 to 220°C at a pressing pressure of 5 MPa. Boards treated with 40 wt% CA exhibited the highest modulus of rupture (53.6 N/mm<sup>2</sup>) and internal bond strength (0.52 N/mm<sup>2</sup>), while those treated with 60 wt% CA showed superior dimensional stability, with a thickness swelling of 14.7% at a pressing temperature of 200 °C. Fourier transform infrared spectroscopy analysis confirmed the formation of ester linkages between the carboxyl groups of CA and the hydroxyl groups of jute stick components, resulting in strong chemical bonding and interfacial adhesion. Therefore, by optimizing the processing parameters, CA-treated jute stick crossbanded board was successfully developed with enhanced mechanical strength and dimensional stability.

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Keywords: Jute stick; Mechanical properties; Cross-banded board; Dimensional stability; Citric acid

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## INTRODUCTION

Wood-based composites play a vital role in construction and interior design, serving as versatile materials for various applications such as plywood, veneers, and laminates. Traditional manufacturing of wood-based composite boards includes the mixing of wood elements (fibers, particles, flakes) with a synthetic or natural adhesive. This mixture is then formed into a mat and hot-pressed under controlled temperature and pressure to cure the adhesive and consolidate the material into a solid panel (Cai and Ross 2010; Lyutyy *et al.* 2024; Amarasinghe *et al.* 2024; Garcia *et al.* 2024; Mbappe and Nkeng 2023; Kristak *et al.* 2025). This process is widely followed, and the production process often relies on urea-formaldehyde (UF) resins (Dunk 1998). Essentially, this results in a notable release of formaldehyde shortly after manufacturing, followed by continuous

emissions that can persist throughout the product's lifespan. The dependence on formaldehyde-based adhesives certainly raises concerns about environmental and health impacts from the emission of volatile organic compounds, hence the increased regulatory control and development of bio-based alternatives (Mulligan *et al.* 2022; Garcia *et al.* 2024; Kristak *et al.* 2025). In this regard, considerable research has already been conducted on alternative production methods, such as the eco-friendly concept of manufacturing binderless boards, which eliminates the need for UF resin. Binderless boards are manufactured by harnessing the inherent bonding potential of the existing chemical components of the raw materials under controlled temperature and pressure conditions. The activation of such chemical components during hot-pressing is an effective approach to facilitate self-bonding within the matrix, resulting in composite board fabrication (Xu *et al.* 2003, 2006; Widyorini *et al.* 2005; Okuda *et al.* 2006; Okuda and Sato 2007). It is reported by Widyorini *et al.* (2005) and Nitu *et al.* (2020) that partial degradation of the lignocellulosic components, namely hemicellulose and lignin, in kenaf core and jute stick fibers during pressing, improved inter-fiber bonding and the dimensional stability of the produced binderless boards. However, even under these conditions, the properties related to the mechanical and dimensional stability of such boards remain insufficient to satisfy industrial requirements. In order to overcome this gap, later works investigated the addition of natural adhesives. Evaluated binders have included tannins-based, lignin-based, protein-based, citric acid based, and ascorbic acid based natural adhesives (Yang *et al.* 2006; Mansouri *et al.* 2007; Krug and Tobisch 2010; Nitu *et al.* 2022; Farhabi *et al.* 2022). Although these biobased composite materials increased environmental safety and reduced the dependence on synthetic resins, the manufacturing process of such materials followed traditional methods. The traditional board manufacturing method has a number of disadvantages despite its widespread adoption. For example, moisture gradients within the mat give rise to internal stresses, blistering, or delamination, and a poor distribution of adhesive can yield local areas of weak bonding (Rosenfeld *et al.* 2022).

In this study, a novel manufacturing process of a composite board named cross-banded board was revealed. Cross-banded boards, with successive layers oriented perpendicular to each other, can have advantages over traditional particleboard and laminated board configurations. This structure has the potential to provide better dimensional stability by opposing the anisotropic swelling and shrinkage, which reduces warping and deformation under fluctuating humidity conditions. Unlike particleboards, where lignocellulosic particles are randomly oriented and held together by synthetic/natural adhesives, cross-banded boards have mechanical stresses distributed more uniformly across layers; therefore, they can possibly provide increased bending strength and stiffness compared to conventional configurations. Moreover, the layered architecture can enhance adhesive penetration and interfacial bonding, which is particularly true for bio-based adhesives such as citric acid, hence minimizing the risk of delamination and internal defects (Rosenfeld *et al.* 2022). Thus, better structural integrity and reduced susceptibility to moisture-induced failure possibly can make the cross-banded boards useful in load-bearing and high-performance applications, where traditional particleboards may fail due to lower strength-to-weight ratio and greater vulnerability to environmental stressors (Wang *et al.* 2022). Thus, the potential of cross-banded configurations to yield mechanically robust products make them a promising alternative to conventional board manufacturing methods. Therefore, in this study, a cross-banded board was manufactured using CA as a natural binder and jute stick.

