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Biofilm and Biocomposite Film prepared from Durian Rind and Pineapple Leaf: Synthesis and Characterization

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Abstract

In this study, biocomposite films were prepared from cellulose fibers of agricultural residues, durian rind, and pineapple leaf, as an alternative to plastic films. Carboxymethyl cellulose (*CMC*) derived from durian rind was synthesized to produce a biocomposite film matrix using different chemical compositions. FTIR analysis of the film matrix showed that the degree of carboxymethyl substitution was not significantly affected by changes in the chemical compositions. For the pineapple cellulose, the steam explosion was combined with alkaline treatment followed by acid hydrolysis then subjected to high shear forces to produce cellulose fibers for the reinforcing material. The biocomposite films were prepared by solution casting at reinforcing fiber concentrations between 0-45 wt.%. SEM studies showed that the fibers were well distributed in the CMC matrix up to a concentration of 45 wt.% due to good compatibility between the two phases. Mechanical testing indicated a 40*%* increase in tensile strength of the biocomposite film containing 30 wt.*%* fiber due to good adhesive force between the film matrix and the reinforcing fibers. Biodegradation experiments showed that the films broke down completely within 18-24 h, demonstrating the film's potential as an environmentally friendly material. This research aligns with Bio-Circular-Green Economic model promoted by the Thai government.

Keywords: Cellulose; Carboxymethyl cellulose; Biocomposite; Durian Rind; Pineapple Leaf.

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1. Introduction

Biofilms and biocomposite films have recently been in the interest of various industries especially in the paper, pharmaceutical, plastic, and packaging manufacturing.[1-4] By using biodegradable materials, environmental pollution from plastic waste could be alleviated. In addition to the biodegradability of these agro-wastes, there are several other benefits in their use including value-adding to scrap, reducing waste, and lowering raw material costs. Consequently, this can lead to a Bio-Circular-Green Economic Model or BCG model, which aligns well with the Thai government's economic goals. There are many sources of biomaterials and for an agricultural country like Thailand, post-harvest residues such as rice straw, pineapple leaf, and corn cop, are abundant. Woods and plants are composed of three main components, *i.e.* cellulose, hemicellulose, and lignin, and the amounts of these components depend on the type of plant; however, most agricultural plants and residues comprise 30-40% cellulose, 10-30% hemicellulose, and 10-20% lignin.[2,5,6] These compounds can be utilized to produce biomaterials and bio-based chemicals.[3,7,8]

Cellulose, which is a natural linear polysaccharide of (1→4)-linked β-d-glucopyranose units which are aggregated to form a highly ordered structure, is the main component in crops. It is biodegradable, biocompatible, environmentally friendly, lightweight, and inexpensive. It also has superior mechanical properties with a high aspect ratio. However, its drawbacks include being water-insoluble and it does not melt.[4,5] Cellulose also slightly dissolves in some solvents and is thermally degraded at high temperatures which limits its fabrication process and applications. It is usually used as a reinforcing material.[9,10] Senthilkumar *et al.* 2022[11] used pulverized pineapple leaf fiber as a reinforcing material in an epoxy matrix and reported a strong fiber and matrix bonding that increased in impact strength and flexural properties of the

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composites. With alkaline treatment, the pineapple fiber surface was rough causing fibrillation in which the bonding strength with the matrix should be increased; however, the compression strength was inferior compared to the untreated fibers due to agglomeration of the high loadings of fibers.[12] Chawalitsakunchai S *et al.* 2021[13] prepared nanocellulose from pineapple leaf via acid hydrolysis and used it as a reinforcing agent in a natural rubber matrix. They reported that with 2 parts of the pineapple leaf nano cellulose per hundred parts of rubber (phr), tensile strength increased by 17%; however, with higher amounts of the cellulose, agglomeration was observed, resulting in reduced mechanical properties. Fiber agglomeration at high concentrations was also observed when durian fibers were used to reinforce high-density polyethylene composites[14] and with pineapple leaf nanofibers reinforcing polyurethane composites[4]. Instead of using strong acids for cellulose digestion, Ravindran *et al.* 2019[15] attempted to use lime juice as an alternative. Using a ball milling technique, high crystallinity and thermally stable nanocellulose from pineapple leaf were achieved. A steam explosion has also been found to be an efficient technique in nano cellulose preparation.[16,17] Besides being used as a reinforcing material, cellulose can also be used for other applications, such as a superabsorbent,[18] Pickering emulsion stabilizer[19] and as pulp and paper.[20]

Carboxymethyl cellulose (CMC) is a cellulose derivative with carboxymethyl groups bound to some of the hydroxyl groups of the glucopyranose monomers that make up the cellulose backbone. CMC is advantageous to cellulose as it is water soluble, facilitating the film and composite fabrication and extending its applicability. It can be synthesized by an alkalization process followed by an etherification process as shown in Fig. 1.[21] Aside from conventional heating usually utilized in the synthesis of CMC, other techniques have also been investigated including steam explosion[22] and microwave heating.[21] While some research studies reported an increase in the degree of carboxymethyl substitution as well as CMC yield with increasing concentrations of sodium hydroxy (NaOH) used in the CMC synthesis,[7,21] some found the opposite effect.[23] Nevertheless, it was agreed that CMC has less crystallinity than that cellulose, making it more soluble in water and solvents.

Thailand is the largest durian producer in Southeast Asia, growing as high as 1.1 million tons in 2020.[24] Only one-fourth to one-third of durian fruit is edible and around 80% of the non-edible part is the rind.[20] Pineapples are also one of the popular tropical fruits that are grown widely in Thailand. In 2020, it was reported that Thailand planted 1.8 million tons of pineapples.[24] One shoot of pineapple has about 40-50 fibrous leaves.[25] With these large amounts of waste in the form of durian rind and pineapple leaf, this research was interested to produce biofilms and biocomposite films purely using cellulose from these two raw materials. The matrix of the film was obtained from CMC synthesized from durian rind while the reinforcing cellulose fibers were from the pineapple leaf. The chemical functional groups, morphology, mechanical properties as well as biodegradability of the cellulose fibers, CMC, and films were investigated. The objective of this work is to affiliate with the Thai government's strategic BCG model. Bioeconomy is achieved by converting these agricultural wastes into valuable materials like biofilms and biocomposite films. The green economy is attained by replacing harmful plastic films with these more environmentally friendly films and a circular economy is realized by the use or recycling of these waste products. Biodegradable biocomposite films are, therefore, a very promising alternative to synthetic plastic films.

2. Materials and methods

2.1 Materials

Mon Thong durian rinds from Rayong, Thailand, was collected from a durian seller in Chonburi, Thailand. Pineapple leaves were obtained from Smooth Cayenne Pineapple farm in Sriracha, Chonburi, Thailand. All the chemicals used in this research were analytical reagent (AR) grade. 30% w/v hydrogen peroxide (H_2O_2), 99% purity isopropanol, 98% purity sulfuric acid (H_2SO_4), and 99.5% purity glycerol were supplied by QRëC^{TM}, New Zealand. 99% purity sodium hydroxide (NaOH) was purchased from Pine Chemical Co., Ltd., India, and 99.7% purity acetic acid was

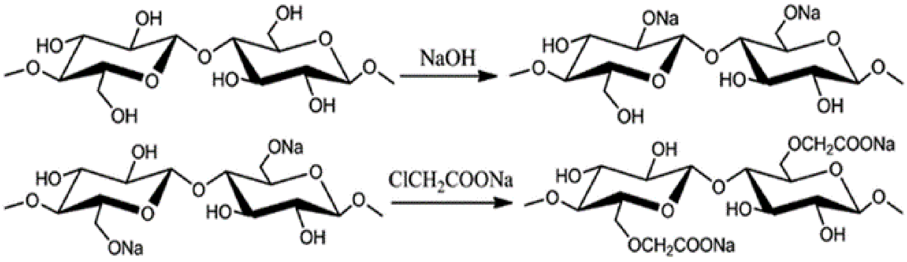

Fig. 1 Carboxymethylation of cellulose. Reproduced with permission from [21].

obtained from Loba Chemie Pvt Ltd., India. Monochloroacetic acid was supplied by S D Fine-Chem Ltd., India, and sodium carbonate (Na_2CO_3) was purchased from KEMAUS, Australia.

2.2 Durian rind cellulose extraction

Durian rinds were cleaned with water, sun-dried, chopped into small pieces, and oven dried at 60 °C overnight. The dry rinds were then ground into a powder with an approximate size range of 0.1-2 mm using a plastic grinder. The alkaline treatment was performed by mixing durian rind powder with 10% w/v NaOH at a ratio of 1 g:10 mL and the mixture was mixed using a magnetic stirrer bar while being heated at 70 °C for 6 h. The black slurry was then filtered out of the mixture using a cheesecloth and the cellulose fibers were washed multiple times with deionized water until a pH of 7 was attained. The fibers were oven dried at 60 °C for 3 h. The delignification process was followed by bleaching using H_2O_2. 10 mL of 30% w/v H_2O_2 was added into 1 g of cellulose fibers and the mixture was heated at 70 °C for 3 h and stirred using a magnetic stirrer bar. The bleached fibers were filtered using a cheesecloth and washed thoroughly until a pH of 7 was reached. The cellulose fibers were again oven-dried at 60 °C for 3 h. Finally, a Mara MR-3339 food processor was used to separate and grind the fibers into powder form. The cellulose powder was sieved into sizes below 100 µm and the cellulose percent yield ($\%Y_c$) was calculated using the weight of extracted cellulose (w_c) and the weight of dried durian rind (w_d) as shown in Eq. (1). The experimental procedure was adopted from Khemkaew and Kaewpirom 2016.[26]

$$\%Y_c = \frac{w_c}{w_d} \times 100 \tag{1}$$

2.3 Carboxymethyl cellulose (CMC) synthesis

As cellulose is insoluble in water, carboxymethyl cellulose which is a water-based material was derived from the durian rind extracted cellulose. 3 g of cellulose was mixed with 10 mL of 30% w/v NaOH and 90 mL of isopropanol and left at room temperature for 30 min. Then, 3.6 g of monochloroacetic acid was added to the mixture and the mixture was stirred using a magnetic stirrer bar at room temperature for 2 h. Aluminum foil was then used to cover the mixture beaker to prevent solvent evaporation prior to leaving the mixture in an oven at 60 °C for 3 h. The mixture was then separated into two layers; a clear solution on the top layer and an opaque layer at the bottom. The top layer was discarded while the bottom layer was then washed with 50 mL of 70% v/v ethanol and neutralized with acetic acid until a pH of 7 was achieved. The suspension was then filtered and washed with ethanol three more times before the solid residues were oven dried at 60 °C overnight. Fig. 2 illustrated the flowchart of the durian rind cellulose extraction (a) and the CMC preparation (b). The percent yield of CMC ($\%Y_{CMC}$) was calculated from the weight of obtained CMC (w_{CMC}) and the weight of cellulose (w_c) as shown in Eq. (2). The experimental procedure was adopted from Rachtanapun *et al.* 2012.[23]

$$\%Y_{CMC} = \frac{w_{CMC}}{w_c} \times 100 \tag{2}$$

CMC is the derivative of cellulose where hydroxyl groups are replaced by carboxymethyl groups and hence, the degree of substitution is expected to be dependent on the number of chemicals used in the alkalization and etherification processes. In order to study the effects of the cellulose-chemicals ratio on the degree of substitution, another two sets of CMC synthesis experiments were carried out, one with 20% more chemicals and the other with 20% fewer chemicals. Table 1 shows the mixture compositions used for CMC synthesis.

