



## Pasting properties of heat–moisture treated canna starches using different plasticizers during treatment



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### ABSTRACT

Different plasticizers (propanol, propylene glycol, glycerol, erythritol, xylitol and sorbitol) were used for plasticizing canna starch during heat–moisture treatment (HMT). Pasting properties of the modified starches were determined and compared with those of native starch and of HMT starch using water as a plasticizer. Canna starch was soaked in 5% (w/w) plasticizer solutions and adjusted to 25% moisture content before heating at 100 °C for 1 h. The least change in paste viscosity was found when water was used as a plasticizer. Viscosity of the modified starches decreased as the molecular weight of plasticizers decreased. Plasticizer content in starch granules increased with decreasing molecular weight of the plasticizer, as well as with increased soaking time (from 10 min to 4 and 24 h). However, pasting profiles of HMT starches prepared by soaking for 4 h were comparable to those soaked for 24 h, indicating that there was an effective limit of plasticizers. The plasticizer content in starch granules played a greater role in HMT than the number of hydroxyl groups.

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### 1. Introduction

Heat–moisture treatment (HMT) involves the treatment of starch at elevated temperatures (90–120 °C) and at restricted moisture content (18–30% moisture w/w) for a certain period of time (Collado & Corke, 1999; Gunaratne & Hoover, 2002). The magnitude of change from such a treatment depends on a number of factors, including chemical composition and structure of starch, treatment time, treatment temperature, and moisture content. Moisture content of starch samples during treatment has been reported to be an important factor affecting the physicochemical properties of the modified products (Adebowale & Lawal, 2003; Collado & Corke, 1999; Watcharatewinkul, Puttanlek, Rungsardthong, & Uttapap, 2009). Water in starch samples acts as a plasticizer, rendering starch polymeric chains more flexible and thus facilitating the rearrangement of amylose/amylopectin unit chains, as evidenced by a greater magnitude of changes in paste viscosity, crystallinity and digestibility of HMT starches with higher moisture content

(Kulp & Lorenz, 1981; Olayinka, Adebowale, & Olu-Owolabi, 2008; Watcharatewinkul et al., 2009).

Plasticizers can be defined as low-molecular-weight substances that are incorporated into a polymer matrix to enhance the flexibility and processability by increasing the spacing between molecular chains and increasing segmental mobility, commonly through reducing the H-bonding between the polymer chains (Mathew & Dufresne, 2002). Besides water, hydrophilic molecules such as glycerol, sorbitol, xylitol and urea have been widely studied as plasticizers, especially in the production of thermoplastic starch (TPS) and starch film. During processing, starch granules are destroyed either by heating with excess water or by heating at relatively high temperature under high shear conditions and with a limited amount of water (Hulleman, Janssen, & Feil, 1998). Interaction of the plasticizer with the starch biopolymer replaces some of the starch–water interactions and reduces the intermolecular entanglements within the biopolymer by inducing stronger hydrogen bonding between the plasticizer and the biopolymer (Chang, Abd Karim, & Seow, 2006; Park & Chinnan, 1995). Glycerol is the most widely used plasticizer in starch-based materials, and is known to reduce the glass transition temperature and increase the flexibility of the derived products (Mathew & Dufresne, 2002). The effects

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of various plasticizers on TPS and starch film properties have been intensively studied and were recently reviewed by [Vieira, da Silva, dos Santos, and Beppu \(2011\)](#) and [Mohammadi Nafchi, Moradpour, Saeidi, and Alias \(2013\)](#).

The plasticizing phenomenon that occurs during HMT of starch differs from that of TPS or starch film, since the starch granules remain in an intact form throughout the treatment process. In this instance, plasticization of starch chains is influenced not only by interactions between the plasticizer and starch chains, but also by the ability of the plasticizer to penetrate into starch granules, as well as its diffusion efficiency through amorphous and semicrystalline regions inside the granules. Therefore, the extent of plasticization is dependent on both the granular structure of the starch (i.e. surface pores, packing/organization of starch chains, and ratio of amylose/amylopectin) and the chemical structure of the plasticizer, including molecular size, composition, and functional groups.

Earlier research related to modification of starch by HMT was solely undertaken using water as a plasticizer. To date, there have been no reports on the use of other plasticizers. We hypothesized that other plasticizers which interact more strongly than water with starch polymeric chains would exhibit a greater effect on the rearrangement of starch chains during treatment, resulting in a more pronounced change in starch properties. As a consequence, the treatment process could be enhanced, with a reduction in treatment time giving rise to more energy savings.