CA, derived from citrus fruits such as lemons and limes, has been considered a natural adhesive because of its good dimensional stability with wood-based materials. It is reported that the addition of CA improved the mechanical properties and dimensional stability of the wood and bark molding (Umemura *et al.* 2012a,b). Following the pioneering work of the previously mentioned author, later on several studies were carried out showing that CA could be useful as an adhesive for board manufacturing (Widyorini *et al.* 2014; Kusumah *et al.* 2016, 2017). CA contains three carboxyl groups, and when these carboxyl groups react with the hydroxyl group, the formation of ester linkage occurs, which is the key factor behind the bonding mechanism (Umemura *et al.* 2012a,b; Kusumah *et al.* 2016). The reaction mechanism between wood and CA entails a two-step esterification process. Initial loss of water from CA, upon heating of the CA results in an anhydride ring, which has a much greater reactivity with -OH groups. After the first ester linkage is formed, the remaining two carboxyl groups in CA can form another anhydride ring and repeat the process, resulting in a crosslink (Schramm and Rinderer 1999; Bischof Vukusic *et al.* 2006). Besides, the crosslinking reactions between poly carboxylic acid and wood were found to proceed *via* cyclic anhydride intermediates rather than by nucleophilic addition of the hydroxyl group and the carbonyl group. In addition, the breakdown of CA into unsaturated acids occurs when it is heated to a temperature of 175 °C. The primary compounds formed during dehydration or decarboxylation are acetone, dicarboxylic acid, aconitic acid, and itaconic acid (Schramm and Rinderer 1999; Wyrzykowski *et al.* 2010). Upon further heating of CA, production of methyl maleic anhydride can occur (Barbooti and Al-Sammerrai 1986; Passauer *et al.* 2009). Thus, temperature plays a crucial role in indicating the performance of CA.

Jute is a commonly known lignocellulosic plant mostly grown in South Asia (Shahinur *et al.* 2022). Jute plant's outer layer is composed of bast fibers, which can be separated from the inner layer of the stem. The bast fibers are long, soft, and lustrous, while it has a woody inner core, known as a jute stick (Ramesh and Deepa 2024). Jute fiber is widely used in textiles, packaging, and automotive nonwovens due to its versatile nature and biodegradability (Shahid *et al.* 2024; Shahinur *et al.* 2022). In contrast, Jute stick is a lignocellulosic biomass composed mainly of cellulose, hemicellulose, and lignin, although there is also a small fraction of ash and extractives. Guha Roy *et al.* (2022) reported that the jute stick, after extraction of fiber, contains approximately 45 to 50% cellulose, 20 to 25% hemicellulose, and 20 to 30% lignin, depending on the maturity of the plant and processing conditions. Such components contribute to the rigid structure in its thermal behavior, suitable for composite board applications. Jute stick was also reported to contain trace minerals and functional groups by Islam *et al.* (2022). During pyrolysis, such minerals responded well to chemical activation, resulting in increased reactivity toward bio-based adhesives. Earlier, the jute stick, being the woody core, usually has been neglected, either being discarded or used in village households (Vaishnavi and Krishnaveni 2025). However, recent research has focused on jute stick for its potential in sustainable board manufacturing, bioenergy, and agricultural applications, emphasizing that it is an abundant renewable biomaterial. This trend change is a result of increased interest in the valorization of agricultural by-products for the development of environmentally friendly materials such as particle board (Nitu *et al.* 2022; Chawla *et al.* 2023), panel production (Lakshmanan *et al.* 2025), biofuels (Sarkar and Wang 2020), and biochar (Alam *et al.* 2023). Therefore, to properly utilize the jute stick, this study developed a simple, formaldehyde-free process for manufacturing an environmentally friendly jute stick cross-banded board, utilizing whole jute sticks without crushing or grinding them, and using CA as a natural binder. This study

aimed to determine the optimum processing conditions of cross-branded jute stick board and characterization of such board materials.

