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Comparative study of physico-mechanical properties, thermal stability and water absorption of biodegradable films prepared from commercial oxidized and cross-linked cassava starches

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Abstract

Physico-mechanical properties, thermal stability and water absorption of biodegradable films from chemically modified cassava starches were investigated. Two types of commercial modified cassava starches as oxidized cassava starch and cross-linked cassava starch were prepared by film casting. Starch solution was prepared at a concentration of 1% w/w (dry basis) and glycerol was used as a plasticizer. Unmodified cassava starch was used as the control film. The obtained films were characterized based on their physical, morphological, mechanical and thermal properties as well as water absorption. Results indicated that oxidized and cross-linked cassava starch films were easily fabricated using the film casting method with glycerol content at 30% w/w of dried starch weight as a plasticizer. Thickness of modified cassava starch films ranged 0.06-0.16 mm. Oxidized cassava starch and cross-linked cassava starch films were transparent. Cassava starch modified by both oxidation and cross-linking methods resulted in improved tensile strength of films at 2.39-4.59 folds with reduced water absorption by 3.36-3.72 folds. Chemical modification with oxidation resulted in cassava starch films with high transparency, smooth surface and high strength and stiffness, while cross-linking produced cassava starch films with high extensibility. Hence, biodegradable films from both oxidized and cross-linked cassava starches have a great potential to be applied to food packaging.

Keywords**:** *biodegradable films, modified cassava starch, morphology, physico-mechanical properties, thermal stability, water absorption*

1. Introduction

Edible films are widely used for food products as mostly fruits, vegetables, candies, and certain nuts. The films enhance the quality of the merchandise and also protect food products against physical, chemical, and biological deterioration (Han, 2014). Normally, edible films and coating materials are biodegradable biopolymers such as proteins, polysaccharides, lipids, and resins. Biodegradability is one of their greatest benefits, along with their edibility (Han, 2014; Zhao & McDaniel, 2005).

Starch is a polysaccharide that is abundant in nature. Starch is composed of two biopolymers as (i) amylose that has a linear structure comprising glucose residues linked through α-(1,4)-glycosidic linkages, and (ii) amylopectin, a branched structure consisting of α-(1,4) glucans with inter-linked α-(1,6)-glycosidic linkages. Generally, starch contains amylose at around 20-25% and amylopectin at around 75-80% depending on the plant sources (Zhang, Rempel, & Liu, 2014). In Thailand, cassava is considered one of the most important economic crops with annual production of around 25 million tons (Piyachomkwan & Tanticharoen, 2011). Biodegradable/edible films from cassava starch provide good flexibility; however, the tensile strength of cassava starch films is low compared to films from other starches (Vu & Lumdubwong, 2016). Cassava starch films also contain many hydroxyl groups in their structure, and these are naturally hydrophilic. Cassava starch can be chemically, physically or genetically modified to enhance its use in many applications (Han, 2014).

Recently, starch has been chemically modified using different methods to replace the hydroxyl groups with other functional groups (Hag et al., 2019). Chemical modification involves the insertion of a new functional group on the starch backbone by acid hydrolysis, cross-linking, acetylation/esterification, dual modification,

oxidation, and grafting to give characteristic properties to the starch (Han, 2014). One advantage of these modified starches is their low cost compared to other biopolymers such as poly(lactic acid) (PLA) and polyhydroxyalkanoates (PHAs) (Han, 2014). Research into the chemical modification of starch has recently gained popularity because native starch is insoluble in water and easily undergoes retrogradation. This problem can be overcome by upgrading starch properties through chemical modifications (Han, 2014; Hag et al., 2019).

Oxidized starch was prepared by treatment with sodium hypochlorite and active chlorine. Water solubility of oxidized starch films is low because oxidation produces carboxyl groups that affect intra-molecular bonding and interaction between the amylose molecules, and this reduces water absorption. Oxidized starch has improved stability with good film-forming properties and lower viscosity. The production of chemical side bonds in different polymer chains is called cross-linking. Many well-known cross-linking reagents include phosphorus oxychloride, epichlorohydrin, sodium tripolyphosphate and sodium trimetaphosphate. Chemical cross-linking agents give a stronger film structure with higher resistance and greater longevity (Hag et al., 2019).

