

Properties of subcritical water-hydrolyzed passion fruit (*Passiflora edulis*) pectin



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ABSTRACT

Pectin extracted from passion fruit (*Passiflora edulis*) using diluted nitric acid was further hydrolyzed in subcritical water to reduce its molecular weight using a batch-type reactor at different temperatures (100, 120, 140, and 160 °C) and heating rates (3.5 and 7.0 °C/min). The subcritical water-hydrolyzed pectins were investigated in their properties including viscosity, molecular mass, degree of esterification, and water adsorption isotherm. The results demonstrated that under more severe conditions the hydrolyzed pectin had lower viscosity and showed different solution characteristics. The molecular mass of pectin also decreased with the severity of subcritical water hydrolysis. The pectins dissolved in distilled water had higher intrinsic viscosities and molecular masses than those dissolved in buffer solution. Degrees of esterification of the hydrolyzed pectins only slightly changed in range of 55–60%. In addition, the moisture adsorption isotherms of the hydrolyzed pectins were evaluated and could be interpreted well with Guggenheim–Anderson–de Boer (GAB) model. The reduction of molecular mass did not significantly affect the water adsorption characteristics of the pectin.

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1. Introduction

Pectin is a heterogeneous anionic polysaccharide mostly found in the primary cell wall and middle lamella of higher plant. The structure of pectin consists of the smooth region which is covalently linked D-galacturonic acid, and the hairy region in which the ramification of other neutral sugars side chains are presented (Brejnholt, 2010). The physical properties of pectin depend on its structures such as chain length, type of branched chain, amount of

esterified group etc. Viscosity is one of the most important properties that must be considered before using pectin. It is affected by the degree of esterification (DE), molecular mass, and ionic strength in the solution (Kar & Arslan, 1999). Recently, we reported that the molecular mass of passion fruit pectin was significantly decreased after subcritical water treatment (Klinchongkon, Chanthong, Ruchain, Khuwijitjaru, & Adachi, 2016). It was reported that the decrease in molecular size of pectin tended to improve an intestinal absorption which might be useful for the cancer and kidney injury treatment (Courts, 2013), as well as promoted the digestion by beneficial bacteria in gastrointestinal tract (Gullón et al., 2013).

Pectin is a hygroscopic material that is often used in the food,

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drug and cosmetic industries as gelling, thickening, water binder, and stabilizing agent (Brejnholt, 2010). Because the initial moisture content of food affects the ingredient mixing step and quality of final product, the information of water sorption characteristics is important. Brejnholt (2010) reported that the equilibrium moisture of high methoxyl (HM) pectin is around 12% at the water activity of 0.7; moreover, most commercial pectin is dried to less than 10% of moisture content and stored in a vapour-tight packaging for good long-term storage. Tsami, Vagenas, and Marinos-Kouris (1992) reported that the water adsorption isotherm of pectin at 25 °C was well fitted with Guggenheim-Anderson-de Boer (GAB) equation. Panchev, Slavov, Nikolova, and Kovacheva (2010) demonstrated that the adsorption of water from pectic substance was influenced by multiple factors. The authors showed that the monolayer moisture content of sunflower pectin was between those of apple and citrus pectins even though the sunflower pectin had the lowest degree of methoxylation and molecular mass.

Pectin is commonly extracted by hot acid solution; however, a non-harmful extraction technique is gaining much interest nowadays. Subcritical water treatment has been applied for pectin extraction in the recent years (Chen, Fu, & Luo, 2015; Klinchongkon et al., 2016; Klinchongkon, Khuwijitjaru, Wiboonsirikul, & Adachi, 2017b; Martínez, Gullón, Schols, Alonso, & Parajó, 2009; Wang, Chen, & Lü, 2014). This technique is environmentally friendly because only water is used. Subcritical water is the water at the temperature between normal boiling point (100 °C) and critical point (374 °C), under the pressure that is high enough to keep the water in the liquid state. The effects of treatment temperature and reaction time of subcritical water treatment could be combined and expressed in term of “the severity factor” for the facility of data comparison (Klinchongkon et al., 2017b; Koomyart et al., 2016; Martínez, Yáñez, Alons, & Parajó, 2010).

Recently, we reported that subcritical water extraction is a feasible method for producing low molecular weight pectic polysaccharides or oligosaccharides from passion fruit peel directly (Klinchongkon et al., 2016; Klinchongkon, Khuwijitjaru, & Adachi, 2017a; Klinchongkon et al., 2017b). However, because the peel contain also cellulose and hemicellulose, therefore, to avoid the contamination of these component, we also investigated the use of pectin extracted by conventional method (hot acid extraction) as the initial raw material for subcritical water hydrolysis (Klinchongkon et al., 2017a). In the previous report, only hydrolysis kinetics was studied, thus, the objective of this study was to evaluate the properties (viscosity, molecular mass, DE, and moisture adsorption isotherm) of the subcritical water-hydrolyzed passion fruit pectin. In addition, solvent effect on viscosity and molecular mass measurements was also evaluated.