## EXPERIMENTAL

### Materials

#### *Preparation of materials*

Jute sticks (*Corchorus capsularis*) were collected from the local market of Khulna district in Bangladesh and air-dried for two weeks under ambient conditions to reduce the surface moisture of the sticks. The sticks were then cut into the same lengths of 12 and 8 inches (30.5 and 20.3 cm), respectively, and oven-dried at 103 °C for 12 hours to bring about a moisture content below 4%. This is according to the preconditioning of lignocellulosic biomass. Citric acid (99.5% anhydrous) was supplied by EMD Millipore Corporation, Billerica, USA, and used as received, without further purification. Previous studies used CA as a bio-based adhesive for wood and fiber composites.

### Methods

#### *Fabrication of cross-banded board*

Citric acid was employed as a bio-based adhesive for fabricating cross-banded boards by dissolving it in distilled water to make 20 wt%, 40 wt%, and 60 wt% concentrations. Oven-dried jute sticks were impregnated in each CA concentration at both vertical and horizontal positions for 5 min. Oven-dried sticks after impregnation were then oven-dried again at 80 °C for 48 h. This temperature was previously determined as appropriate for the pre-drying treatment of the lignocellulosic substrates without compromising their integrity. To form the mats, the oven-dried jute sticks were arranged parallel to each other, but perpendicular to the previously formed layer, which was then hot-pressed with an applied pressure of 5 MPa at 160 to 220 °C for 10 min. The board target density during pressing was kept constant at approximately 0.8 g/cm<sup>3</sup> by controlling the thickness. Five boards for each condition were prepared, and samples were then kept at 25 °C and 60% relative humidity for one week before testing. The boards were prepared with standardized dimensions of 300 × 200 × 8 mm (length × width × thickness).

#### *Evaluation of mechanical properties and dimensional stability*

Three-point bending tests determining the static bending strength were conducted on a universal testing machine (Shimadzu AG-50KNXplus, Japan) with a 150 mm span and a loading rate of 10 mm/min, using 300 × 50 × 8 mm specimens. The modulus of rupture (MOR) and modulus of elasticity (MOE) were measured using the above-mentioned three-point bending test. Internal bonding (IB) strength was determined using eight 50 × 50 mm specimens per board. Water absorption (WA) and thickness swelling (TS) were measured after identical specimens were immersed in water at room temperature for 24 hours. A statistical analysis was carried out using one way ANOVA with a significance level of 0.05, followed by Tukey's Honestly Significant Difference (HSD) test. Means within the same column followed by the same letter (A, B, C, D) were not significantly different ( $p > 0.05$ ).

*Fourier transform infrared spectroscopy (FTIR)*

Fourier Transform Infrared Spectroscopy was carried out for chemical characterization on powdered samples of both raw jute sticks and CA-bonded boards. Samples were oven-dried at 60 °C for 12 h prior to analysis. Spectra were recorded using a Spectrum Two FT-IR Spectrometer (PerkinElmer, USA) over the range of 4000 to 1000 cm<sup>-1</sup>, with 64 scans at a resolution of 4 cm<sup>-1</sup> using the Universal Attenuated Total Reflectance (UATR) mode, enabling rapid and non-destructive identification of functional groups associated with esterification and lignocellulosic bonding.

