



## Original Research Article

# Development of Biofoam Trays from Cassava Starch Blended with Citric Acid-modified Starch and Sugarcane Bagasse Cellulose Fiber

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### ABSTRACT

Cassava starch, regarded as a major biopolymer of Thailand, has high potential as a raw material for producing biodegradable packaging. Cassava starch foam has undesirable properties for food packaging application including high water absorption as well as low strength and elasticity. This work developed and improved physical and mechanical properties as well as water resistance of cassava starch based foam trays by blending with 50% citric acid-modified starch and 0-15% sugarcane bagasse cellulose fiber by weight of starch. The higher viscosity of batter caused by the addition of citric acid-modified starch and cellulose fiber resulted in less expansion ability of starch foam and greater amount of batters required for baking foams. The foams containing 50% citric acid-modified starch or 5-10% sugarcane bagasse fiber by weight of starch exhibited significantly improved compressive strength (0.86 MPa and 0.47-0.74 MPa, respectively as compared to 0.27 MPa for native cassava starch foam) and modulus (1.95 MPa and 1.74-1.89 MPa, respectively as compared to 1.13 MPa for native cassava starch foam) but lesser water absorption capacity (2.54 g/g sample and 2.52-2.63 g/g sample, respectively as compared to 3.07 g/g sample from native cassava starch). The physical (i.e. density and color) and mechanical properties (i.e. compressive strength and modulus) of foam trays from cassava starch containing 50% citric acid-modified starch or 10% sugarcane bagasse fiber were comparable to commercial foam trays from cassava starch blended with fiber.

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### INTRODUCTION

Cassava starch, regarded as a major biopolymer in Thailand, has high potential as a raw material for producing biodegradable

materials. Cassava starch-based foams, similar to those made from other starches, has undesirable properties including high water absorption, less strength and elasticity, which limit their applications, especially for food packaging (Kaisangsri et al., 2012; Pornsuksomboon et al., 2016). Various methods have been

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applied to overcome their drawbacks including additions of plasticizers, chemically modified starch (Kaewtatip *et al.*, 2014; Pornsuksomboon *et al.*, 2016) as well as other biodegradable polymers (Kaisangsri *et al.*, 2014).

Mechanical property and water sensitivity of cassava starch based foams was significantly affected by functional groups of derivatives and crosslinking degree (Kaewtatip *et al.*, 2014). Besides chemical crosslinking agents available for starch modification, citric acid, an organic and non-toxic acid, has been proven to be effective as crosslinking agent (Ma *et al.*, 2009). Citric acid with three carboxyl groups can react with hydroxyl groups of starch to form ester linkages. The effects of citric acid-modified starch on improvement of water resistance of baked cassava starch foam have been reported by Pornsuksomboon *et al.* (2016). However, citric acid-modified starch prepared by dry heat method on starch foam characteristics has not been reported elsewhere.

The addition of reinforcing agents to prepare starch-based composite materials has been widely applied. These include natural ligno-cellulosic fibers such as sugarcane bagasse (Vercelhez *et al.*, 2012), malt bagasse (Mello & Mali, 2014), eucalypt cellulose pulp (Schmidt & Laurindo, 2010), kraft (Kaisangsri *et al.*, 2012; 2014), jute and flax (Soykeabkeaw *et al.*, 2004), grape stalk (Engel *et al.*, 2019), wood fiber (Vargas-Torres *et al.*, 2017), wheat and cotton (Bénézet *et al.*, 2012) as well as other pulps. Although their reinforcing effects on properties of starch foams have been previously studied (Vercelhez *et al.*, 2012; Mello & Mali, 2014), various natural fibers were mainly incorporated in natural forms or ground particles without any pretreatments (i.e. alkaline treatment and bleaching).

This work aimed to improve physical and mechanical properties as well as water resistance of cassava starch based foam trays by blending with citric acid-modified starch prepared by dry heat method and sugarcane bagasse cellulose fiber for food packaging application.

## MATERIALS AND METHODS

### Materials and chemicals

Native cassava starch containing 10% moisture was obtained from Chorchaiwat Industry Co., Ltd. (Chon Buri, Thailand). Sugarcane bagasse with 40-50% moisture provided by Eastern Sugar and Cane Co., Ltd. (Sa Kaeo, Thailand) was dried in an oven at 60 °C and sieved through a 15 mesh screen to remove fine powder. Glycerol was purchased from Vidhyasom (Bangkok, Thailand). All other chemicals used in this study were of food grade.