Recently, biodegradable/edible films from oxidized cassava starch (de Pauli, Quast, Demiate, & Sakanaka, 2011; Sondari & Iltizam, 2018; Dai, Zhang, & Cheng, 2019; Oluwasina, Olaleye, Olusegun, Oluwasina, & Mohallem, 2019) and cross-linked cassava starch (Marques et al., 2006; Gutiérrez, Tapia, Pérez, & Famá, 2015; Gutiérrez, Morales, Pérez, Tapia, & Famá, 2015; Seligra, Jaramillo, Famá, & Goyanes, 2016) have been reported. Biodegradable/edible films from oxidized cassava starch demonstrated higher tensile strength, Young's modulus and elongation at break, lower moisture content/or more hydrophobicity as well as higher brightness and transparent as compared to unmodified cassava starch. In addition, biodegradable/edible films from cross-linked cassava starch exhibited as higher tensile strength and elongation at break, higher water barrier/or lower solubility in water compared to unmodified cassava starch.

2. Objectives

This research aimed to study the physico-mechanical properties such as thickness, optical properties, microstructure, tensile properties of modified cassava starch films from commercial oxidized and cross-linked cassava starches as well as their thermal stability and water absorption. Generally, the physico-mechanical properties, thermal stability and water absorption play a vital role in further biodegradable film applications for customer perceptions. Moreover, film prepared from unmodified cassava starch was used as a control film.

3. Materials and methods

3.1 Materials

Native cassava starch (Dragon Fish brand) was purchased from Tong Chan Registered Ordinary Partnership (Thailand). Commercial oxidized cassava starch and commercial cross-linked cassava starch were supplied by First Starch International Co., Ltd. (Thailand). Commercial grade glycerol was used as received from Global Green Chemicals Public Company Limited (Thailand).

3.2 Preparation of biodegradable films from modified cassava starches

Biodegradable films from modified cassava starches; i.e. oxidized cassava starch and cross-linked cassava starch were prepared using film casting according to the method from Vu and Lumdubwong (2016). Modified cassava starches were mixed with distilled water at a concentration of 1% w/w (dry basis). Each modified starch suspension was heated to 80 °C and mixed at 290 rpm using a magnetic stirrer (SI Analytics GmbH, Germany). The starch suspensions were magnetically stirred for 1 h to obtain a clear solution. Commercial glycerol was then added to each modified starch solution at 30% w/w of dried starch weight. The modified starch solutions were continuously stirred at 290 rpm at 80 °C for 5 min. Each modified starch solution (400 mL) was poured onto a square 30 cm × 30 cm acrylic plate and dried in a hot-air oven (FD53, WTB binder, USA) at 45±5 °C for 24 h. The films were peeled off and stored in a desiccator containing silica gel at ambient temperature until required for further analyses. Native cassava starch was prepared and stored in the same manner and used as the control film.

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3.3 Characterization and property testing of biodegradable films from modified cassava starches

Thickness of each sample was measured using a micrometer (Mitutoyo, Japan). Each sample was cut into a rectangular shape 15 mm × 100 mm and thickness was measured at five positions. Optical properties were also measured as color and opacity percentage using a UltraScan VIS colorimeter (HunterLab, USA) in transmittance mode, following the classification system of CIELab and illuminant D65 (daylight) (HunterLab, 2001). Surface and cross-sectional morphological observations were carried out using a JSM-6610V scanning electron microscope (JEOL, Japan) at an acceleration voltage of 15 kV. Samples were directly mounted on a stub using a two-sided carbon tape, followed by coating with a thin layer of gold for surface morphology observation. For cross-sectional morphology observation, samples were first broken in liquid nitrogen before mounting on the stub and coating with gold. Thermogravimetric analysis was carried out at a temperature range of 30-500 °C and heating rate of 20 °C/min using a TGA/DSC1 (Mettler Toledo, Switzerland). All measurements were performed under nitrogen atmosphere with a flow rate of 50 mL/min. Each sample was evaluated in duplicate. Tensile properties of the samples were tested according to ASTM D882-02 (2002) using a TA-XT plus (Texture Analyzer, UK). Each sample was cut into a rectangular shape with dimensions of 100 mm × 15 mm and then conditioned in a desiccator containing magnesium nitrate salt solution (25±5 °C, 50±5 %RH) for at least 48 h. Preconditioned samples were then placed between the grips of the testing machine with an initial grip separation of 50 mm and speed of 12.5 mm/min. Each sample was evaluated in triplicate. Water absorption of the samples was tested according to ASTM D570-98 (1998). Each sample was cut into a square shape with dimensions of 2.54 mm × 2.54 mm and then dried at 105 °C for 1 h to remove the moisture. All samples were stored in a desiccator until they achieved ambient temperature. Each sample was initially weighed (W_0) and then placed in distilled water (20 mL) for 2 h. After removal, each sample was immediately wiped with tissue papers and weighed (W_1). Water absorption percentage was calculated according to equation (1).