Table 1. Chemical compositions used for CMC synthesis.

Chemicals	Benchmark	+20%	-20%
Durian rind cellulose (g)	3	3	3
30% w/v sodium hydroxide (mL)	10	12	8
Isopropanol (mL)	90	108	72
Monochloroacetic acid (g)	3.6	4.3	2.9

2.4 Pineapple cellulose preparation

The pineapple leaf fibers were extracted using a water-retting extraction technique followed by chemical reactions. The leaves were cut to 20 cm lengths and soaked in water at room temperature for 14 days. After 14 days, the fibers were separated by hand from the soaked pulp, rinsed thoroughly with water, and sun-dried for two days. Steam explosion of the fibers from the pineapple leaves was slightly modified from the method used by Cherian *et al.* 2010.[16] The fibers were chopped to 10 cm lengths and 100 mL of 5% w/w NaOH was mixed with 10 g of the fibers. The mixture went under an autoclave process at 125 °C for 15 min. The fibers were washed with deionized water until a pH of 7 was attained and then filtered using a cheesecloth. The samples were then oven-dried at 90 °C for 30 min. Next, the delignification process was carried out using 50% w/v H_2O_2. 6 g of steam-exploded fibers were bleached with 100 mL H_2O_2 at 80 °C for 2 h. The fibers were subsequently washed with deionized water until pH 7 and filtered using a cheesecloth. The samples were oven dried at 90 °C for 30 min and sieved to obtain sizes less than 300 µm. The fibers were further digested using 64% w/w H_2SO_4. 100 mL of H_2SO_4 was added to 5 g of bleached fibers and heated to 50 °C for 30 min. The mixture was then washed thoroughly with deionized water until reaching pH 7 and

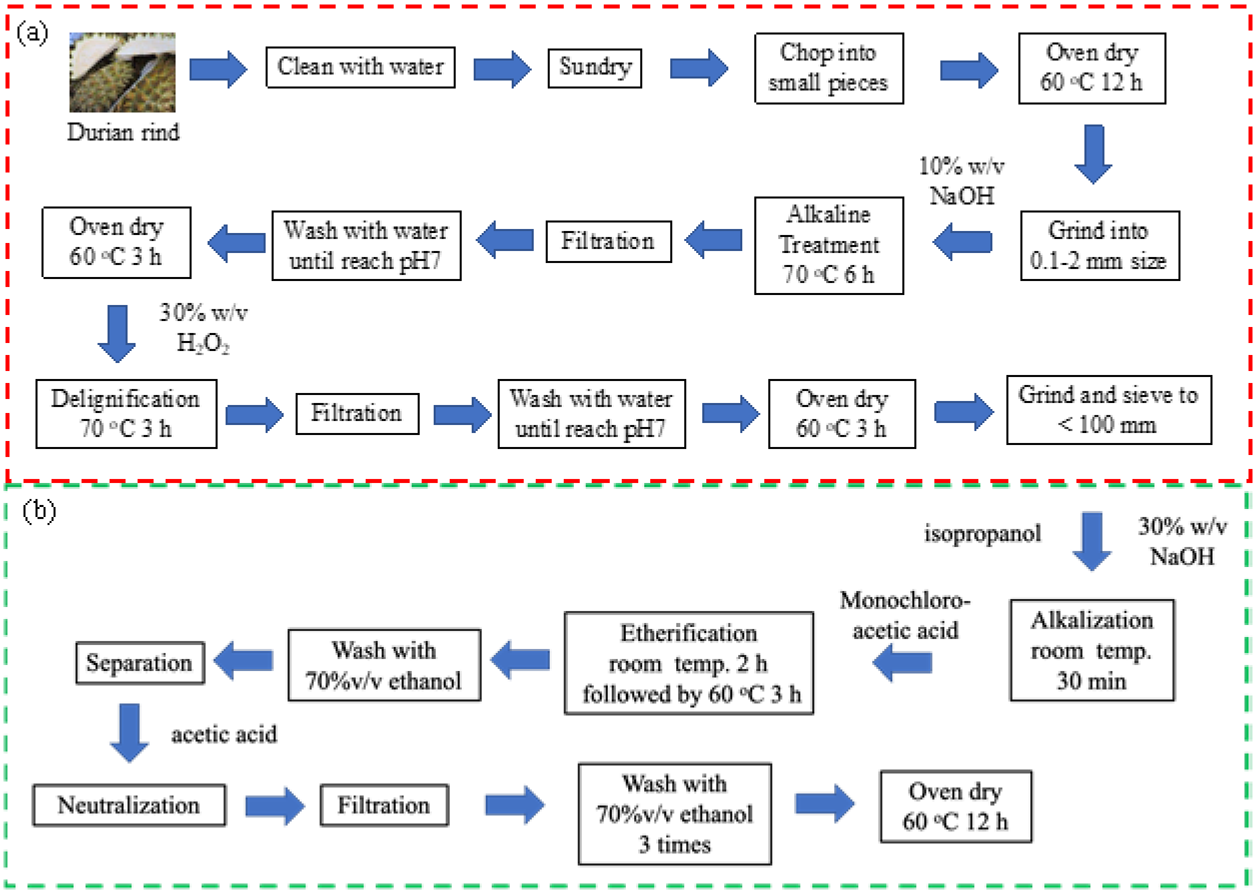

Fig. 2 Durian rind cellulose extraction (a) followed by CMC preparation (b).

filtered using filter paper. The fibers were again oven dried at 90 °C for 30 min and sieved to obtain sizes less than 100 µm. Three different pineapple cellulose fiber concentrations in distilled water were prepared at 1, 3, and 6% w/v, and a high-speed food processor was used to blend the suspensions for 2-3 h to obtain homogeneous solutions. However, it was observed that the solutions obtained from cellulose fiber concentrations of 1 and 3% w/v showed sedimentation of the fibers during storage and hence, they were discarded. This may be due to the low fiber concentrations which had fewer chances to be cut by the food processor blades to form fine fibers. Only the cellulose fiber concentration at 6% w/v was stable and used for further experiments. The pineapple cellulose fiber preparation flowchart is illustrated in Fig. 3.

2.5 Biofilm and biocomposite film preparation

Approximately 3 g of CMC obtained from the benchmark formula was dissolved in a calculated amount of distilled water as shown in Table 2 at 80 °C using a magnetic stirrer for 10 min. The solution was left to cool down to room temperature. For the blank test, only glycerol at a composition of 30% w/w of the weight of CMC, equivalent to 0.9 g of glycerol, was added to the solution. For the cellulose fiber reinforced film, the 6% w/v pineapple cellulose fiber solution prepared in Section 2.4 was added at the weight concentration of cellulose fibers of 15, 30, and 45 wt.% of the weight of CMC. Table 2 illustrates the biofilm and biocomposite film compositions. The solution preparation started by dissolving CMC in distilled water at 80 °C using a magnetic stirrer for 10 min. After the solution was left to cool down to room temperature, glycerol, and pineapple cellulose fiber solution were added and mixed well to form a homogeneous solution. 100 g of each composition was used to cast the film using a doctor blade film coater at 2000 µm thickness on an acrylic sheet. The acrylic sheet was oven dried at 100 °C for 4 h after which the film was removed from the sheet and kept in a zip-lock bag for further characterization.