### Preparation of citric acid-modified starch

Citric acid-modified starch was prepared from native cassava starch according to the method of Chotineeranat *et al.* (2018). Cassava starch (50 g) was mixed with 10% citric acid solution (15 mL) previously adjusted to pH 5.5. The mixture was kept at 4 °C overnight for moisture equilibration, dried at 50 °C to obtain less than 10% moisture content, ground and sieved through a 100 mesh. The mixture was further heated at 130 °C for 9 h in an oven, washed 3 times with water and precipitated before drying at 50 °C. The degree of esterification and crosslinking was calculated according to the following equations, respectively (Menzel *et al.*, 2013; Mei *et al.*, 2015).

$$\text{Degree of esterification} = (162 \times W) / [(100 \times M) - (M-1) \times W]$$

W = (% by weight of substituent)

$$= \text{bound citrate (g)} / [\text{sample (g)} - \text{bound citrate (g)}] \times 100$$

M = molecular weight of citric acid (= 175.1 g/mole)

Di esterified citric acid = Total esterified citric acid - Mono esterified citric acid

### Extraction of cellulose fiber from sugarcane bagasse

Sugarcane bagasse was subjected to an alkaline treatment using 25% NaOH (by dried weight of fiber) at fiber to liquor ratio of 1:9 at 165 °C for 2 h in a pulping unit (Model L+VVP1 229/300 152, Hato-tuote oy, Finland). The extracted cellulose fiber was thoroughly washed with water to neutral pH. Chemical compositions of the fiber including alpha-cellulose (T203 cm-22 and T264 om-22), hemicellulose, lignin (T222 om-21), extractives which are solvent-soluble and non-volatile materials in pulp (T204 cm-17 and T264 om-22) and ash were analyzed according to TAPPI standard test methods. Experiments were carried out in duplicate.

### Preparation of starch-based foam tray

Starch foam trays were prepared from various formulations as presented in Table 1 based on our previous study (unpublished data) in which native cassava starch was blended with 0-100% citric acid-modified starch (data not shown). Native cassava starch or native starch blended with citric acid-modified starch at 1:1 (50% citric acid-modified starch) was compounded with the cellulose fiber extracted from sugarcane bagasse ranging from 0 to 15 g/100 g starch. Pregelatinized cassava starch was added at 2.5 g/100 g total starch to prevent starch sedimentation. Water contents in formulations containing cellulose fiber were added up to obtain similar ratios between solids and water in order to prepare homogeneous batters.

All components were mixed for 10 min using a food mixer (KM201, Kenwood Limited, Havant, Hampshire, UK) and equilibrated at room temperature (25±2 °C) for 3 h. The batters were then thermo-molded into tray shapes using a compression molding machine (Service Technologyplus Co., Ltd., Thailand) operating at 170 °C for 3 min. The foam trays were removed from mold and stored at 25±2 °C and 50% relative humidity for 7 days prior to further analysis.

The batter amount of each formulation was fixed in order to fill up the mold and obtain a well-shaped tray as shown in Table 1. These values were dependent on fiber contents and batter viscosity of each formulation based on our previous study.

### Characterization of starch foam tray properties

#### Color

The color parameters of the starch foam trays were determined using a colorimeter CM-3500d (Konica Minolta Sensing Americas, Inc.). The CIE lab scales coordinating L\* (lightness) from 0 to 100, a\* from -a (greenness) to +a (redness) and b\* from -b (blueness) to +b (yellowness) were recorded. Measurements were carried out in triplicate.

### Moisture, thickness and density

The moisture contents of the starch foam trays were analyzed by drying samples in a hot air oven (Binder FD53, BINDER GmbH, Germany) at 105 °C overnight. The tray thickness was measured by a thickness gauge (Mitutoyo M-7301, Mitutoyo Corporation, Japan). Their density was calculated from the weight (g) and volume (cm<sup>3</sup>) of each sample. All measurements were performed in triplicate.

