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Effect of pre-dehydration treatment on the *in vitro* digestibility of starch in cookie

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ABSTRACT

In order to understand the effect of pre-dehydration on the *in vitro* digestibility of cookie starch, cookie dough samples were dehydrated by vacuum treatment, and melting temperature (T_m) of the crystalline amylopectin in the dough, internal temperature and water content of the dough during baking, and non-hydrolysed starch content of the obtained cookies were investigated. The T_m of crystalline amylopectin increased with decreased water content of the dough, and the result was described as a T_m-curve. The internal temperature of non-dehydrated dough surpassed the T_m-curve during baking. Pre-dehydrated dough, on the other hand, always indicated a lower internal temperature than the T_m-curve. The non-hydrolysed starch content obtained under a given condition increased significantly with a decrease in the initial water content of cookies. This will be because the melting of crystalline amylopectin was prevented, at least partially, during baking.

1. Introduction

Starch is composed of amylose, amylopectin, and other minor ingredients (*e.g.*, lipid and phosphate) depending on the botanical origin (Jacobs & Delcour, 1998; Tester, Karkalas, & Qi, 2004). The amylose content of waxy, normal, and high-amylose starch is less than 15%, 20–23%, and approximately 60%, respectively. Amylose is an essentially linear polysaccharide, and the degree of polymerisation (DP) is in the range of 500–6000 glucose units. Amylose usually exists in an amorphous form in starch granules, but can also be observed as an amylose–lipid complex. On the other hand, amylopectin is a large branched polysaccharide, with a DP in the range of 3×10^5–3×10^6 glucose units. Amylopectin consists of both crystalline (double helix) and amorphous parts. When starch is heated to a certain temperature, the crystalline amylopectin melts and converts to the amorphous form. The physical state of amylopectin affects its digestibility, because crystalline amylopectin is more stable chemically and physically than the amorphous form. Reduction of starch digestibility has attracted much attention in recent years with respect to potential beneficial effects on metabolic responses (Dona, Pages, Gilbert, & Kuchel, 2010; Fuentes-Zaragoza, Riquelme-Navarrete, Sánchez-Zapata, & Pérez-Álvarez, 2010; Hoover & Zhou, 2003).

Recent efforts to understand the digestibility of starch have led to the development of *in vitro* starch digestibility assays (Champ, 1992; Englyst, Kingman, & Cummings, 1992; Goñi, García-Diz, Mañas, & Saura-Calixto, 1996; McCleary, McNally, & Rossiter, 2002; Perera, Meda, & Tyler, 2010). Englyst et al. (1992) classified starch into rapidly digested starch, slowly digested starch, and resistant starch. Rapidly digested starch is starch that is readily converted to glucose. Many starchy food products contain a large amount of rapidly digested starch. Slowly digested starch is resistant to digestion, but can be completely converted to glucose; this contributes to a reduction of the glycemic response (Englyst, Englyst, Hudson, Cole, & Cummings, 1999). Resistant starch is a type of starch that is not hydrolysed by digestive enzymes. Reduction of the glycemic response and production of colonic butyrate fermentation by intestinal flora have been reported as associated health benefits (Brouns, Kettlitz, & Arrigoni, 2002; Fuentes-Zaragoza et al., 2010).

The susceptibility of starch to digestion depends on crystalline type (A- or B-pattern), amylopectin chain length, starch granule size, and amylose content (Chung, Liu, Lee, & Wei, 2011; García-Alonso, Jiménez-Escrig, Martín-Carrón, Bravo, & Saura-Calixto, 1999; Hu, Zhao, Duan, Linlin, & Wu, 2004; Lu et al., 2011; Themeier, Hollmann, Neese, & Lindhauer, 2005). The crystalline amylopectin in cereal starch (*e.g.*, corn, wheat, and rice) and in tuber starch (*e.g.*, potato) usually contributes to slowly digested starch and resistant starch, respectively. Nevertheless, the application of crystalline amylopectin is limited to non-heated food products, because crystalline amylopectin melts almost completely when starch is heated in the presence of substantial water. On the other hand, crystalline (retrograded) amylose, which plays a role in resistant starch, is characterised by a high melting temperature (T_m = 104–158 °C), even in the presence of substantial water. Thus,

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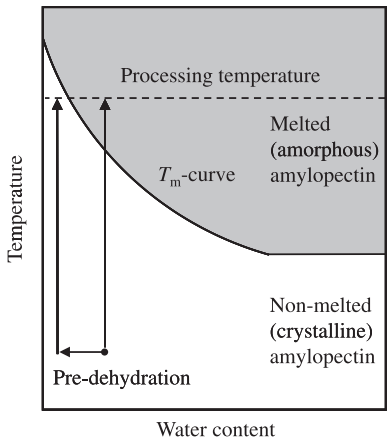

Fig. 1. Typical T_m-curve of crystalline amylopectin in a starchy product.

crystalline amylose has attracted much attention as a source of resistant starch in food products (Haralampu, 2000; Shamai, Bianco-Peled, & Shimoni, 2003; Sievert & Pomeranz, 1989). However, amylose usually exists in an amorphous form in starch granules, and thus the degree of crystallisation must be enhanced by further treatments (*e.g.*, autoclave treatment). In addition, amylose is a secondary constituent of starch, and thus a specific starch (*e.g.*, high amylose starch) is required for industrial production.