2.6 FTIR characterization

A Thermo Scientific Nicolet iS50 FT-IR, USA, equipped with a horizontal Smart ARK flat ZnSe crystal cell was used to carry out FTIR investigation according to ASTM E1421. The crystal window where the sample was placed allowed 12 reflections per measurement, enhancing consistency and accuracy. The spectra were acquired over the range of 4000-650 cm^{-1} with a resolution of 4 cm^{-1} for 32 scans against a background spectrum recorded from the clean empty cell. The operations were done at a room temperature of 21 °C. Before

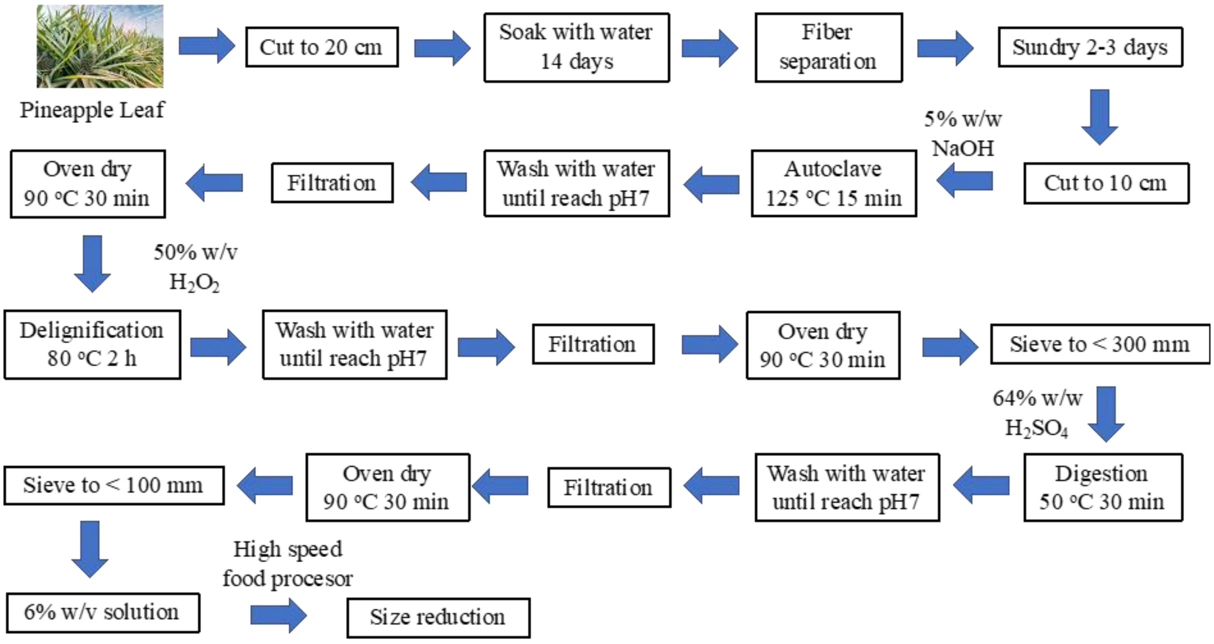

Fig. 3 Pineapple cellulose fiber preparation flowchart.

Table 2. Biofilm and biocomposite film compositions.

Films	CMC (g)	Glycerol (g)	Cellulose fiber solution (mL)	Distilled water (mL)
0 wt.% fiber (neat film)	3	0.9	0 (equivalent to 0 g fiber)	100
15 wt.% fiber	3	0.9	7.5 (equivalent to 0.45 g fibers)	92.5
30 wt.% fiber	3	0.9	15 (equivalent to 0.9 g fibers)	85
45 wt.% fiber	3	0.9	22.5 (equivalent to 13.5 g	77.5

analysis, 2 mg of the sample was mixed with 100-200 mg of dry potassium bromide (KBr). The mixture was well-ground and pressed by a hydraulic press to form a clear disc.

2.7 Film thickness and film color

A Mitutoyo Digital Indicator Thickness Gauge with a resolution of 0.01 mm was used to measure the film thickness. The measurement was done with three films of each composition and five positions for each film to get average and deviation values. Film colors in terms of L*, a*, and b* values as well as whiteness index (WI) and yellowness index (YI) were analyzed using an UltraScan XE, HunterLab, Virginia, USA. It is a dual-beam xenon flash spectrophotometer with a wavelength range from 360 to 750 nm. The test was performed in total transmission mode with an area viewport size of 1 inch.

2.8 SEM characterization

The morphology and distribution of the pineapple leaf cellulose fibers in the CMC matrix were investigated using Scanning Electron Microscopy (SEM). The analysis was performed using an LEO 1450 VP operated at 15 kV. A Polaron Range Model SC 7620 Ion Sputter Coater was used for the gold sputtering of the samples.

2.9 Mechanical properties of the films

A TA.XT plusC Texture Analyzer, Stable Micro Systems Ltd., UK was used to characterize the mechanical properties of the films according to ASTM D882-02. The biofilms and biocomposite films were cut into 1×10 cm^2 pieces. Eight to ten films were tested for each sample to obtain an average value and standard deviation. The test was performed in tension mode using a test speed of 10 mm/s to obtain tensile strength and percent elongation at break.

2.10 Photodegradation and biodegradation of the films

Photodegradation of the films was studied under LED lightbulbs with a luminous flux of 4500 Lm at wavelengths of 400-800 nm. The lightbulbs were attached to the inside lid of a carton box. Nine films of 1×5 cm^2 sizes for each composition were placed inside the box. A thermocouple was also placed inside the box to monitor the temperature within the box. The physical appearances of the films were observed daily for 3 consecutive days.

Biodegradation of the films was conducted to imitate the decomposition in a landfill. Soil with the moisture of 60% RH was added into a plastic plant pot at a thickness of 4 cm. Three pieces of the film of size 1×5 cm^2 for each composition were placed on the soil. A plastic mesh was placed on top of the films and more soil was added to a total thickness of 12 cm, equivalent to the films being buried at a depth of 8 cm from the soil surface. The film appearances were monitored every 6 h up to 24 h.

3. Results and discussions

3.1 Cellulose and CMC Yields

It has been reported that most plants are composed of 30-50% cellulose (with some containing up to 70-90% such as Ramie or Chinese grass, pineapple leaf, and cotton), 20-40% hemicellulose, and 10-30% lignin.[2,5,6,27] Masrol *et al.* 2015[20] and Penjumras *et al.* 2014[28] reported similar cellulose contents of approximately 31-33% in durian rind while higher contents of 47 and 57% were reported by Charoenvai 2014[14] and Lubis *et al.* 2018[27] respectively. This research study used NaOH to extract cellulose from durian rind and H_2O_2 to delignify the cellulose and it was found that the yield of the extracted cellulose from durian rinds was 18.26±3.45%. Similar processes were applied to extract the cellulose from pineapple leaf in this work along with a steam explosion in an autoclave which resulted in cellulose yields of approximately 59.81±4.24%, which is lower than those obtained by Cherian *et al.* 2011[4] and Tanpichai and Witayakran 2016[25] who reported yields of 76-78%. Once our bleached fibers were further digested using H_2SO_4, the yields reduced slightly to 57.65±3.98%. Different cellulose contents which lead to different mechanical and thermal properties were found in different pineapple cultivars, planting climatic conditions, and fiber extraction processes.[9,29]

Once the cellulose from the durian rind underwent the carboxymethylation process using sodium mono chloroacetate acid as a reagent to form CMC, the sodium carboxymethyl groups replaced the hydroxyl groups in the cellulose, causing the weights of CMC to increase. The CMC yields were in the range of 133-155%, depending on the degree of substitution, and agreed with those reported by Rachtanapun *et al.* 2012.[23] It was observed that the CMC yield from the benchmark chemical composition used in the carboxymethylation process was comparable to that of the reduced formula (*i.e.* -20%). When the chemical compositions were increased by 20% (*i.e.* +20%), the CMC yield increased by almost 20%. However, results from a further investigation which will be shown later, found that there were no significant advantages gained from CMC extracted from the +20% formula. Consequently, the benchmark formula was chosen for biocomposite film preparation. Table 3 summarizes the yields achieved in this study.

Table 3. The percent yield of extracted cellulose and CMC.

Materials	Percent yield (%)
durian rind extracted cellulose via alkaline treatment	18.26±3.45
pineapple leaf extracted cellulose via alkaline treatment	59.81±4.24
pineapple leaf extracted cellulose after acid hydrolysis	57.65±3.98
CMC	
Benchmark	133.20±8.68
+20%	155.32±8.51
-20%	138.41±1.20

3.2 FTIR studies

The functional groups of cellulose extracted from durian rind and pineapple leaf, CMC, and the composite film were analyzed using FTIR as shown in Fig. 4. The FTIR spectra of durian rind cellulose (Fig. 4(a)) show a broad band between 3200-3600 cm^{-1} with a peak at 3340 cm^{-1} corresponding to -OH stretching from hydroxyl groups. The C-H stretching from polysaccharide groups occurs at 2902 cm^{-1} and the C=O stretching of carbonyl groups is shown at 1596 cm^{-1}. The band at 1166 cm^{-1} was due to the ether group (-O- stretching). The peak at 2360 cm^{-1} demonstrated the presence of C=O, resulting from the CO_2 adsorption of cellulose from the atmosphere.[8,18] Similar spectra bands for the cellulose extracted from durian rind were reported in various studies.[3,14,23,27,28] The extracted cellulose from pineapple leaf also showed a similar FTIR spectra profile (Fig. 4(b)) to that of the durian rind cellulose and is in agreement with results reported by Balakrishnan *et al.* 2017[17] and Chen *et al.* 2021.[19] After alkalization and etherification of the cellulose, the hydroxyl groups of cellulose were replaced by carboxymethyl groups, resulting in a dramatic decrease in the -OH spectra at 3200-3600 cm^{-1} and C-H spectra at 2800-3000 cm^{-1}. Instead, spectral bands representing the carboxyl groups (COO-) and their salts were observed at approximately 1400-1600 cm^{-1}. These findings were similar to the findings by Golbaghi *et al.* 2017[22] who prepared CMC from sugarcane bagasse and Kaewprachu *et al.* 2022[7] who synthesized CMC from Palmyra palm fruit husk. In addition, different NaOH concentrations used for carboxymethyl substitution did not show any noticeable differences in the FTIR spectra (Figs 4(c)-(e)), in agreement with the studies of Rachtanapun *et al.* 2012[23] and Kaewprachu *et al.* 2022.[7] As a result, it was expected that the differences in the degree of substitution were not crucial and hence, the benchmark chemical composition was used for further experiments for biocomposite film preparation. FTIR spectra bands of the composite film with 45 wt.% pineapple leaf cellulose as shown in Fig. 4(f), were