### Mechanical properties

Mechanical properties the starch foam trays were analyzed by the modified method of ASTM D1621-73 (1973) using a texture analyzer TA-XT2i (Texture Technologies Corp., Scarsdale, N.Y., USA) equipped with a cylindrical probe (25 mm diameter). The tray samples were cut to 20 x 20 mm<sup>2</sup> and subjected to compression test at 50% deformation and a test speed of 2.5 mm/sec. Measurements were carried out in 10 replicates.

### Water absorption capacity

Starch foam trays were determined for their water absorption capacity according to the modified method of ABNT NBR NM ISO 535 (1999). The tray samples were cut to 20 x 20 mm<sup>2</sup>, weighed immediately before ( $m_1$ ) and after ( $m_2$ ) immersion in distilled water (20 mL) at room temperature (25±2 °C) for 1 min. The water absorption capacity was calculated from the following equation. Measurements were performed in triplicate.

$$\text{Water absorption capacity (WAC)} = (m_2 - m_1) / m_1$$

### Statistical analysis

Mean values and standard deviations were calculated and compared by a one-way analysis of variance (ANOVA) using a Statgraphics software. Multiple comparisons of group means were computed using a Duncan's multiple range test (DMRT).

## RESULTS AND DISCUSSION

### Citric acid-modified cassava starch and cellulose fiber from sugarcane bagasse

Citric acid-modified cassava starch contained the total, mono- and di-esterified citric acid extents of 0.0065, 0.0017 and 0.0048, respectively. These degrees of esterification indicated that most esterified citric acid was cross-linked with starch molecules.

The cellulose fiber extracted from sugarcane bagasse with 51.5% yield contained 74.8% alpha cellulose, 19.9% hemicellulose, 1.7% lignin and 1.3% ash contents. These fibers were slightly brownish, bulky and can be homogenously dispersed in starch batter.

### Baked cassava starch foam trays

The foam trays from native cassava starch blended with 0, 25, 50, 75 and 100% citric acid-modified starch were previously prepared. Native cassava starch blending with 50%

citric acid-modified starch was chosen for further study according to their foam density not greater than commercial foam trays from cassava starch blended with fiber (data not shown).

The native cassava starch foam trays (N) showed smooth surface (Fig. 1) with some cracks occasionally observed. The foam trays from native starch/citric acid-modified starch blend (NC) had smooth surface without any crack but became slightly more yellowish and heavier than the native starch foam trays (Fig. 1). The viscosity of native starch/citric acid-modified starch blend batter became significantly higher (peak viscosity of native starch and citric acid-modified starch were 308 and 504 RVU, respectively, data not shown), thus more batter was required to make a complete tray. The addition of cellulose fiber also increased the batter viscosity leading to high amount of batter required as well. All starch foam trays containing cellulose fiber (NC and NC+F) also presented smooth surface with homogenously dispersed fiber (Fig.1). Their color became more yellowish progressively with increasing fiber contents as supported by increasing  $b^*$  values.

### Characteristics and properties of starch foam tray

#### Color

The color of cassava starch based foam trays were significantly affected by the addition of citric acid-modified starch and cellulose fiber contents (Table 2). Lightness ( $L^*$ ), redness ( $a^*$ ) and yellowness ( $b^*$ ) of cassava starch foam trays were in the ranges of 76-77, -0.4-1.6 and 1.4-8.0, respectively. The addition of citric acid-modified starch resulted in decreased  $L^*$  values but increased  $a^*$  and  $b^*$  values. Similar changes in color of cassava starch trays were determined with increasing cellulose fiber contents as the fiber imparted a brown color corresponding with previous research reported by Verdelhez *et al.* (2012).