Therefore, the objective of the present study was to investigate the effect of plasticizers having different structures on the pasting properties and microstructure of HMT starch. As mentioned by [Mali, Sakanaka, Yamashita, and Grossmann \(2005\)](#), the most effective plasticizers will generally resemble most closely the structure of the polymer they plasticize. Thus, the plasticizers used in this study were mainly in the polyol group: propylene glycol (3C, 2OH); glycerol (3C, 3OH); erythritol (4C, 4OH); xylitol (5C, 5OH); and sorbitol (6C, 6OH). Propanol (3C, 1OH) was also investigated to assess the effect of the number of hydroxyl groups, while water was used as a reference. As a follow-up to our previous study ([Watcharatwinkul et al., 2009](#)), canna starch was chosen for the experiment.

## 2. Materials and methods

### 2.1. Materials

Edible canna was grown on experimental plots at the Rayong Field Crops Research Center, Rayong, Thailand. Eight-month-old rhizomes were harvested, and the starch was isolated according to a procedure described by [Puncha-arnon, Puttanlek, Rungsardthong, Pathipanawat, and Uttapap \(2007\)](#). Propanol (liquid,  $M_W$  60.10) and glycerol (liquid,  $M_W$  92.09) were purchased from QR&C (Johor Baharu, Malaysia). Propylene glycol (liquid,  $M_W$  76.09), erythritol (powder,  $M_W$  122.12), xylitol (powder,  $M_W$  152.15) and sorbitol (powder,  $M_W$  182.17) were obtained from Sigma-Aldrich (St. Louis, MO, USA).

### 2.2. Heat–moisture treatment of canna starch

#### 2.2.1. Standard procedure

Canna starch was adjusted to a moisture content of 25% by soaking 100 g of starch in 100 ml of water for 24 h at 25 °C with constant stirring. Excess water in the equilibrated slurry was drawn out by vacuum suction to obtain a cake with moisture content around 40%. The starch cake was then air-dried to allow the moisture content to drop to 25%. The obtained starch sample was placed in a 200 ml screw-capped bottle, heated at 100 °C for 1 h, and dried at 40 °C overnight.

#### 2.2.2. Effect of plasticizers

HMT canna starches were prepared following the standard procedure, except that individual samples were soaked in 5% w/w propanol, propylene glycol, glycerol, erythritol, xylitol or sorbitol solution instead of water.

#### 2.2.3. Effect of washing before HMT

HMT canna starches were prepared following the standard procedure, except that after soaking in plasticizer solution, the starch cake obtained was repeatedly washed (adding water and suction) three times to wash out the plasticizer solution before drying.

#### 2.2.4. Effect of soaking time

HMT canna starches were prepared following the standard procedure, except that the soaking time was changed from 24 h to either 10 min or 4 h.

### 2.3. Granule morphology

Native and HMT starch granules were stained with 0.2%  $I_2/KI$  and observed under a light microscope (Eclipse E200; Nikon, Tokyo, Japan).

### 2.4. Pasting properties

Starch slurries were prepared at a concentration of 8% (w/w) using distilled water as a dispersing medium (unless not specified). Pasting profiles of the slurries were determined by a rapid visco analyzer (RVA-3D; Newport Scientific, Warriewood, Australia) with a paddle rotating at a fixed speed of 160 rpm. Each starch slurry was heated from 40 to 92.5 °C at a rate of 3 °C/min, maintained at 92.5 °C for 15 min, and then cooled to 40 °C at the same rate.

### 2.5. Light microscopy of starch gels

Starch gels obtained from rapid visco analysis (RVA) experiments were stained with 0.2%  $I_2/KI$  and observed under a light microscope at 100 $\times$ .

### 2.6. Determination of plasticizer content in soaking solution

Canna starch (50 g) was soaked in 50 ml of 5% (w/w) plasticizer solution with constant stirring at 25 °C. Two-milliliter samples of starch slurries were taken at 0, 0.2, 1.0 and 2.0 h, and then every 2 h until 24 h. Each sample was centrifuged at 5000 rpm for 5 min. The supernatant was filtered through 0.45  $\mu$ m filter paper, and plasticizer remaining in the supernatant was determined by high-performance liquid chromatography (HPLC). The HPLC system consisted of a pump (LC-20AD; Shimadzu, Kyoto, Japan), an injector, a column (VertiSep<sup>TM</sup> OA, 8  $\mu$ m, 7.8  $\times$  300 mm; Vertical Chromatography, Nonthaburi, Thailand), a refractive index (RI) detector (RID-10A; Shimadzu), and a computer with data analysis software (CLASS-VP version 6.14 SP2; Shimadzu). Temperature of the column was maintained at 50 °C, and the flow rate of the mobile phase (filtered 0.005 N  $H_2SO_4$ ) was set at 0.8 ml/min. A 40  $\mu$ L sample of supernatant was injected into the HPLC system and the RI value was recorded. The concentration of plasticizer in the supernatant was calculated from the peak area of each sample compared with the peak area of the corresponding standard plasticizer solution.

### 2.7. Statistical analysis

All analyses were carried out in duplicate. The experimental data were analyzed using analysis of variance (ANOVA) and expressed as mean values  $\pm$  standard deviations. A Duncan test was conducted to

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