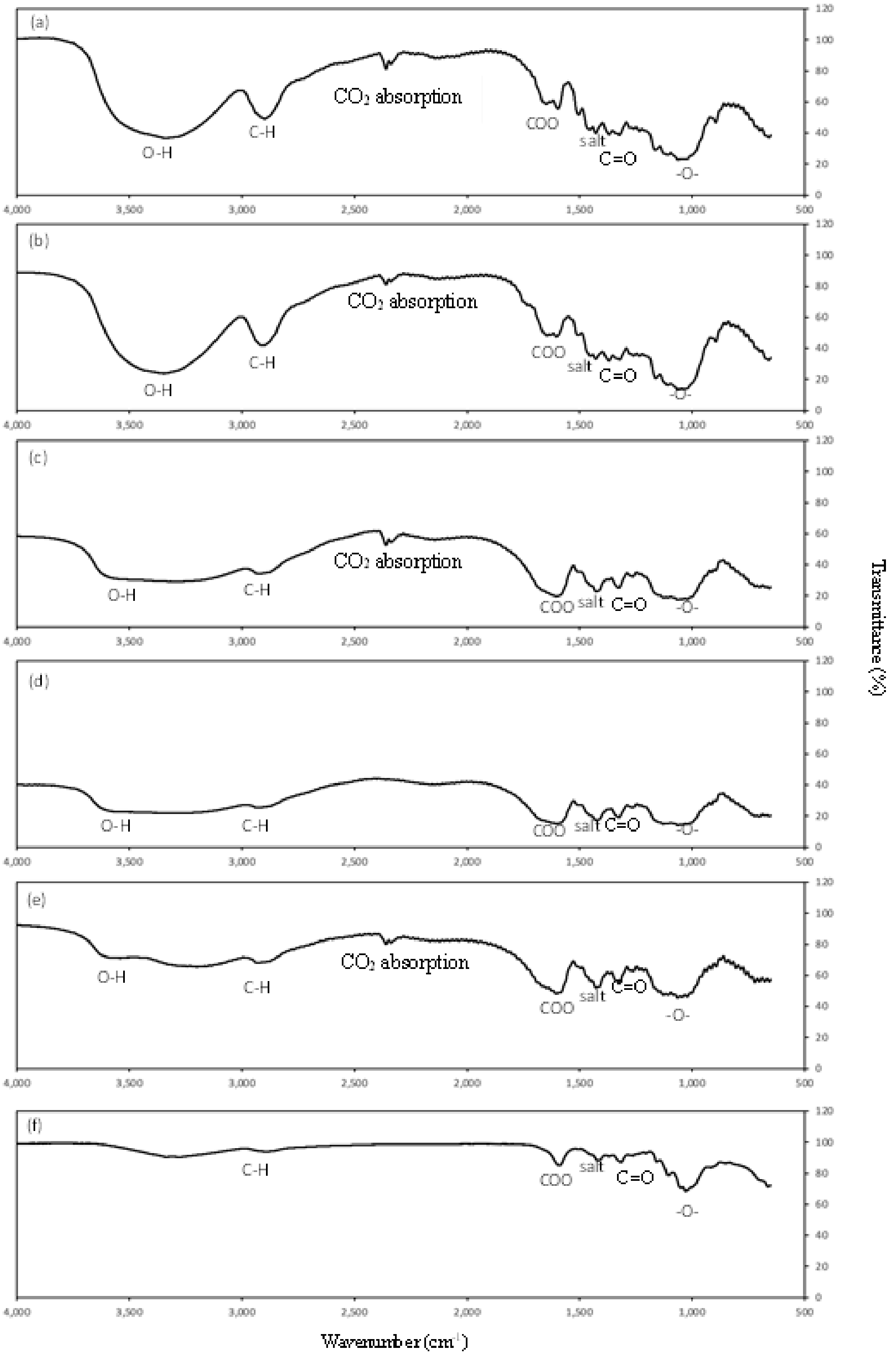

Fig. 4 FTIR spectra of cellulose from durian rind (a); cellulose from pineapple leaf (b); CMC benchmark formula (c); CMC +20% formula (d); CMC -20% formula (e); composite film with 45 wt.% cellulose fiber (f).

similar to those of cellulose (reinforcing fibers) and CMC (matrix) due to the similarity of their functional groups. However, the intensities of the pineapple leaf cellulose spectra were lower than those of cellulose and CMC due to its lower purity, and the bands at 1592 cm^{-1} (C=O), 1318 and 1417 cm^{-1} (-O-), and 1000-1620 cm^{-1} (C-O) were predominant.

3.3 Film thickness and color

Table 4 illustrates the film thickness and film color including the whiteness index (WI) and yellowness index (YI) of the biofilms and biocomposite films fabricated using CMC from durian rind as a film matrix and cellulose fiber extracted from pineapple leaf as a reinforcing material. The results show that the difference in thickness of films prepared from CMC of different chemical compositions was indistinguishable. When the pineapple cellulose was added, the thickness increased slightly as the amounts of fiber increased. In terms of the film color, the pineapple cellulose fiber was whiter in color than that of CMC powder which was slightly yellowish. The composite films were therefore whiter than the neat biofilms as L^* values were higher (see Fig. 5) and hence the whiteness indexes were slightly higher as well and the yellowness indexes were lower. Without the pineapple cellulose, the neat films prepared from CMC at different chemical compositions were comparable in terms of colors. Our neat CMC films have L^* and whiteness index (WI) values of around 88-89 and 93-94, respectively, which are higher than those of 80.59 for L^* and 69.91 for WI of the films prepared by Rachtanapun *et al.* 2012[23] at the same NaOH concentration of 30% w/v. Rachtanapun *et al.* 2012[23] also reported that the durian CMC film's yellowness increased with alkaline pH when the NaOH concentrations ranged between 0-40% w/v and decreased with the pH when the NaOH concentrations were between 40-60% w/v. They suggested that the color of the CMC films depended on the competitive reaction between NaOH and monochloroacetic acid.

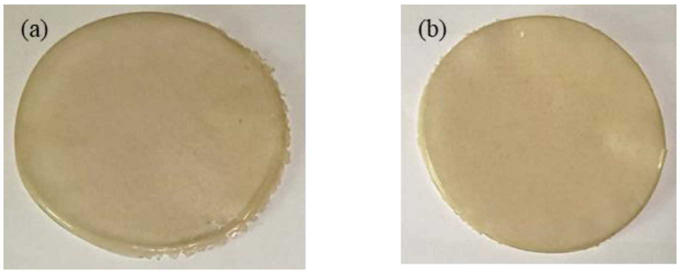

Fig. 5 CMC film (a) and composite film at 45 wt.% pineapple leaf cellulose fiber.

Table 4. Film thickness and color.

Films	Thickne	Color value			WI	YI
		L*	a*	b*		
Bench	0.0911±	88.4	0.27	11.67	93.14	18.86±
+20%	0.0893±	88.9	0.27	12.42	93.36	19.98±
-20%	0.1006±	89.3	0.40	11.37	94.41	18.19±
15	0.1055±	88.4	0.76	10.74	93.21	17.34±
30	0.1109±	90.0	0.89	7.55	93.94	11.97±
45	0.1211±	93.6	0.94	4.54	95.13	7.16±4

3.4 SEM

The morphology of the cellulose fibers extracted from pineapple leaf as well as the dispersion of the fibers in the film composites were studied by SEM and illustrated in Figs 6-10 and 11, respectively. Fig. 6 demonstrates the uneven surface morphology of raw pineapple leaf fibers (before cellulose extraction). The fibers are held as bundles by lignin and other impurities, having a bundle diameter of approximately 50 µm. Similarly, Tanpichai and Witayakran 2018[25] reported the diameter of raw pineapple leaf fibers was in the range of 20-80 µm. The fibers are rod-like in shape with a rough surface and high aspect ratio. However, once they were alkaline treated coupled with a steam explosion, the skin became smooth and the fiber bundles were split as the hemicellulose was hydrolyzed and the lignin was depolymerized (shown in Fig. 7). These findings agreed with Cherian *et al.* 2010,[16] Balakrishnan *et al.* 2017,[17] and Tanpichai and Witayakran 2018,[25] but contradicted the findings by Ravindran *et al.* 2019[15] who observed rougher surfaces for pineapple leaf fibers after NaOH treatment. The bleaching process using H_2O_2 caused defibrillation as shown in Fig. 8. The diameter of the fibers was about 2-4 µm, close to that of bleached pineapple leaf fiber reported by Balakrishnan *et al.* (2017) 2017,[17] Thien *et al.* 2022[18] and Tanpichai and Witayakran 2018.[25] Fig. 9 illustrates SEM images of pineapple leaf cellulose after acid digestion. The images clearly show that the fiber aspect ratio has dramatically decreased. The fibers are shorter, entangled, and agglomerated due to strong hydrogen bonding interactions. The interwoven fiber and agglomeration were also observed in previous studies.[4,17] However, when 6% w/v of cellulose in distilled water underwent mechanical breakage using a high-speed food processor for 2-3 h, the agglomerates were broken up, and short cellulose fibers of 2-4 µm diameters and lengths of approximately 20 µm were obtained as shown in Fig. 10. The resulting fibers were not in nano-sizes as the shearing efficiency of the food processor was not high enough.

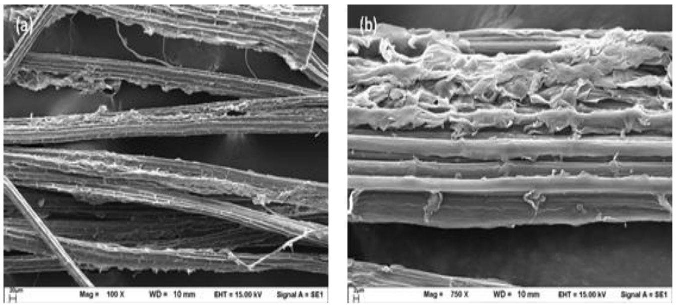

Fig. 6 SEM micrographs of raw pineapple leaf fiber before cellulose extraction at 100X magnification (a); at 750X magnification (b).

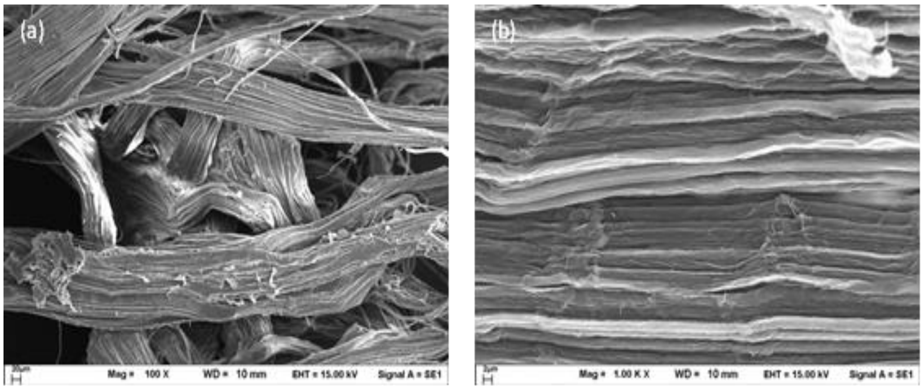

Fig. 7 SEM micrographs of alkaline treated steam exploded pineapple leaf cellulose at 100X magnification (a); at 1000X magnification (b).