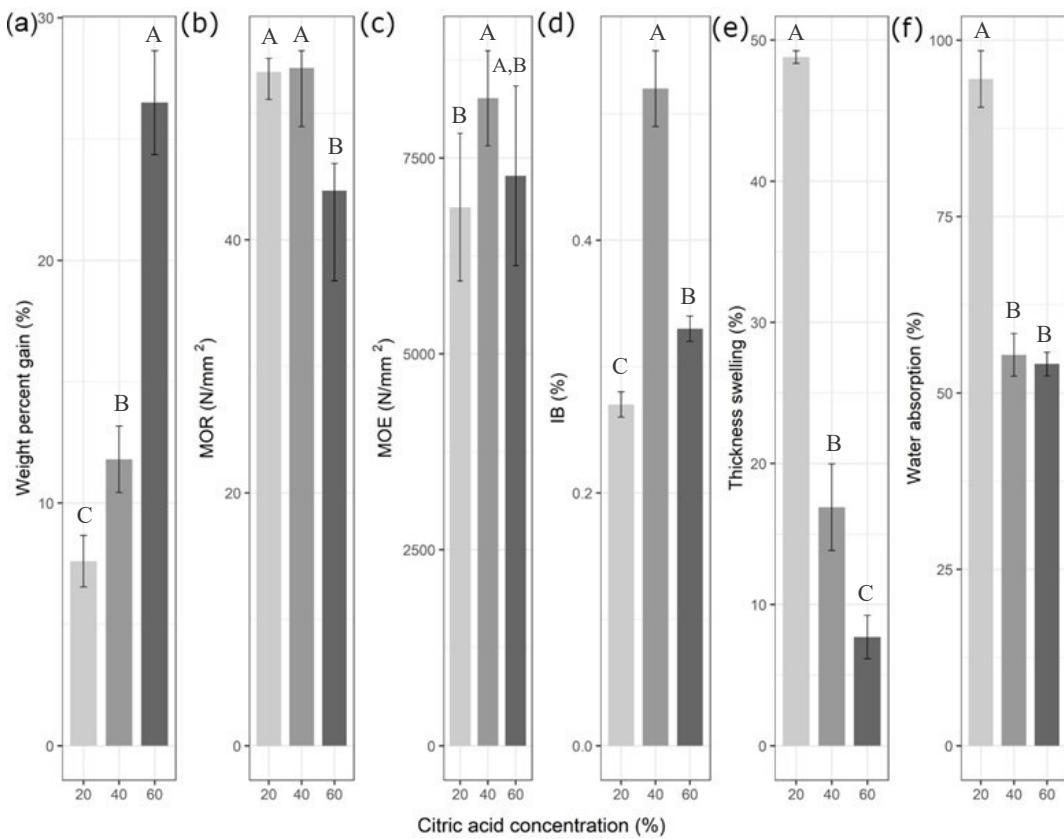
*Thermogravimetric analysis (TGA)*

TGA was performed on composite samples to investigate their thermal stability using a LABSys Evo STA (Simultaneous Thermal Analysis, Setaram Instrumentation, France). Prior to testing, all the specimens were manually cut into small fragments and then grounded into fine powders using a high-speed blender. The powdered samples were oven-dried at 60 °C for 12 h to eliminate residual moisture. For each of the dried samples, approximately 5 mg was subjected to thermal scanning from room temperature to 600 °C under a nitrogen atmosphere with a flow rate of 30 mL/min at a constant heating rate of 5°C/min. The protocol is in good agreement with established methodologies to characterize polymeric and lignocellulosic composites, where degradation stages and thermal transitions of interest for material performance could be identified.

## RESULTS AND DISCUSSION

### Effects of Different Concentrations of CA on Mechanical Properties and Dimensional Stability

The different concentrations of CA had significant influence on the dimensional stability and mechanical properties of the manufactured boards. Boards were pressed at 200 °C for 10 min at 5 MPa. With the increase in CA concentration from 20 wt% to 60 wt%, a remarkable darkening of the board surface was evident upon hot pressing, which indicated a deeper extent of chemical interaction. The weight percentage gain significantly increased with an increased CA concentration up to about 26% at 60 wt%, which was over twice as much as that of the 40 wt% treatment (Fig. 1a). The mechanical properties, such as MOR value, showed no significant difference between 20 wt% and 40 wt% CA-treated samples; however, MOR significantly decreased at 60 wt% (Fig. 1b), which was likely due to the embrittlement effect caused by excessive cross-linking. On the other hand, the MOE and IB strength significantly increased at 40 wt% but significantly decreased at 60 wt% (Fig. 1c,d). The average values of MOR, MOE, and IB at 40 wt% were 53.6 N/mm<sup>2</sup>, 8262.3 N/mm<sup>2</sup>, and 0.52 N/mm<sup>2</sup>, respectively, exceeding E50-F160 structural plywood of Japanese Agricultural Standards. The effects of different concentrations of CA on the thickness swelling and water absorption of the jute stick crossbanded boards are shown in Figs. 1 (e, f). The WA and TS values of the board significantly decreased with increasing concentration of the CA. The value of TS decreased from 48.8% at 20 wt% to 7.7% at 60 wt%. Similarly, the WA value at 20 wt% CA was 94.5%, whereas at higher concentrations, such as 60 wt%, the value was reduced to 54.1%. This may be due to the higher weight gain of the samples due to CA impregnation, as shown in Fig. 1a.