$$\text{Water absorption percentage} = [(W_1-W_0)/W_0] \times 100 \quad (1)$$

4. Results and discussion

4.1 Thickness of biodegradable films from modified cassava starches

Table 1 shows the thickness of different films. The thickness of the oxidized cassava starch film was 0.21 mm, where (Bierkle et al., 2011) modified cassava starch solution easily spread on in the acrylic plate. The lowest thickness of the cross-linked cassava starch film solution (de Pauli et al., 2011) due to degradation of the polymer through partial depolymerization and partial cleavage of glycosidic bonds that lowered molecular mass

Table 1 Thickness of films prepared from modified cassava starches

Sample	Thickness (mm)
Unmodified cassava starch	0.21±0.02[a]
Oxidized cassava starch	0.06±0.01[b]
Cross-linked cassava starch	0.16±0.07[a]

Data are reported as mean±SD (n=5). Different small letters indicate significant difference at $p < 0.05$ (Duncan's new multiple range test).

4.2 Optical properties of biodegradable films from modified cassava starches

The optical properties of polymer films are related to brightness, color (both greenness/redness and blueness/yellowness), and opacity (transparency). Figure 1 demonstrates the appearances of different films. All films exhibited visual transparency (Figures 1a-1c).

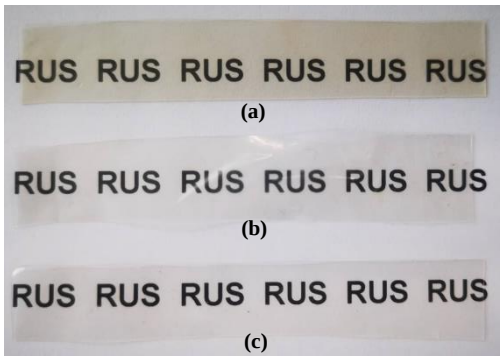

Figure 1 Appearances of different films: (a) unmodified cassava starch (b) oxidized cassava starch and (c) cross-linked cassava starch

Table 2 shows L^*, a^*, b^* values and the opacity percentage of different films corresponding to brightness, greenness/redness, blueness/yellowness, and degree of transparency, respectively. L^* values of all films were not significantly different, implying the same level of brightness for all films. For a^* value, cross-linked cassava starch film showed little greener than films from unmodified and oxidized cassava starches similar to the results revealed from Dai et al. (2019). They also mentioned that it may be difficult to completely distinguish these native and modified starch films by using one of L^*, a^* and b^* values due to the minor differences (Dai et al., 2019). The b^* value of the unmodified cassava starch film was significantly higher than those of oxidized and cross-linked cassava starches (Table 2) that were more yellowish (see Figure 1a). This might be explained by removing of color pigments in native cassava starch by oxidizing and cross-linking agents. Xu et al (2018) also reported that anhydrous ethanol washing must be performed to remove pigments in root tubers (Xu et al., 2018). Opacity percentage indicates the transparency of films. This is important for good visibility of the product by consumers. Opacity percentage of oxidized cassava starch film was the lowest at 1.77% suggesting good transparency (Table 2).

Table 2 Optical properties of films prepared from modified cassava starches

Sample	Optical properties			
	L^*	a^*	b^*	Opacity (%)
Unmodified cassava starch	65.06±1.08^a	-1.60±0.11^a	5.07±0.70^a	3.53±0.50^a
Oxidized cassava starch	65.12±0.64^a	-1.67±0.02^a	3.25±0.19^b	1.77±0.42^b
Cross-linked cassava starch	66.54±0.24^a	-1.88±0.03^b	2.95±0.20^b	3.83±0.40^a

Data are reported as mean±SD (n=5). Different small letters in the same column indicate significant difference at $p < 0.05$ (Duncan's new multiple range test).

4.3 Morphological properties of biodegradable films from modified cassava starches

Figure 2 shows surface and cross-sectional morphologies of the samples. The unmodified cassava starch film exhibited small pieces of starch granules (at the white arrows) in the micrograph (Figure 2a), suggesting that the starch granules were partially disrupted, while oxidized and cross-linked cassava starch films displayed smooth surfaces with homogenous structures (Figures 2b-2c). In terms of the cross-section, oxidized cassava starch films were smoother than the control and cross-linked cassava starch films. Their smoother cross-sectional surfaces may explain the lowest opacity percentage from Table 2. Similarly, Pelissari, Andrade-Mahecha Sobral and Menegalli (2013) found that banana starch film was denser and had a more homogeneous polymeric structure than banana flour film, resulting in higher smoothness, gloss, and transparency (Pelissari et al., 2013).