#### Moisture, thickness and density

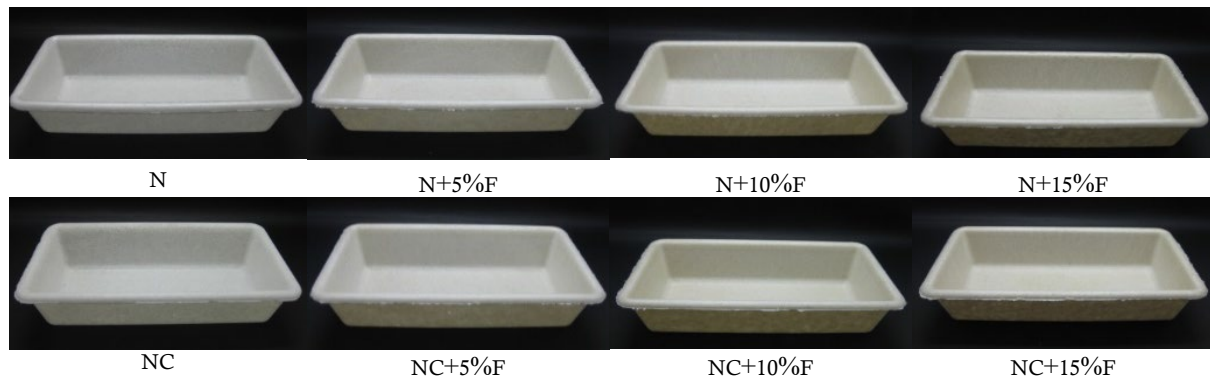
The moisture contents and thickness of the cassava foam trays containing citric acid-modified starch and cellulose fiber were not statistically different at  $p \leq 0.05$  (Table 3). All cassava starch-based foams had significantly higher moisture contents than commercial foam tray due to their hygroscopic nature derived from high number of hydrophilic hydroxyl groups (Soykeabkaew *et al.*, 2015).

The density of native cassava starch foam, however, increased significantly from 0.17 g/cm<sup>3</sup> after blending with citric acid-modified starch to 0.30 g/cm<sup>3</sup> and progressively increased with increasing fiber contents (0.21-0.27 g/cm<sup>3</sup> and 0.40-0.46 g/cm<sup>3</sup>, respectively). All cassava starch foam samples had comparable density with other starch-based foams previously reported by other researchers (Verdelhez *et al.*, 2012; Kaisangsri *et al.*, 2014; Pornsuksomboon *et al.*, 2016) as well as commercial foam tray, but considerably higher density than commercial EPS foam (0.02 g/cm<sup>3</sup>).

**Table 1.** Formulations and compositions of batters for preparing cassava starch foam trays

Samples	Compositions (g)						Batter amount (g)
	Native cassava starch	Citric acid-modified starch	Pregelatinized cassava starch	Water	Glycerol	Sugarcane bagasse fiber	
N	97.5	0	2.5	100	5	0	30
N+5%F	97.5	0	2.5	105	5	5	30
N+10%F	97.5	0	2.5	110	5	10	35
N+15%F	97.5	0	2.5	115	5	15	35
NC	48.75	48.75	2.5	100	5	0	45
NC+5%F	48.75	48.75	2.5	105	5	5	45
NC+10%F	48.75	48.75	2.5	110	5	10	47.5
NC+15%F	48.75	48.75	2.5	115	5	15	50

N = Native cassava starch, F = Fiber from sugarcane bagasse, C = Citric acid-modified starch

**Figure 1.** Appearances of foam trays from native cassava starch and native cassava starch/citric acid-modified starch blends containing sugarcane bagasse cellulose fiber**Table 2.** Color parameters of foam trays from native cassava starch and native cassava starch/citric acid-modified starch blends containing sugarcane bagasse cellulose fiber

Samples	Color parameters		
	L*	a*	b*
N	76.75 + 0.68 <sup>abc</sup>	-0.39 + 0.02 <sup>s</sup>	1.40 + 0.10 <sup>f</sup>
N+5%F	76.38 + 0.88 <sup>bcd</sup>	0.06 + 0.05 <sup>f</sup>	4.82 + 0.45 <sup>d</sup>
N+10%F	77.15 + 0.86 <sup>ab</sup>	0.61 + 0.06 <sup>c</sup>	6.13 + 0.39 <sup>c</sup>
N+15%F	77.28 + 0.67 <sup>a</sup>	1.11 + 0.07 <sup>d</sup>	7.67 + 0.27 <sup>b</sup>
NC	75.68 + 0.77 <sup>d</sup>	-0.31 + 0.04 <sup>s</sup>	2.29 + 0.18 <sup>c</sup>
NC+5%F	75.92 + 0.92 <sup>cd</sup>	0.64 + 0.06 <sup>c</sup>	6.61 + 0.31 <sup>c</sup>
NC+10%F	76.98 + 1.33 <sup>ab</sup>	1.24 + 0.13 <sup>c</sup>	7.69 + 0.59 <sup>b</sup>
NC+15%F	75.71 + 0.55 <sup>d</sup>	1.57 + 0.11 <sup>b</sup>	8.01 + 0.31 <sup>b</sup>
commercial foam tray	73.44 + 0.64 <sup>c</sup>	3.30 + 0.19 <sup>a</sup>	16.03 + 0.63 <sup>a</sup>

Different letters in the same column indicate significant differences between means ( $p \leq 0.05$ , Duncan's multiple range test).