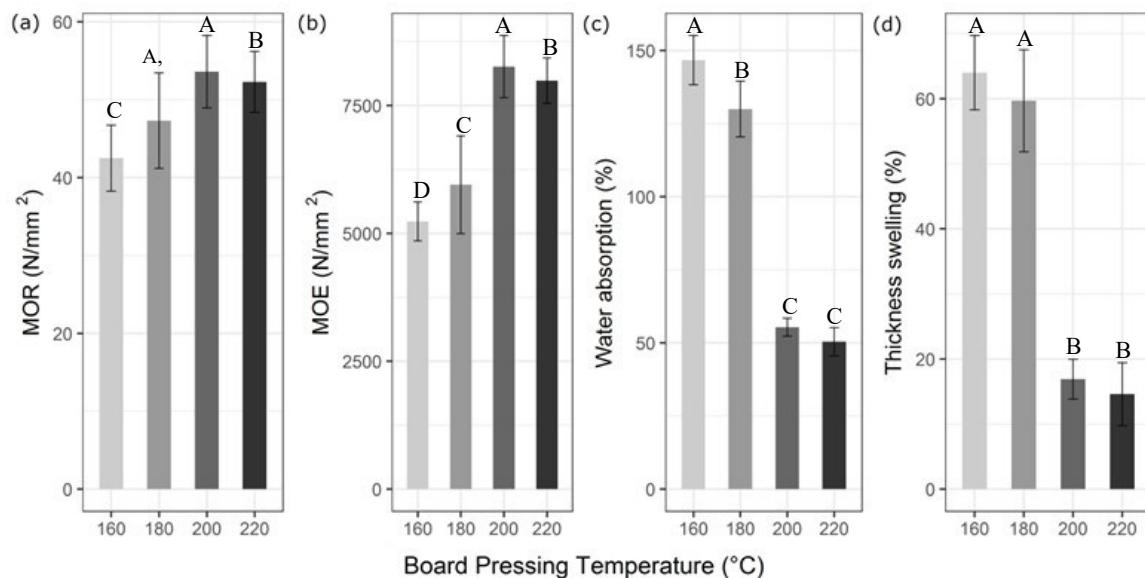
**Fig. 1.** Effects of citric acid concentration on weight percentage gain (a), mechanical properties (b,c), internal bond strength (d), thickness swelling (e) and water absorption (f) of jute stick cross-banded board. The pressing temperature used was 200°C, and the pressing time was 10 min. (Note: The letters A, B, and C represent the significance levels in statistical analysis. Values having different letters signify that they are significantly different. Throughout the study, the letters will bear the same meaning as mentioned.)

The overall improvement of mechanical properties and dimensional stability of the boards at 40 wt% might be due to an effective penetration of CA into the jute sticks, which resulted in a moderate weight gain. This appears to have triggered a strong chemical reaction between CA and jute sticks under applied temperature and pressure conditions. As reported earlier, upon heating CA, a sequential process of anhydride ring formation occurs, which increases reactivity toward hydroxyl (-OH) groups of jute stick, facilitating the formation of multiple ester linkages. This sequential process is directly linked to promoting the establishment of crosslinks, which have the potential to reduce the hygroscopicity of the samples. Thus, the hot-pressing enhanced the mechanical properties and dimensional stability of the resultant material, which are pivotal in the bonding mechanism of the CA-bonded board (Kusumah *et al.* 2016; Umemura *et al.* 2012a). However, at lower CA concentration (20 wt%), the impregnation of jute stick might be inadequate, resulting in lower weight percentage gain, thus possibly lowering the MOE and dimensional stability. On the contrary, at higher CA concentration (60 wt%), there were increased levels of ester linkages due to higher CA content, possibly contributing to enhanced dimensional stability. It has been reported that a higher percentage of CA can contribute to enhanced adhesiveness in the manufactured boards (Bischof Vukusic *et al.* 2006). However, with higher percentage of CA, the material is expected to have a higher degree of ester linkage formation, which can lead to increased brittleness. Thus, at higher concentrations of CA

(60 wt%), the manufactured boards likely underwent embrittlement, resulting in lower mechanical properties. Overall, the tradeoff between mechanical properties and dimensional stability at higher concentrations of CA indicates that optimization of CA concentration at 40 wt% is essential for balanced board performance.

### Effects of Pressing Temperatures on Mechanical Properties and Dimensional Stability

In an attempt to optimize processing conditions, the influence of pressing temperature on the mechanical and dimensional properties of jute stick boards bonded with 40 wt% CA was explored. As depicted in Fig. 2, MOR and MOE significantly improved with increasing pressing temperature from 160 to 200 °C, attaining maximum values of 53.6 and 8262 N/mm<sup>2</sup>, respectively. However, a further temperature increment to 220 °C led to a significant MOR reduction due to the thermal degradation and embrittlement of the surface layer (Yang *et al.* 2014). This can be explained by the decomposition of hemicelluloses, starting at around 220 °C (Parveen *et al.* 2011), and the thermal decomposition of CA around the same temperature (Barbooti and Al-Sammerrai 1986).