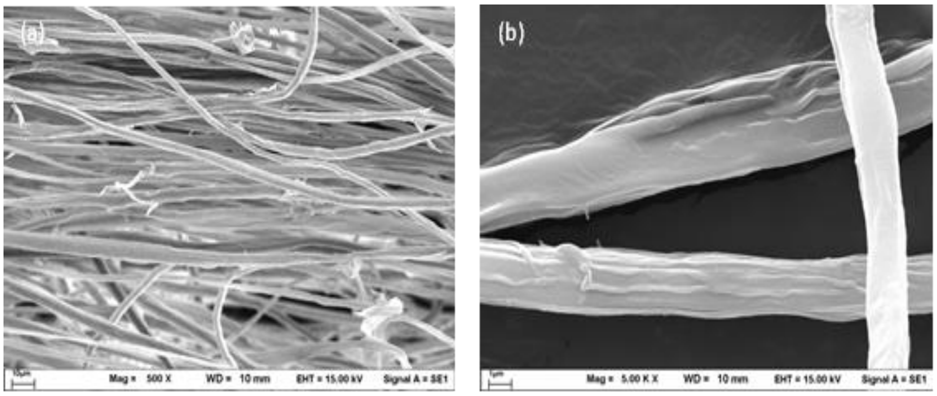

Fig. 8 SEM micrographs of bleached pineapple leaf cellulose at 500X magnification (a); at 5000X magnification (b).

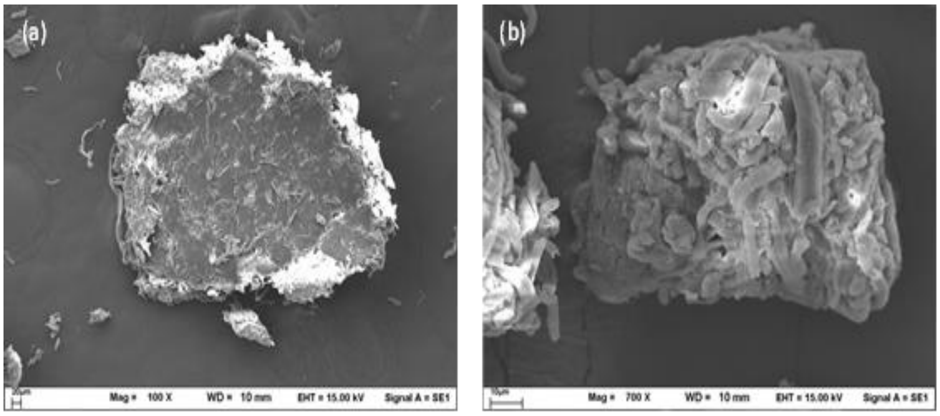

Fig. 9 SEM micrographs of hydrolyzed pineapple leaf cellulose at 100X magnification (a); at 700X magnification (b).

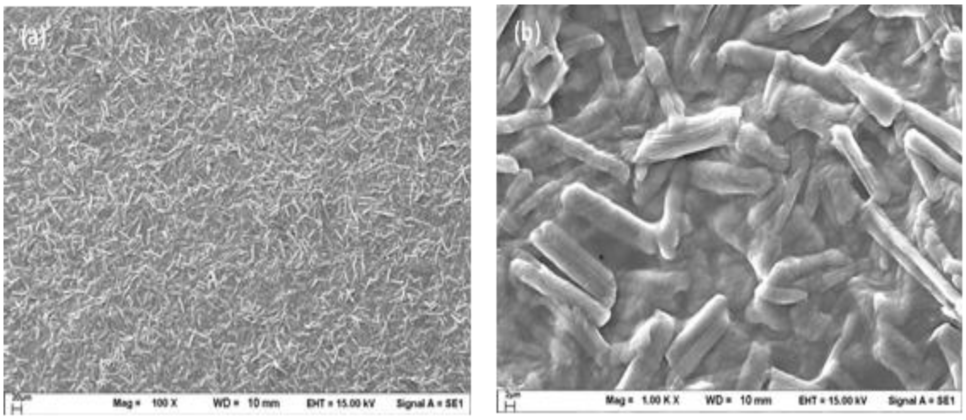

Fig. 10 SEM micrographs of pineapple leaf cellulose after mechanical shearing at 100X magnification (a); at 1000X magnification (b).

The distribution of pineapple leaf celluloses in the durian rind CMC films at different fiber concentrations are presented in Fig. 11. The lines observed on the neat film (Fig. 10(a)) are due to scratches on the acrylic sheets used in the film fabrication process. The cellulose fibers were randomly oriented and considerably homogenously distributed in the CMC films as shown in Figs. 10(b)-(d). This might be due to the compatibility between the matrix and reinforcing phases as they both were derived from the cellulose of agricultural plants. However, the results from tensile testing which is discussed in the following section indicated a slight decrease in tensile strength of the composite film at 45 wt.% fiber compared to 30 wt.% fiber, implying that a slight degree of fiber agglomeration has occurred.

3.5 Mechanical Properties

Typical stress-strain curves of the composite films are shown in Fig. 12 and the average tensile strength and average percent elongation to break of the composite films are illustrated in Fig. 13. The stress-strain curves indicated the neat biofilm was more ductile with less tensile strength and higher elongation than the composite films. The tensile strength of neat durian rind CMC film was found to be 14.90±3.94 MPa which is almost 50% lower than that of Palmyra palm CMC film reported by Kaewprachu *et al.* 2022[7] and significantly lower than that of durian rind CMC film studied by Rachtanapun *et al.* 2012.[23] This may be due to the absence of a plasticizer in the durian rind CMC film prepared by Rachtanapun *et al.* 2012,[23] resulting in high tensile strength of 80-140 MPa. Percent elongation at break of the durian rind CMC film prepared by Rachtanapun *et al.* 2012[23] was also considerably low at around 2-3% whereas the elongation of the durian rind CMC film prepared in this study was 22.63±4.63%. Zhao *et al.* 2019[3] reported the tensile strength and elongation at the break of durian cellulose film to be 44 MPa and 6%, respectively. The low percent elongation at the break of their films can also be attributed to the absence of a plasticizer.

When pineapple leaf cellulose fibers were incorporated into the CMC film, the tensile strength of the films improved. The tensile strength of the composite films increased as the amount of the fibers increased as expected for any composite materials when the reinforcing materials are well dispersed.[4,30] The composite films with 15, 30, and 45 wt.% fiber filler exhibited a tensile strength of 15.63±4.43, 21.03±2.25, and 20.01±4.74 MPa, respectively. Although the tensile strength of these composite films was lower than those of engineering polymers such as polypropylene (PP)[31] and polycarbonate (PC)[32,33] which have a tensile strength of 23 and 35-55 MPa, respectively, it was comparable to that of low-density polyethylene (LDPE) films used for packaging and construction,[34,35] starch-based biodegradable films[36-38] as well as cellulose nanocrystal films.[1] In comparison with

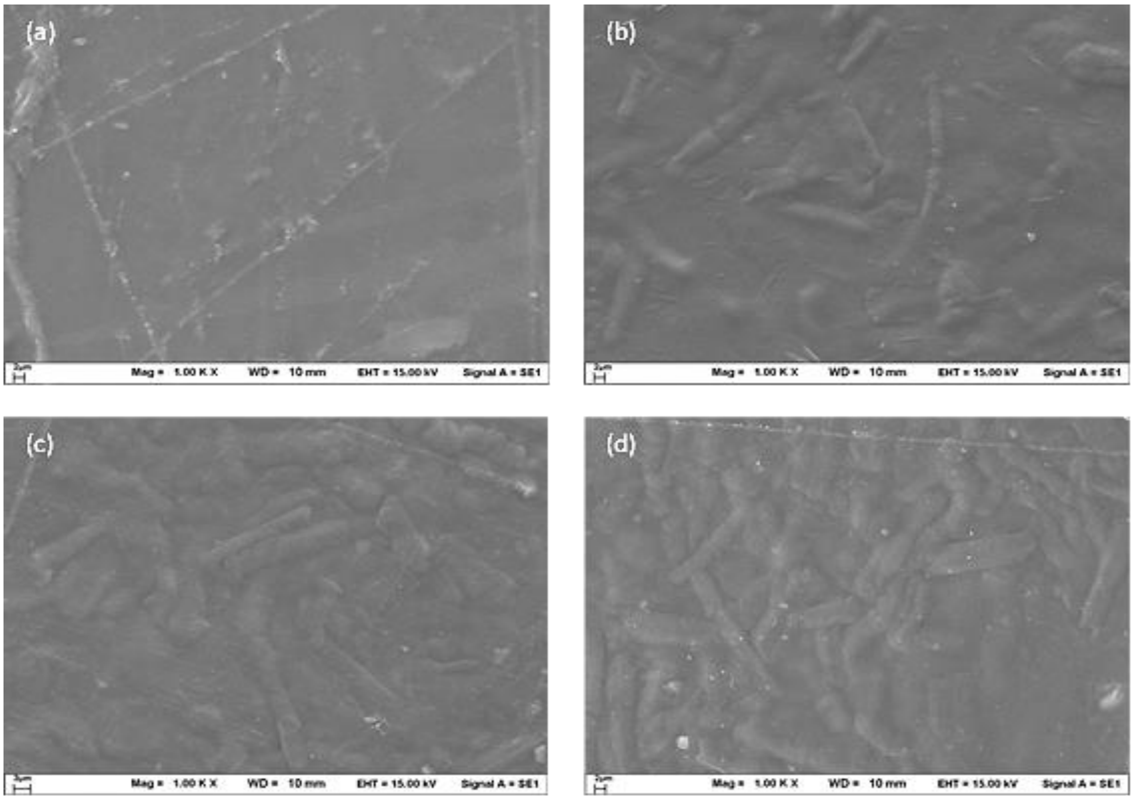

Fig. 11 SEM micrographs of composite films at different pineapple leaf cellulose concentrations at 0 wt.% (a); 15 wt.% (b); 30 wt.% (c); 45 wt.% (d).