It is known that T_m of crystalline amylopectin increases with a decrease in water content (Biliaderis, Page, Maurice, & Juliano, 1986; Roos, 1995; Tester & Debon, 2000). Thus, the melting of crystalline amylopectin in low-moisture starchy food products can be partially prevented, depending on the type of thermal processing involved (Bravo, Englyst, & Hudson, 1998; Goñi, Bravo, Larrauri, & Saura Calixto, 1997; Kingman & Englyst, 1994; Primo-Martín, van Nieuwenhuijzen, Hamer, & van Vliet, 2007). Taking this into account, the remaining crystalline amylopectin in low-moisture starchy food products would increase with a decrease in the initial water content (Fig. 1). Furthermore, these types of products are usually eaten without secondary heating, and thus, the remaining crystalline amylopectin in the products will be ingested. Consequently the digestibility of low-moisture starchy food products can be reduced by dehydration before thermal processing (pre-dehydration).

In order to gain insight into the suggestion, cookies were employed as a typical low-moisture starchy food product, and the effect of pre-dehydration on crystalline amylopectin T_m in the cookie dough was investigated using differential scanning calorimetry (DSC). Furthermore, internal temperature and water content changes of the cookie dough during baking, as well as *in vitro* digestibility of the obtained cookies, were investigated, and the relationship between crystalline amylopectin melting during baking and the *in vitro* digestibility of cookies was discussed.

2. Materials and methods

2.1. Preparation of cookie dough and cookies

Wheat flour, unsalted butter, sugar, and egg were purchased at a local market. The butter (80 g), whole egg (50 g), and sugar (120 g) were mixed and wheat flour (200 g) was added with brief stirring. The mixture was covered with plastic wrap (polyvinylidene chloride) and held at 4 °C for 2 h. Food composition of the materials and dough is listed in Table 1. The dough was formed into columns (φ 30 mm × 5 mm), and dehydrated by pressure vacuum below 8 kPa at 30 °C for up to 6 h. The dehydrated dough samples were closed into a polyethylene bag, and stored at 4 °C for at least 16 h in order to equilibrate water distribution in the dough.

Table 1
Composition of the cookie dough sample and its constituents (%, w/w on a wet basis).

	Water	Protein	Fat	Carbohydrate	Ash
Unsalted butter[a]	15.8	0.5	83.0	0.2	0.5
Sugar[a]	0.6	0.0	0.0	99.4	0.0
Wheat flour[a]	12.0	8.2	1.7	77.6	0.5
Whole egg[b]	76.1	12.3	10.3	0.3	1.0
Cookie dough[c]	16.8	5.1	16.6	61.1	0.4

[a] From the label of product.
[b] From a general food composition database.
[c] Calculated value.

The cookie dough samples were baked using a drying oven (OFW-300S, As One Co., Osaka, Japan). The oven was adjusted to 180 °C, and the cookies were placed on a metal plate and baked for 12 min. The obtained cookies were cooled to room temperature, and stored in a polyethylene bag for at least 24 h. For comparison, non-heated cookie, which was vacuum-dried for 68 h, was also prepared.

2.2. DSC measurement

The T_m of crystalline amylopectin was investigated using a DSC (DSC120; Seiko Instruments Inc., Tokyo, Japan). Alumina powder was used as a reference, and the temperature and heat flow were calibrated with indium and distilled water. Individual dehydrated cookie dough samples were homogeneously mixed before measurement. The mixture (20–50 mg) was hermitically sealed in a pressure-resistant aluminium pan. For comparison, T_m of crystalline amylopectin from a wheat flour–water mixture was also investigated. In order to adjust the water content of the wheat flour, dried wheat flour was held under various relative humidity conditions at 25 °C. The equilibrium of water sorption was confirmed gravimetrically, and the wheat flour–water mixture (3–10 mg) was hermitically sealed in the pan. Heat scanning was performed at 5 °C/min in the temperature range between 0 and 200 °C. The DSC thermogram was analysed using EXSTAR6000 interfaced with the DSC. All tests were performed in triplicate and the results averaged.

2.3. Polarised microscopic study

The DSC thermogram of cookie dough samples showed multiple peaks, as shown in a later section. In order to identify the melting peak of crystalline amylopectin from the DSC thermogram, a polarised microscopic study was carried out. DSC scanning was stopped at several temperatures, and the sample was removed from the DSC pan. The removed sample was set on a glass plate, and polarised crosses due to crystalline amylopectin was observed microscopically (BH-2 connected U-PMTVC; Olympus, Co., Tokyo, Japan).

2.4. Water content

Water content of the samples was examined gravimetrically by drying at 105 °C for 18 h. All tests were performed in duplicate or triplicate and the results averaged.

2.5. Baking process of cookie dough

In order to understand the temperature change of dough during baking, a thermocouple (type K, φ = 1 mm) was inserted into the centre part of the cookie dough, and the dough's internal temperature was measured during baking (LR8100E; Yokogawa Electric