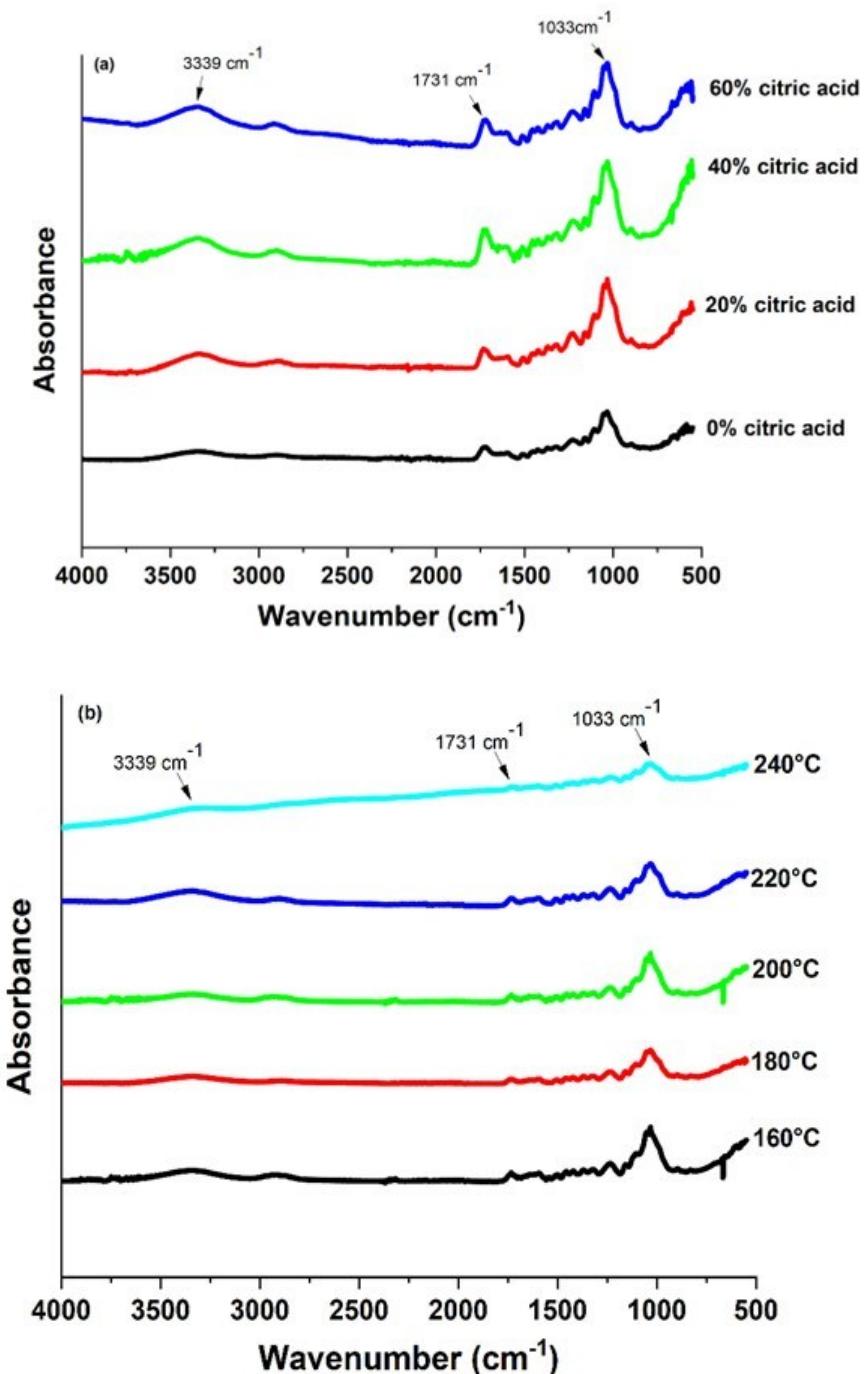
**Fig. 2.** Effects of pressing temperatures on mechanical properties (a,b) and dimensional stability (c,d) of jute stick cross-banded board treated with 40 wt% CA

A pressing temperature of 200 °C seemed optimal because it possibly allowed an effective melting of CA, hence subsequent esterification reactions between hydroxyl groups in the jute stick matrix and enhanced adhesive bonding. At temperatures above 200 °C, the degradation of CA and hemicelluloses became more pronounced, and volatile by-products formed, interfering with the adhesion of the core layers (Kusumah *et al.* 2017). Dimensional stability, as indicated by thickness swelling and water absorption, also improved with increased pressing temperatures, with maximum dimensional stability being attained at 200 to 220 °C. This is due to the more effective cross-linking between CA and lignocellulosic components at higher temperatures, as already reported for wood-based molding (Umemura *et al.* 2012b). However, lower pressing temperatures (160 to 180 °C) might be inadequate for good adhesion, resulting in significantly lower dimensional stability. Overall, the results suggest that a pressing temperature of 200 °C represents an

optimum for adhesive activation, allowing mechanical performance and dimensional integrity to be maximized in CA-bonded jute stick boards.