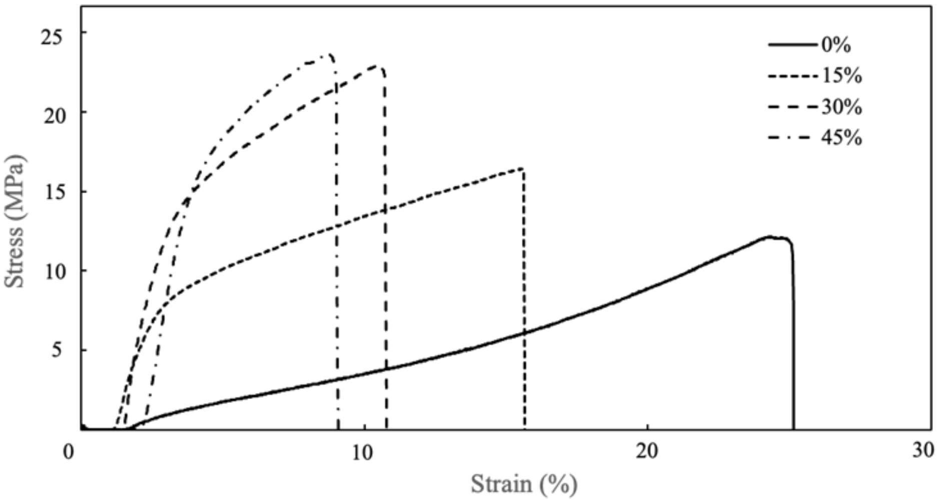

Fig. 12 Stress-strain curves of composite films at different pineapple leaf cellulose concentrations at 0 wt.% (a); 15 wt.% (b); 30 wt.% (c); 45 wt.% (d).

commercial biodegradable polylactic acid (PLA), our biofilms and biocomposite films still had considerably poorer mechanical properties.[39,40]

The results showed that only a 5% increase in tensile strength was attained when 15 wt.% fiber was incorporated in comparison to the neat film. However, as the fiber content increased to 30 wt.%, a 40% increase in tensile strength was achieved when compared with the neat film. A slight tensile strength reduction of 5% of the composite film was observed when the fiber filler content increased from 30 to 45 wt.%. When the cellulose fibers agglomerated in the polymer matrix, a decrease in the tensile strength of the composite films was previously reported.[13,14] Thaithae *et al.* 2016[32] also observed the deterioration of tensile strength and elastic modulus of polycarbonate and multi-walled carbon nanotube composites due to poor carbon nanotube dispersion at high concentrations. In this research, the tensile strength of the composite films at 30 and 45 wt.% fibers was comparable to each other, suggesting the 30 wt.% cellulose was well distributed in the CMC matrix and with a higher amount of the fibers at 45 wt.%, agglomeration could have occurred. The increases in tensile strength of the composite films suggest that the two phases are compatible. The compatibility of the matrix and reinforcing phases was also confirmed by the SEM images. As the reinforcing fibers bonded well with the CMC matrix, the stress transference between the two phases was improved, resulting in higher tensile strength of the composites. In addition, Kowshik *et al.* 2022[41] demonstrated that the eggshell fillers served as crack restriction and arrest in the polyester matrix. The degree of crack deviation and arrest depended on the physical bonding between matrix and filler which relied on various parameters such as particle size and shape as well as interfacial surface area.

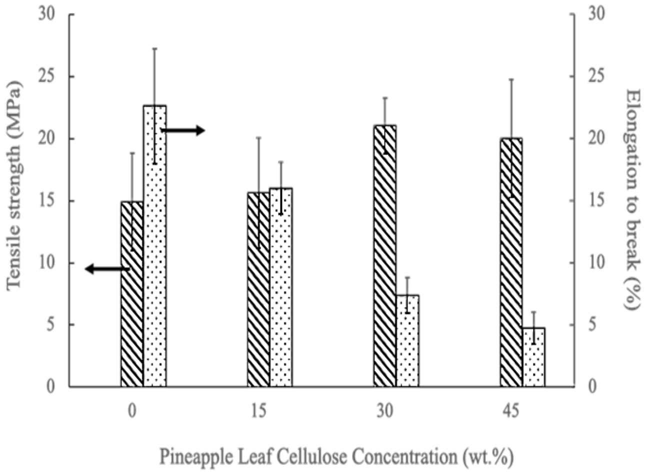

Fig. 13 Mechanical properties of composite films at different pineapple leaf cellulose concentrations at 0 wt.% (a); 15 wt.% (b); 30 wt.% (c); 45 wt.% (d).

In general, the trend of percent elongation at break with the reinforcing material loadings is opposite to tensile strength. This is because fibers usually have inherent high strength and stiffness properties and hence do not elongate much. Furthermore, fibers reduce the mobilities of polymer chains in the matrix. The matrix, on the other hand, is more elastic especially when plasticizers are included. As the tensile strength of the composite films increased with cellulose fiber loadings, the percent elongation at break decreased. The percent elongation to break of the films with 0, 15, 30, and 45 wt.% cellulose fibers were 22.63±4.63, 16.05±2.08, 7.38±1.41, and 4.75±1.28 %, respectively. While 15 wt.% cellulose fiber filler caused a slight increase of 5% in tensile strength of the

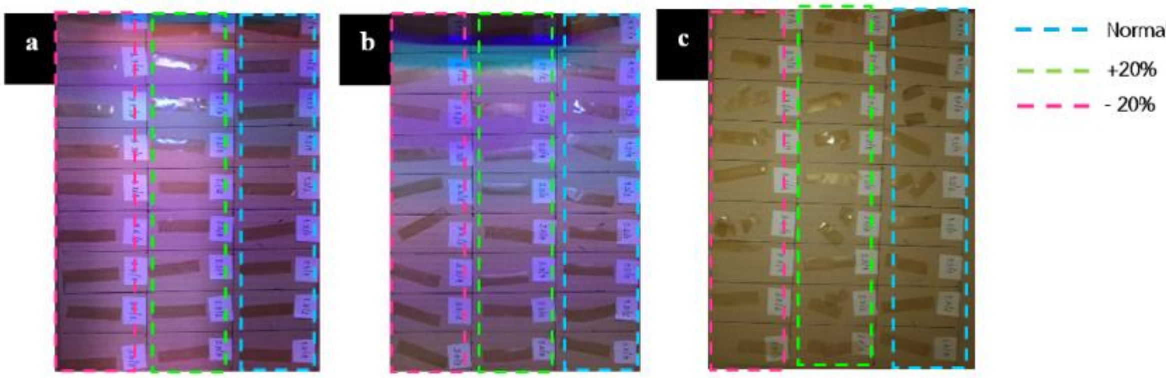

Fig. 14 Photodegradation of neat CMC films prepared from different chemical concentrations on day 1 (a); day 2 (b); day 3 (c).

neat film, a 30% decrease in elongation to break was observed, indicating a strong effect of the fibers on the ductility of the film. Whilst a significant increase in tensile strength was observed for the 30 wt.% fiber composite film, a drastic reduction in elongation to break of nearly 70% was observed compared to the neat film. The tensile strength of the composite films with 30 and 45 wt.% fiber contents was comparable as expected due to the fiber agglomeration, further reduction in the elongation to break was noticed. The reduction in elongation to break with the addition of cellulose fibers was in agreement with Lee *et al.* 2018[30] and Chawalitsakunchai *et al.* 202.[13]

3.6 Photodegradability and biodegradability

Photodegradation of the neat CMC films was studied using LED lights with a luminous flux of 4500 Lm. The conditions inside the carton box at the beginning of the test were 37% Relative Humidity and a temperature of 30 ºC. It was observed that the humidity slightly decreased while the temperature increased to approximately 44 ºC after 3 days. The films' moisture also reduced as the films became harder and less elastic. On day 3, all the films were brittle and broke into pieces as shown in Fig. 14. However, the color changes of the film were not distinguishable by the eye. There was no noticeable difference between the neat CMC films prepared from different chemical compositions.

Unlike photodegradation, when the films were buried in the soil at 60% Relative Humidity and at a temperature of 30 °C at the beginning, the films absorbed water, became gel-like, and stuck with the plastic mesh after 6 h. At 12 h, the films started to dissolve and decompose because of the moisture of the soil. At 18-24 h, all the films were completely degraded as shown in Figs. 15-16. The biodegradability of the neat CMC films of different chemical compositions could not be distinguished; however, the biocomposite films with higher amounts of cellulose fibers, which are water-insoluble, tended to take a longer time to completely decompose. It is well known that the biodegradability of biofilms relies on the hydrolysis of the film with moisture in the soil in the presence of oxygen, temperature, and bacteria. The longer biodegradation period of the cellulose composite films compared to the CMC film was due to CMC's better solubility in water hence, easier to be hydrolyzed. Zhao *et al.* 2019[3] studied the biodegradability of cellulose films prepared from durian rind and found that the films were completely biodegraded in soil within 4 weeks. Whereas commercial PLA possesses excellent mechanical properties, its degradation time was also longer than that of our composite films. Kalita *et al.* 2021[42] revealed that there was a sudden drop in PLA molecular weight under abiotic degradation due to dissolution

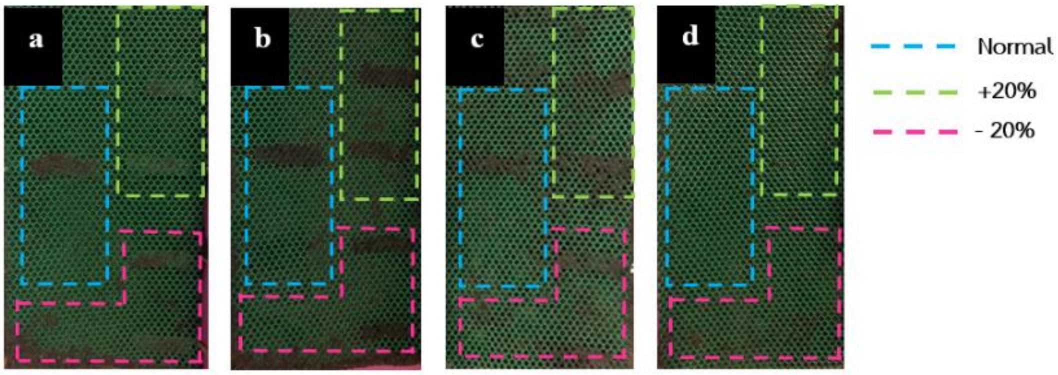

Fig. 15 Biodegradation of neat CMC films prepared from different chemical concentrations at 6 h (a); 12 h (b); 18 h (c); 24 h (d).