**Table 3.** Moisture, thickness and density of foam trays from native cassava starch and native cassava starch/citric acid-modified starch blends containing sugarcane bagasse cellulose fiber

Samples	Moisture (%)	Thickness (mm)	Density (g/cm <sup>3</sup> )
N	8.43 + 0.22 <sup>a</sup>	2.58 + 0.05 <sup>a</sup>	0.17 + 0.00 <sup>b</sup>
N+5%F	8.56 + 0.17 <sup>a</sup>	2.41 + 0.04 <sup>b</sup>	0.21 + 0.00 <sup>s</sup>
N+10%F	8.47 + 0.01 <sup>a</sup>	2.36 + 0.06 <sup>b</sup>	0.25 + 0.01 <sup>f</sup>
N+15%F	8.14 + 0.09 <sup>a</sup>	2.40 + 0.06 <sup>b</sup>	0.27 + 0.01 <sup>c</sup>
NC	8.79 + 0.09 <sup>a</sup>	2.39 + 0.05 <sup>b</sup>	0.30 + 0.01 <sup>d</sup>
NC+5%F	8.75 + 0.01 <sup>a</sup>	2.36 + 0.03 <sup>b</sup>	0.40 + 0.01 <sup>c</sup>
NC+10%F	8.51 + 0.03 <sup>a</sup>	2.30 + 0.09 <sup>b</sup>	0.43 + 0.03 <sup>b</sup>
NC+15%F	8.26 + 0.1 <sup>a</sup>	2.35 + 0.06 <sup>b</sup>	0.46 + 0.02 <sup>a</sup>
commercial foam tray	7.04 + 0.08 <sup>b</sup>	2.46 + 0.02 <sup>ab</sup>	0.39 + 0.02 <sup>c</sup>

Different letters in the same column indicate significant differences between means ( $p \leq 0.05$ , Duncan's multiple range test).

Citric acid-modified starch possessed stronger molecular structure relative to native cassava starch due to its crosslinking between starch molecules by citric acid. This led to restricted mobility of starch molecules and higher swelling extent before granule rupture when heated and thus causing higher viscosity of citric acid-modified starch paste. The cellulose fiber can partially absorb water resulting in higher viscosity causing less expansion ability and porous structure of starch foams. Higher weight, resistance to swelling and expansion of cellulose fiber was responsible for increased density of cassava starch based foams (Carr *et al.*, 2006; Glen, Orts, & Nobes, 2001; Kaisangsri *et al.*, 2012; 2014). The density of the foams obtained, therefore, increased and more batter was required as well. These results are corresponding with previous works reporting that foam density was inversely proportional to expansion capability (Kaisangsri *et al.*, 2014).

### Mechanical properties

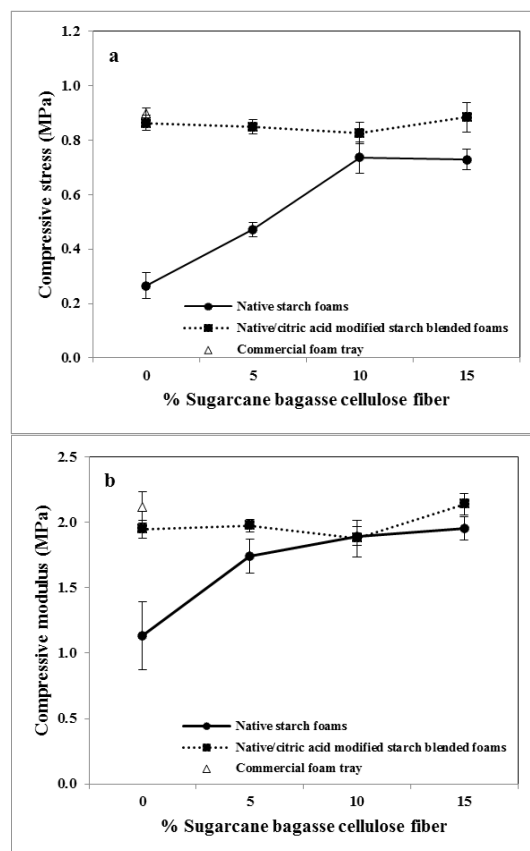
Compressive strength of the native cassava starch-based foam significantly increased from 0.27 MPa to 0.86 MPa when blended with citric acid-modified starch (Fig. 2a) indicating the strengthening effect of cross-linked starch. The compressive strength of native/citric acid-modified starch blended foam was comparable with that of commercial foam tray from cassava starch blended with fiber (0.90 MPa). The compressive strength of native starch foam was also progressively enhanced to 0.41-0.74 MPa with increasing cellulose fiber contents. The interactions between fiber and native cassava starch possibly promoted the foam strength as previously reported by Shogren *et al.* (2002).