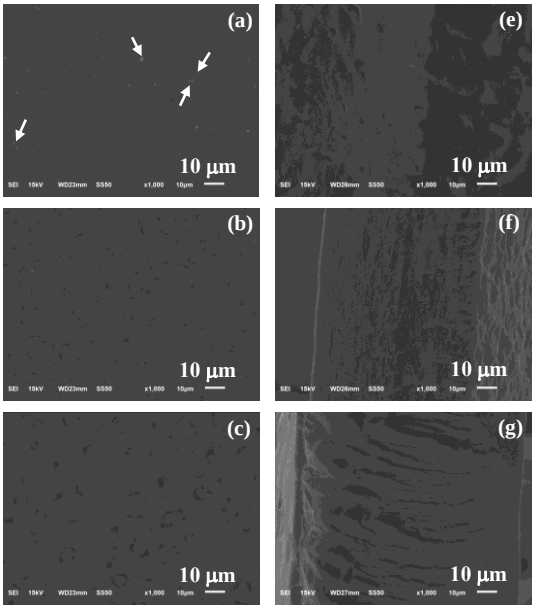

Figure 2 SEM micrographs at 1,000X of films prepared from modified cassava starches: (a) unmodified cassava starch (b) oxidized cassava starch and (c) cross-linked cassava starch as well as cross-sectional morphology at 1,000X of different films: (d) unmodified cassava starch (e) oxidized cassava starch and (f) cross-linked cassava starch

4.4 Thermal stability and decomposition temperature of biodegradable films from modified cassava starches

Figures 3A and 3B show weight loss and derivative thermogravimetry (DTG) of the samples as a function temperature range from 30-500 °C. All samples demonstrated three-step weight loss at temperature ranges of 36.2-146.7 °C, 153.9-279.9 °C, and 168.3-401.9 °C, corresponding to evaporation of water, degradation of glycerol as well as degradation of cassava starch, respectively (Figures 3Aa-3Ac) (Jaramillo, Gutiérrez, Goyanes, Bernal, & Famá, 2016). Weight loss at less than 250 °C of cross-linked cassava starch film (Figure 3Ac) was lower than the unmodified cassava starch and oxidized cassava starch films (Figures 3Aa-3Ab). This indicated that thermal stability of the cross-linked cassava starch film was higher than the control and oxidized cassava starch films at temperatures lower than 250 °C possibly due to less water molecules in the sample. However, at temperature above 250 °C, thermal stability of the cross-linked cassava starch film was lower than those of the control and oxidized cassava starch films, indicating low thermal resistance of modified cassava starch with cross-linking. This possibly due to the degradation of some starch molecules in cross-linked starch film when its cured at high temperature similar to the results were reported from Reddy and Yang (2010). They found that cross-linked corn starch film showed a higher weight loss than the non-cross-linked corn starch film at temperature of 220-320 °C (Reddy & Yang, 2010). Residual mass of biodegradable films from modified cassava starches (Figures 3Ab-3Ac) was higher than the control (Figure 3Aa), indicating higher impurities and inorganic components due to chemical substances used for oxidation (Zhang, Wang, Zhao, & Wang, 2012) and cross-linking (Marques et al., 2006). Decomposition temperature (T_d) of each component in the material was observed as peaks in the DTG thermogram (Figure 3B). The T_d of the control film was 326.5 °C (Figure 3Ba). The T_d of oxidized cassava starch film (326.4 °C) was similar to the control, whereas T_d of cross-linked cassava starch film was lower (308.0 °C) (Figures 3Bb-3Bc).

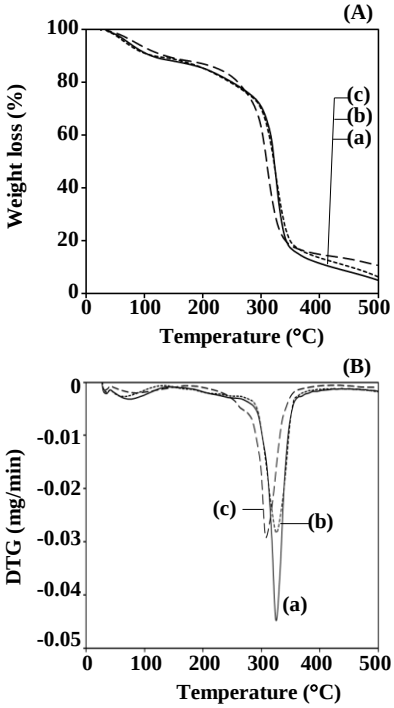

Figure 3 (A) Weight loss and (B) DTG of films prepared from modified cassava starches: (a) unmodified cassava starch (b) oxidized cassava starch and (c) cross-linked cassava starch using the TGA technique