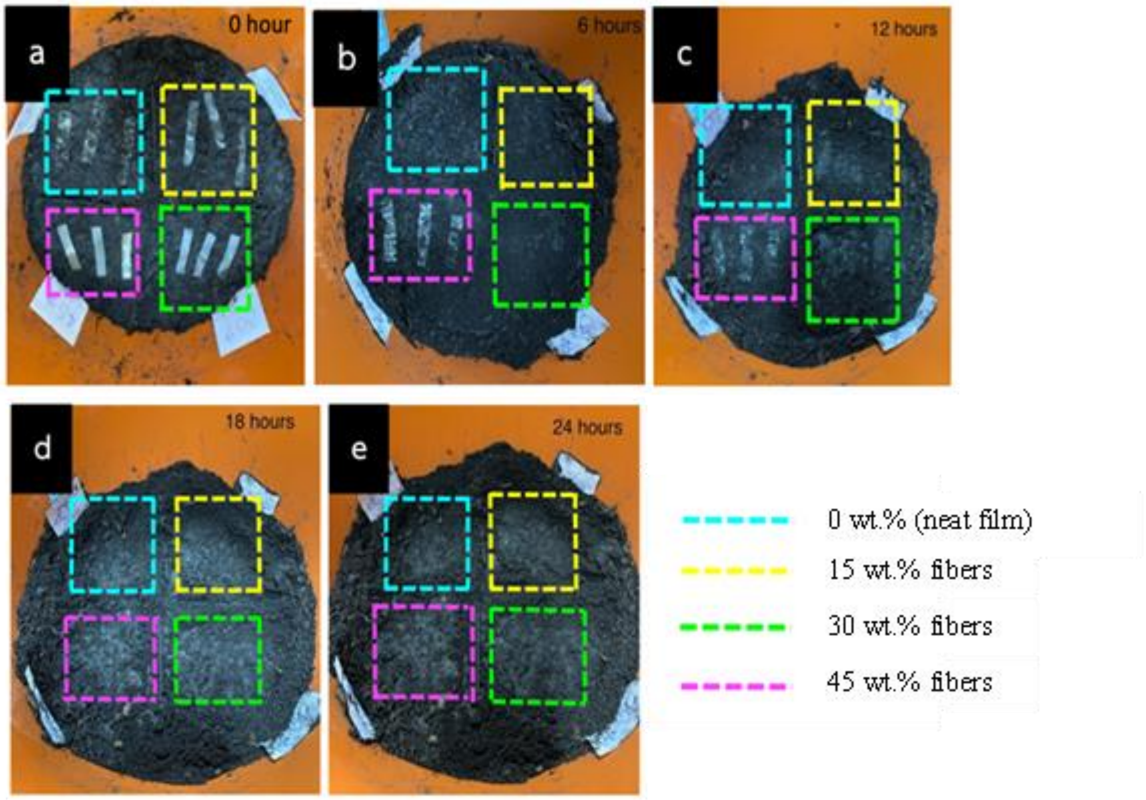

Fig. 16 Biodegradation of composite films of different pineapple leaf cellulose concentrations at 0 h (a); 6 h (b); 12 h (c); 18 h (d); 24 h (e).

and surface erosion of the polymer causing chain scission. However, chain scission alone could not cause a completedecomposition of the polymer. Microbial action played a major role in the biodegradation of the biopolymers and 90% carbon mineralization of PLA under composting conditions was observed at 90 days. Zhou *et al.* 2022[39] found that silver nanoparticles had antimicrobial activity and could hinder or delay PLA decomposition. It was reported that after 30 days of aerobic biodegradation, carbon dioxide generated from CMC film was twice as much as that from PLA.[1] By incorporating 43 wt.% plasticized cassava starch into LDPE film, 12% degradation of the composite film was achieved at 66 days under soil burial.

4. Conclusions

In this study, biofilm and biocomposite films derived from agricultural wastes, *i.e.* durian rind and pineapple leaf, were successfully fabricated via a solution casting method. The matrix of the film was prepared from carboxymethyl cellulose (CMC) from durian rind whereas the pineapple leaf cellulose was incorporated into the film as a reinforcing fiber. Durian rind cellulose was extracted using an alkaline treatment and delignification process. CMC was achieved by substituting hydroxyl groups in the cellulose with carboxymethyl groups through alkalization and etherification processes. The effects of chemical composition used in these two processes on the hydroxyl group substitution as well as the physical properties of the CMC films were studied. The range of chemical composition studied was ±20% of the normal formula. FTIR investigation as well as the degradation study suggested that the range of chemical composition studied did not show any distinguishable changes in film properties. Steam explosion, acid hydrolysis, and mechanical shearing were incorporated in the preparation of fine pineapple leaf cellulose fibers for use as the reinforcing material. The biocomposite films were prepared at fiber contents of 15, 30, and 45 wt.%. SEM images illustrated the homogenous distribution of the fibers in the polymer matrix; however, the tensile test results suggested that fiber agglomeration at a fiber content of 45 wt.% may have occurred. Tensile strengths of the biofilm and biocomposite films were in the range of 15-21 MPa which is comparable to some synthetic plastic films. The highest tensile strength of 21 MPa, a 40% improvement over the neat CMC film, was achieved from a composite film containing 30 wt.% cellulose fiber. At the highest fiber content of 45 wt.%, the composite film's mechanical properties and biodegradation period were found to be negatively affected. This is thought to be due to cellulose fiber's lower solubility in water compared to the CMC films. Nevertheless, these biocomposite films were found to dissolve with the moisture contained in the soil and were fully degraded within 24 h, significantly faster than the degradation periods of commercial synthetic plastic films.

The concept of the research undertaken has been driven by the Thailand government's Bio-Circular-Green (BCG) economic strategy. This study has demonstrated the potential for value-adding to agricultural scraps as well as waste reduction. Further investigation and development on the usage of durian rind and pineapple leaves as alternative materials in various industries are highly recommended. This research could potentially also be extended to the use of other agricultural wastes.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

References

[1] J. F. Santos, C. Valls, O. Cusola, M. B. Roncero, Composites of cellulose nanocrystals in combination with either cellulose nanofibril or carboxymethylcellulose as functional packaging films, *International Journal of Biological Macromolecules*, 2022, **211**, 218-229, doi: 10.1016/j.ijbiomac.2022.05.049.

[2] O. Faruk, A. K. Bledzki, H.-P. Fink, M. Sain, Biocomposites reinforced with natural fibers: 2000-2010, *Progress in Polymer Science*, 2012, **37**, 1552-1596, doi: 10.1016/j.progpolymsci.2012.04.003.

[3] Guili, Zhao, Biodegradable and transparent cellulose film prepared eco-friendly from durian rind for packaging application, *Food Packaging and Shelf Life*, 2019, **21**, 100345, doi: 10.1016/j.fpsl.2019.100345.

[4] B. M. Cherian, A. L. Leão, S. F. de Souza, L. M. M. Costa, G. M. de Olyveria, M. Kottaisamy, E. R. Nagarajan, S. Thomas, Cellulose nanocomposites with nanofibres isolated from pineapple leaf fibers for medical applications, *Carbohydrate Polymers*, 2011, **86**, 1790-1798, doi: 10.1016/j.carbpol.2011.07.009.

[5] D. Pradhan, A. K. Jaiswal, S. Jaiswal, Emerging technologies for the production of nanocellulose from lignocellulosic biomass, *Carbohydrate Polymers*, 2022, **285**, 119258, doi: 10.1016/j.carbpol.2022.119258.

[6] K. A. Khalid, A. A. Ahmad, T. Len-Kelly Yong, Lignin extraction from lignocellulosic biomass using sub- and supercritical fluid technology as precursor for carbon fiber production, *Journal of the Japan Institute of Energy*, 2017, **96**, 255-260, doi: 10.3775/jie.96.255.

[7] P. Kaewprachu, C. Jaisan, S. Rawdkuen, W. Tongdeesoontorn, W. Klunklin, Carboxymethyl cellulose from Young Palmyra palm fruit husk: synthesis, characterization, and film properties, *Food Hydrocolloids*, 2022, **124**, 107277, doi: 10.1016/j.foodhyd.2021.107277.

[8] D. B. K. Lim, H. Gong, Highly stretchable and transparent films based on cellulose, *Carbohydrate Polymers*, 2018, **201**, 446-453, doi: 10.1016/j.carbpol.2018.08.080.

[9] A. L. Leão, B. M. Cherian, S. Narine, S. F. Souza, M. Sain, S. Thomas, The use of pineapple leaf fibers (PALFs) as reinforcements in composites, *Biofiber Reinforcements in Composite Materials*, 2015, 211-235, doi: 10.1533/9781782421276.2.211.

[10] S. S. Todkar, S. A. Patil, Review on mechanical properties evaluation of pineapple leaf fibre (PALF) reinforced polymer composites, *Composites Part B: Engineering*, 2019, **174**, 106927, doi: 10.1016/j.compositesb.2019.106927.