However, this increment was not statistically significant when the fiber content increased from 10% to 15% possibly due to inhomogeneous distribution of fiber at high content (Shmidt & Laurindo, 2010). This finding was corresponding with previous work reporting that 5-10% aspen fiber apparently improved tensile strength of corn starch baked foams (Shogren *et al.*, 2002). Similar result was reported for cassava starch foam with 2-10% malt bagasse (Mello & Mali, 2014). In contrast, the fiber addition hardly affected the strength of foam trays from citric acid-modified starch blend (0.83-0.88 MPa) probably caused by inhomogeneous distribution of fiber in viscous paste of native/crosslinked cassava starch blend. Similar changes in compressive modulus indicative of foam stiffness were determined as presented in Fig. 2b.

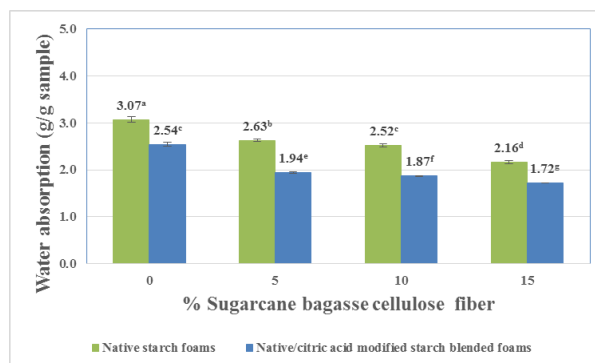
### Water absorption capacity

Water absorption capacity of cassava starch-based foam after water immersion (3.07 g/g sample) was highly suppressed by blending with citric acid-modified starch (2.54 g/g sample). These values of native starch and native/citric acid-modified starch blended foams significantly decreased to 2.16-2.63 and 1.72-1.94 g/g sample, respectively, with increasing cellulose fiber contents (Fig. 3). High number of hydroxyl groups which can strongly interact with water molecules was responsible for the highest water absorption capacity of the native starch foam (Pornsuksomboon *et al.*, 2016). The results confirmed the effects of both citric acid-modified starch and cellulose fiber on reducing water absorption of cassava starch foam which could be explained by less hydrophilic characteristics of cross-linked starch and

cellulose fiber. Citric acid-modified starch possessed lower number of hydroxyl groups as a consequence of crosslinking with citric acid (Ma *et al.*, 2009), while cellulose, a major component of fiber, contained higher crystallinity than native starch (Bénézet *et al.*, 2012; Mali *et al.*, 2010).



**Figure 2.** Compressive stress (a) and modulus (b) of foam trays from native cassava starch and native cassava starch/citric acid-modified starch blends containing sugarcane bagasse cellulose fiber



**Figure 3.** Water absorption capacity of foam trays from native cassava starch and native cassava starch/citric acid-modified starch blends containing sugarcane bagasse cellulose fiber

## CONCLUSIONS

Biodegradable starch-based foam trays were produced from cassava starch and sugarcane bagasse, raw materials economically important in Thailand, with improved mechanical and water

resistant characteristics by baking process. Cassava starch foam trays containing 50% citric acid-modified starch or 10% cellulose fiber extracted from sugarcane bagasse possessed highly improved mechanical properties and water resistance. Citric acid-modified starch highly contributed to enhanced mechanical properties, while cellulose fiber from sugarcane bagasse was responsible for both mechanical properties and water absorption capacity of cassava starch-based foam trays.

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