2. Materials and methods

2.1. Materials

Ripe passion fruit was purchased from a market in Pathumthani province, Thailand. Absolute ethanol (99.5%) was purchased from Junsei Chemical (Tokyo, Japan). Dextrans T10, T70, and T500 with weight-average molecular mass of 1.05×10^4 , 7.12×10^4 , and 5.07×10^5 Da, respectively, were purchased from Phadia (Uppsala, Sweden). CH_3COOK , MgCl_2 , K_2CO_3 , $\text{Mg}(\text{NO}_3)_2$, KI , NaCl , KCl , NaOH , HCl , Na_2HPO_4 , NaH_2PO_4 , HNO_3 , and phenolphthalein were purchased from Wako Pure Chemical Industries (Osaka, Japan). KOH , LiCl , and K_2SO_4 were purchased from Nacalai Tesque (Kyoto, Japan).

2.2. Preparation of passion fruit pectin

The passion fruit peel was cut into small pieces, washed with

clean water, blanched with hot water at 95 °C for 1 min, dried at 60 °C for 24 h in a hot-air oven, and milled to a smaller size using a food blender (Klinchongkon et al., 2016). The passion fruit pectin was extracted using diluted acid as reported in our previous work (Klinchongkon et al., 2017a). Briefly, the dried passion fruit peel was mixed with a 50 mM HNO_3 solution (1:50 w/v) in a capped glass bottle, and then heated at 80 °C for 25 min in a temperature controlled water bath. The hydrolysate was filtered through a nylon cloth, evaporated to reduce the volume using a vacuum rotary evaporator (N-1100, Eyela, Tokyo, Japan), and mixed with double volume of absolute ethanol. The mixture was kept in a refrigerator at 4 °C for 30 min. After that, the precipitated pectin was separated by filtering using nylon cloth and washed three times with absolute ethanol. Finally, the pectin was dried at 60 °C for 24 h and milled using a mortar and pestle. This pectin was used as an initial raw material for subcritical water hydrolysis.

2.3. Subcritical water hydrolysis

A 30 g/L of passion fruit pectin solution was prepared by gently dissolving the pectin (from 2.2) in distilled water under magnetic stirring for 5 h at room temperature. Then, the solution was centrifuged at 8.9×10^3 rpm, 4 °C for 10 min (LX-120, Tomy, Tokyo, Japan) to separate the insoluble solid. After that, 80 g of the solution ($M_w = 197$ kDa, DE = 60%, $\eta = 84.20$ cP) was added into a pressure-resistant stainless steel vessel (net volume 125 mL; Taiatsu Techno, Osaka, Japan). The vessel was tightly closed and then heated at 100, 120, 140, or 160 °C using a mantle heater (200 W, Heater Engineer, Tokyo, Japan) connected with a TXN 700B temperature controller (As One, Osaka, Japan). The temperature increasing rate varied at 3.5 and 7.0 °C/min. The vessel was immediately cooled in an ice bath after internal vessel temperature reached to the desired temperature. The temperature profile inside the vessel for each condition during heating and cooling processes is shown in Fig. 1. Since the treatments were operated non-isothermally, the effects of treatment time and temperature were expressed as the severity factor ($\ln R_0$) (Overend, Chornet, & Gascoigne, 1987), which is defined by Eq. (1):

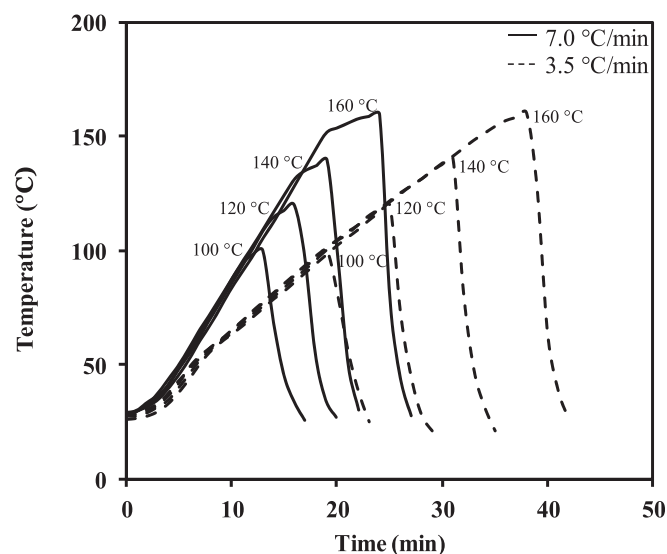


Fig. 1. Temperature profile of subcritical water hydrolysis at 3.5 and 7.0 °C/min.

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