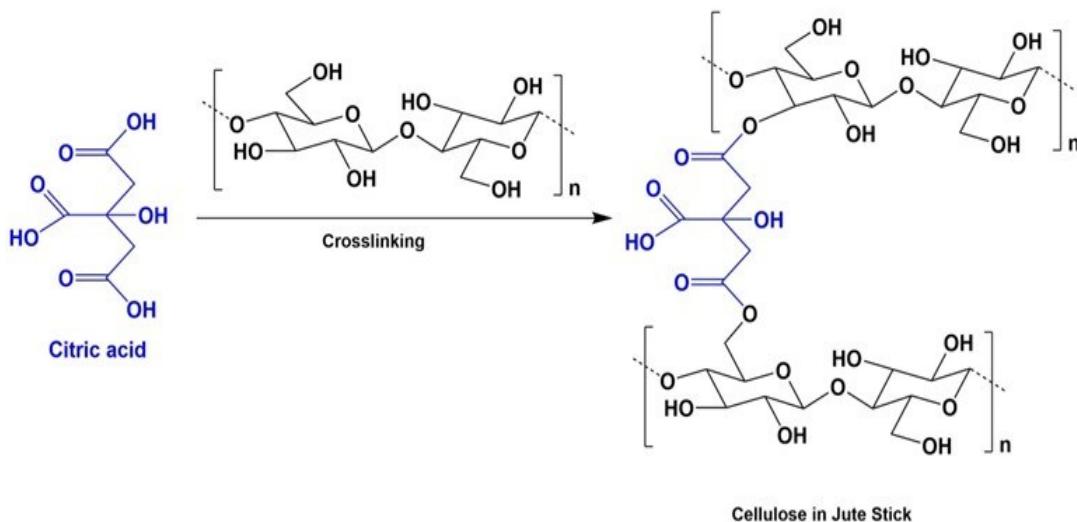
### FT-IR Analysis

FT-IR analysis was performed on the boards impregnated with different concentrations of CA and on raw jute sticks without CA treatment, as shown in Fig. 3a.



**Fig. 3.** a) FTIR analysis of jute stick cross-banded board treated with 0 wt%, 20 wt%, 40 wt% and 60 wt% CA, b) FTIR analysis of jute stick cross-banded board pressed at 160, 180, 200, 220, and 240 °C

The absorbance peak at  $1731\text{ cm}^{-1}$  corresponds to the C=O stretching vibration of ester groups, while the band at  $1033\text{ cm}^{-1}$  is attributed to the C–O stretching of the ester linkage. The increasing intensity of these two peaks with higher CA concentrations indicates the formation of a greater number of ester bonds, suggesting more extensive cross-linking between CA and the hydroxyl groups of jute components. Furthermore, the absorption band associated with hydroxyl groups (–OH) near  $3300\text{ cm}^{-1}$  showed both a reduction in intensity and a slight shift with increasing CA concentration, reflecting the consumption of free –OH groups. This reduction confirms esterification and the formation of hydrogen bonds between CA and cellulose (Yang *et al.* 1996; Žagar and Grdadolnik 2003). Therefore, boards treated with a higher concentration of CA exhibited higher dimensional stability (Kusumah *et al.* 2016, 2017) compared to boards bonded with lower concentrations of CA. In addition, FT-IR analysis was conducted for boards treated with 40 wt% CA at different pressing temperatures (Fig. 3b). Interestingly, the characteristic peaks at  $1731$  and  $1033\text{ cm}^{-1}$  were decreased with increasing pressing temperature. This suggests that higher temperatures led to partial degradation of hemicellulose and CA, resulting in reduced ester linkage formation and accelerated evaporation of hydroxyl-containing compounds during hot pressing. Considering the cellulose structure of the jute sticks, the possible esterification mechanism is illustrated in Fig. 4 (Basak *et al.* 2025).