[11] K. Senthilkumar, M. Chandrasekar, O. Y. Alothman, H. Fouad, M. Jawaid, M. A. Azeem, Flexural, impact and dynamic mechanical analysis of hybrid composites: Olive tree leaves powder/ pineapple leaf fibre/epoxy matrix, *Journal of Materials Research and Technology*, 2022, **21**, 4241-4252, doi: 10.1016/j.jmrt/2022.11.036.

[12] K. Senthilkumar, N. Saba, M. Chandrasekar, M. Jawaid, N. Rajini, S. Siengchin, N. Ayrilmis, F. Mohammad, H. A. Al-Lohedan, Compressive, dynamic and thermo-mechanical properties of cellulosic pineapple leaf fibre/polyester composites: influence of alkali treatment on adhesion, *International Journal of Adhesion and Adhesives*, 2021, **106**, 102823, doi: 10.1016/j.ijadhadh.2021.102823.

[13] W. Chawalitsakunchai, P. Dittanet, S. Loykulnant, P. Sae-oui, S. Tanpichai, A. Seubsai, P. Prapainainar, Properties of natural rubber reinforced with nano cellulose from pineapple leaf agricultural waste, *Materials Today Communications*, 2021, **28**, 102594, doi: 10.1016/j.mtcomm.2021.102594.

[14] S. Charoenvai, Durian peels fiber and recycled HDPE composites obtained by extrusion, *Energy Procedia*, 2014, **56**, 539-546, doi: 10.1016/j.egypro.2014.07.190.

[15] Lakshmipriya, Ravindran, Novel processing parameters for the extraction of cellulose nanofibres (CNF) from environmentally benign pineapple leaf fibres (PALF): structure-property relationships, *International Journal of Biological Macromolecules*, 2019, **131**, 858-870, doi: 10.1016/j.ijbiomac.2019.03.134.

[16] B. M. Cherian, A. L. Leão, S. F. de Souza, S. Thomas, L. A. Pothan, M. Kottaisamy, Isolation of nanocellulose from pineapple leaf fibres by steam explosion, *Carbohydrate Polymers*, 2010, **81**, 720-725, doi: 10.1016/j.carbpol.2010.03.046.

[17] P. Balakrishnan, M. S. Sreekala, M. Kunaver, M. Huskić, S. Thomas, Morphology, transport characteristics and viscoelastic polymer chain confinement in nanocomposites based on thermoplastic potato starch and cellulose nanofibers from pineapple leaf, *Carbohydrate Polymers*, 2017, **169**, 176-188, doi: 10.1016/j.carbpol.2017.04.017.

[18] D. Van Hong Thien, D.-N. Lam, H. N. Diem, T. Y. N. Pham, N. Q. Bui, T. N. T. Truc, D.-T. Van-Pham, Synthesis of cellulose-g-poly(acrylic acid) with high water absorbency using pineapple-leaf extracted cellulose fibers, *Carbohydrate Polymers*, 2022, **288**, 119421, doi: 10.1016/j.carbpol.2022.119421.

[19] Y. Chen, H. Zhang, X. Feng, L. Ma, Y. Zhang, H. Dai, Lignocellulose nanocrystals from pineapple peel: preparation, characterization and application as efficient Pickering emulsion stabilizers, *Food Research International*, 2021, **150**, 110738, doi: 10.1016/j.foodres.2021.110738.

[20] S. R. Masrol, M. H. I. Ibrahim, S. Adnan, Chemi-mechanical Pulping of Durian Rinds, *Procedia Manufacturing*, 2015, **2**, 171-180, doi: 10.1016/j.promfg.2015.07.030.

[21] A. Abdulhameed, H. M. Mbuvi, E. O. Changamu, F. M. Maingi, Microwave synthesis of Carboxymethylcellulose (CMC) from Rice Husk, *IOSR Journal of Applied Chemistry*, 2019, **12**, 33-42, doi: 10.9790/5736-1212013342.

[22] L. Golbaghi, M. Khamforoush, T. Hatami, Carboxymethyl cellulose production from sugarcane bagasse with steam explosion pulping: experimental, modeling, and optimization, *Carbohydrate Polymers*, 2017, **174**, 780-788, doi: 10.1016/j.carbpol.2017.06.123.

[23] P. Rachtanapun, S. Luangkamin, K. Tanprasert, R. Suriyatem, Carboxymethyl cellulose film from durain rind, *LWT – Food Science and Technology*, 2012, **48**, 52-58, doi: 10.1016/j.lwt.2012.02.029.

[24] Durian: Farming area, Production, and Production per area, Office of Agricultural Economics, 2022, www.oae.go.th (accesses 19 April 2022).

[25] S. Tanpichai, S. Witayakran, All-cellulose composites from pineapple leaf microfibers: structural, thermal, and mechanical properties, *Polymer Composites*, 2018, **39**, 895-903, doi: 10.1002/pc.24015.

[26] S. Khemkaew, S. Kaewpirom, Effects of glycerol and PEG-10 dimethicone on properties of biofilm from durian rind, *The Journal of Industrial Technology*, 2016, **2**, 11-21 (in Thai).

[27] R. Lubis, S. W. Saragih, B. Wirjosentono, E. Eddyanto, Characterization of Durian Rinds Fiber (Durio zubinthinus, Murr) from North Sumatera, *AIP Conference Proceedings*, 2018, **2049**, 020069, doi: 10.1063/1.5082474.

[28] P. Penjumras, R. B. A Rahman, R. A. Talib, K. Abdan, Extraction and chatacterization of cellulose from durian rind, *Agriculture and Agricultural Science Procedia*, 2014, **2**, 237-243, doi: 10.1016/j.aaspro.2014.11.034.

[29] A. R. S. Neto, M. A. M. Araujo, R. M. P. Barboza, A. S. Fonseca, G. H. D. Tonoli, F. V. D. Souza, L. H. C. Mattoso, J. M. Marconcini, Comparative study of 12 pineapple leaf fiber varieties for use as mechanical reinforcement in polymer composites, *Industrial Crops and Products*, 2015, **64**, 68-78, doi: 10.1016/j.indcrop.2014.10.042.

[30] M. C. Lee, S.C. Koay, M. Y. Chan, M. M. Pang, P. M. Chou, K. Y. Tsai, Preparation and Characterzation of Durian Husk Fiber Filled Polylactic Acid Biocomposites, *MATEC Web of Conferences*, 2018, **152**, 02007, doi: 10.1051/matecconf/201815202007.

[31] P. Wattanachai, B. Buasathain, C. Antonio, S. Roces, Open Loop Recycling of Recycled Polypropylene for Motorcycle Saddle Application, *ASEAN Journal of Chemical Engineering*, 2017, **17**, 60-76, doi: 10.22146/ajche.49556.

[32] W. Thaithae, C. Antonio, P. Wattanachai, Properties characterisation of polycarbonate and multiwalled carbon nanotubes composites prepared by solution technique, *Asia-Pacific Journal of Chemical Engineering*, 2016, **11**, 34-50, doi: 10.1002/apj.1940.

[33] P. Boonruam, S. Uppasen, S. Soisuwan, C. Antonio, P. Wattanachai, Natural rubber to replace acrylonitrile butadiene styrene in polycarbonate blends and composites, *Engineering and Applied Science Research*, 2021, **48**, 287-294, doi: 10.14456/easr.2021.31.

[34] T. Kojnoková, F. Nový, L. Markovičová, T. Liptáková, Changes of mechanical properties of protective polyethylene films applied in transport bottles and containers for liquid media after exposure to selected liquid media, *Transportation Research Procedia*, 2021, **55**, 731-736, doi: 10.1016/j.trpro.2021.07.041.

[35] O. Szlachetka, J. W.-Dobrev, A. Baryła, M. Dohojda, Low-density polyethylene (LDPE) building films – Tensile properties and surface morphology, *Journal of Building Engineering*, 2021, **44**, 103386, doi: 10.1016/j.jobe.2021.103386.

[36] K. D. Tafa, N. Satheesh, W. Abera, Mechanical properties of tef starch based edible films: Development and process optimization, *Heliyon*, 2023, **9**, e13160, doi: 10.1016/j.heliyon.2023.e13160.

[37] E. Kibirkstis, V. Mayik, R. Zatserkovna, K. Vaitasius, A. Stepanenko, R.K. Janutiene, I. Venyte, and P.P. Danilovas, Study of physical and mechanical properties of partially biodegradable LDPE polymeric films and their application for printing and packaging, *Polymer Testing*, 2022, **112**, 107646, doi: 10.1016/j.polymertesting.2022.107646.

[38] I. C. Nwuzor, H. C. Oyeoka, S. C. Nwanonenyi, G. O. Ihekweme, Biodegradation of low-density polyethylene film/plasticized cassava starch blends with central composite design for optimal environmental pollution control, *Journal of Hazardous Materials Advances*, 2023, **9**, 100251, doi: 10.1016/j.hazadv.2023.100251.

[39] J. Zhou, B. Wang, C. Xu, Y. Xu, H. Tan, X. Zhang, Y. Zhang, Performance of composite materials by wood fiber/polydopamine/silver modified PLA and the antibacterial property, *Journal of Materials Research and Technology*, 2022, **18**, 428-438, doi: 10.1016/j.jmrt.2022.02.113.

[40] T. Tabi, T. Ageyeva, J. G. Kovacs, The influence of nucleating agents, plasticizers, and molding conditions on the properties of injection molded PLA products, *Materials Today Communication*, 2022, **32**, 103936, doi: 10.1016/j.mtcomm.2022.103936.

[41] S. Kowshik, S. Sharma, S. R. U, M. Shettar, P. Hiremath, A. Upadhyaya, Investigation on the effects of uncarbonised, carbonised and hybrid eggshell filler addition on the mechanical properties of glass fibre/polyester composites, *Engineered Science*, 2022, **18**, 121-131, doi: 10.30919/es8d679.

[42] N. K. Kalita, N. A. Damare, D. Hazarika, P. Bhagabati, A.

Kalamdhad, V. Katiyar, Biodegradation and characterization study of compostable PLA bioplastic containing algae biomass as potential degradation accelerator, *Environmental Challenges*, 2021, **3**, 100067, doi: 10.1016/j.envc.2021.100067.

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