4.5 Tensile properties of biodegradable films from modified cassava starches

Figure 4 shows tensile strength (Figure 4A), Young's modulus (Figure 4B) and elongation at break (Figure 4C) of samples. Tensile strength, Young's modulus and elongation at break of the unmodified cassava starch film were 0.73 MPa, 0.76 MPa, and 13.34 %, respectively (Figure 4a). Chemical modification (both oxidation and cross-linking) of cassava starch improved the tensile strength of films. Oxidized cassava starch film exhibited higher tensile strength and Young's modulus than the unmodified cassava starch film at 4.59 and 3.76 folds, respectively (Figure 4b). This implied that stronger film resulted from scission of some glycosidic linkages and introduction of bulky carbonyl groups in the starch molecules, making it difficult for the linear fractions to rearrange (de Pauli et al., 2011). Moreover, elongation at break of cross-linked cassava starch film increased to 2 folds (Figure 4c), suggesting extensible film because of the cross-linking network among the starch molecules (both inter and intramolecular linkages) as well as lengthened starch molecules (Xu, Canisag, Mu, & Yang, 2015).

4.6 Water absorption of biodegradable films from modified cassava starches

Table 3 demonstrates water absorption of different films. Water absorption of oxidized and cross-linked cassava starch films ranged at 201-222% and were lower than the unmodified cassava starch film (746%). This was explained by the increased interactions between the starch molecules as well as strong inter and intramolecular bonds promoted by oxidation and cross-linking, making the whole structure less water absorbent (Fonseca et al., 2015; Xu et al., 2015).

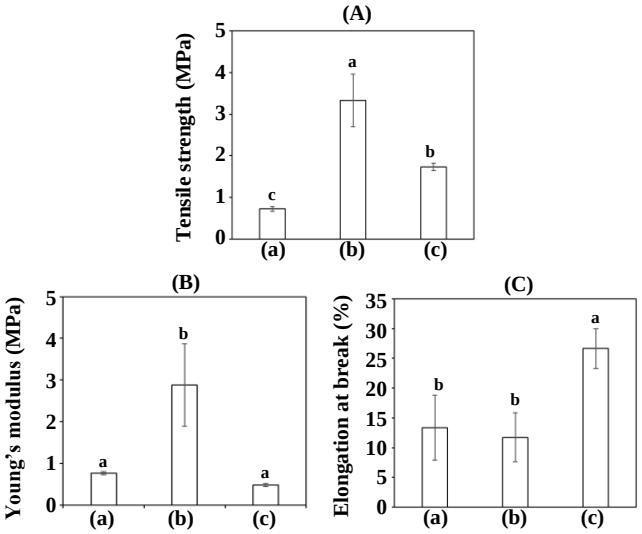

Figure 4 Tensile strength (A), Young's modulus (B) and elongation at break (C) of films prepared from modified cassava starches: (a) unmodified cassava starch (b) oxidized cassava starch and (c) cross-linked cassava starch. Data are reported as mean±SD (n=3). Different small letters in the bar graph indicate significant difference at $p < 0.05$ (Duncan's new multiple range test)

Table 3 Water absorption of films prepared from modified cassava starches

Sample	Water absorption percentage (× 100%)
Unmodified cassava starch	7.46 ± 1.25^{a}
Oxidized cassava starch	2.22 ± 0.13^{b}
Cross-linked cassava starch	2.01 ± 0.72^{b}

Data are reported as mean±SD (n=3). Different small letters in the same column indicate significant difference at $p < 0.05$ (Duncan's new multiple range test).

5. Conclusion

Both modified cassava starches; i.e. oxidized cassava starch and cross-linked cassava starch were prepared by the film casting method using 30% w/w of glycerol as a plasticizer. Thickness of modified cassava starch films ranged 0.06-0.16 mm. Oxidized and cross-linked cassava starch films showed high transparency. Oxidized and cross-linked cassava starch films recorded higher tensile strength, but lower water absorption than the control film due to chemically modified starch structures induced by oxidation and cross-linking. Oxidized cassava starch films had good transparency with a smooth surface, high strength and stiffness, whereas cross-linked cassava starch films exhibited high extensibility.

6. Acknowledgements

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