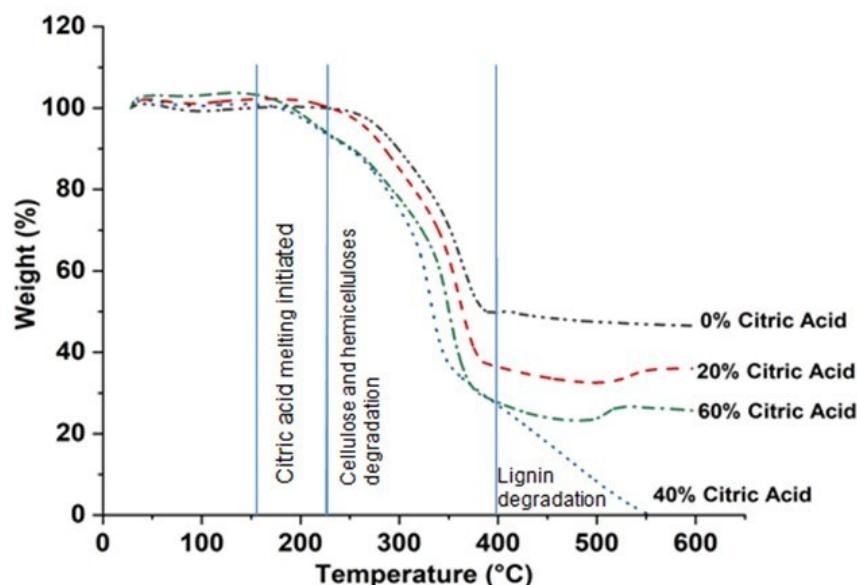


**Fig. 4.** Possible crosslinking between cellulose units in jute stick and citric acid through ester linkage formation

### TG Analysis

TG analysis of jute sticks cross-banded boards treated with different concentrations of CA was carried out, as presented in Fig. 5. It has been reported that pure CA degrades sharply in one step, centered at  $214\text{ }^{\circ}\text{C}$  (Tisserat *et al.* 2012); while in the present research work, CA-treated boards showed a more gradual weight loss profile, confirming proper ester linkage between CA and jute stick components. The degradation of raw jute sticks (0% citric acid) started at  $240\text{ }^{\circ}\text{C}$ , while the CA-treated boards initiated their weight losses at lower temperatures, indicating altered thermal stability due to chemical modification. Also, increasing CA concentration resulted in increased total weight loss, which most likely occurred because of the presence of thermally labile residues of CA and its dehydrated skeleton, which started to degrade at around  $160\text{ }^{\circ}\text{C}$  and extended degradation

over a wider temperature range when compared to pure CA (Reda 2011; Tisserat *et al.* 2012). Generally, the largest mass losses were observed within 220 to 400 °C, in line with the thermal degradations of cellulose and hemicelluloses (Parveen *et al.* 2011). Beyond 400 °C, raw jute stick remained quite stable; however, the sample treated with 40 wt% CA showed a pronounced and continuous mass loss up to ~550 °C. Such an extended degradation phase is attributed to lignin degradation, which generally takes place within a broad temperature range above 400 °C (Williams and Besler 1993; Raveendran *et al.* 1996; Min *et al.* 2007; Yang *et al.* 2007). Overall, the 0% citric acid sample maintained higher residual weight at this stage, indicating greater thermal stability, whereas 20 to 60% CA specimens showed accelerated decomposition and reduced thermal stability. These results confirmed that CA modified the thermal behavior of jute stick boards through esterification and increased polymeric interactions.



**Fig. 5.** TGA of jute stick cross banded board as a function of the concentrations of CA

## CONCLUSIONS

1. Cross-banded jute stick boards impregnated with citric acid (CA) were successfully fabricated by optimizing the CA concentrations and pressing temperatures. The optimal manufacturing conditions were determined to be a pressing temperature of 200 °C for 10 minutes using 40 wt% CA. Under these conditions, the board exhibited a modulus of rupture (MOR) of 53.6 N/mm<sup>2</sup>, a modulus of elasticity (MOE) of 8260 N/mm<sup>2</sup>, and an internal bond (IB) strength of 0.52 N/mm<sup>2</sup>.
2. The enhanced mechanical performance is attributed to the effective reaction between the carboxyl groups of citric acid and the hydroxyl-containing components of the jute stick, which promoted strong interfacial bonding within the cross-banded structure.
3. This paper proposed a simplified manufacturing approach and an alternative, formaldehyde-free route to manufacture lignocellulosic composites. Furthermore, it provided a sustainable pathway for furniture, partition panels, and other eco-friendly packaging applications.

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