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Effects of extrusion treatment on physicochemical properties and *in vitro* digestion of pregelatinized high amylose maize flour

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A B S T R A C T

High amylose maize flour (HAMF), normal maize flour (NMF), extruded normal maize flour (ENMF) and extruded high amylose maize flour (EHAMF) were employed to analyze the effects of the extrusion process on their physicochemical properties and *in vitro* digestion. The disruption degree of NMF and HAMF under extrusion followed the degree of gelatinization order. HAMF and EHAMF had higher onset temperature, peak temperature and conclusion temperature but lower melting enthalpies in comparison with NMF and ENMF. Extrusion treatment decreased viscosity and transformed A-type and B-type starch in NMF and HAMF into B + V-type. The solubility of all samples and swelling power of NMF and HAMF showed an increase, while swelling power of extruded maize flour decreased with increasing temperature. HAMF and EHAMF had a higher content of resistant starch but a lower content of slowly digestible starch and rapidly digestible starch than NMF and ENMF.

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1. Introduction

Maize is a rich nutritional source of vitamins, minerals and functional elements, which can meet high demands of the growing society. Starch is the major component of maize accounting for about 70%. Normal maize starch is commonly composed of approximately 25% amylose and 75% amylopectin. The starch with more than 50% amylose is defined as high amylose starch. It is beneficial for industrial and food applications and it has been widely used as a bonding agent, setting agent, corrugated case, degradable plastic, food additive, packing material, and cream or gelatin substitution, etc. Some previous papers have reported extrusion parameters of high amylose maize starch and properties of extruded high amylose maize starch. Lai and Kokini (1992) estimated the viscous heat effects in slit flows of 98% amylopectin and 70% amylose corn starches under extrusion treatment. Shrestha et al. (2010) studied the enzymatic resistance and structural organization in extruded high amylose maize starch. However, high amylose flour is different from high amylose starch in application and processing due to their different components and properties. To investigate physiochemical properties, *in vitro* digestibility of extruded high amylose flour may provide much more

Abbreviations: AACC, American Association of Cereal Chemistry; AOAC, Association of Official Analytical Chemists; DG, degree of gelatinization; DSC, differential scanning calorimeter; EHAMF-I, extruded high amylose maize flour with initial moisture content of 12.47%; EHAMF-II, extruded high amylose maize flour with initial moisture content of 19.00%; ENMF, extruded normal maize flour; HAMF, High amylose maize flour; NMF, normal maize flour; RDS, rapidly digestible starch; RS, resistant starch; RVA, rapid visco-analyser; SDS, slowly digestible starch; SEM, scanning electron microscope; SOL, solubility; SP, swelling power; T_0, onset temperature; T_p, peak temperature; T_c, conclusion temperature; ΔH, enthalpy change; ΔT, the difference between conclusion temperature and onset temperature.

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valuable information for processing of high amylose puffed food.

One way to overcome the stability of high amylose maize is using extrusion cooking. It has been widely used to produce expanded snack foods, ready-to-eat cereals and pet foods in recent years. The high temperature with short time extrusion cooking has a significant effect on modification of the physicochemical properties of starch-based raw material. The thermal energy released by viscous dissipation during extrusion quickly changes the quality of raw material including viscosity, internal structure, texture, digestibility, palatability, etc. Specifically, the quality of products considerably depends on material sources as well as on processing parameters such as extruder type, screw configuration, feed moisture, temperature profile in the barrel sections, screw speed and feed rate. In the case of extrusion treatment on biopolymers, raw materials have to experience a necessary phase transition, in which the sudden pressure decrease induces certain amount of water to vaporize which shapes an expanded porous structure (Padmanabhan and Bhattacharya, 1989).

However, starch structure becomes partially destroyed and changes into more easily digestible forms altering its enzymatic resistance as a result of the extrusion treatment. Many other factors could make contribution to enzymatic resistance such as high pressure cooking/extrusion, heating time, temperature, amylose content, moisture content, and the presence of additives including sugar, lipid, and dietary fiber (Escarpa et al., 1997). According to its sensitivity to enzymatic digestion, starch can be divided into three types: rapidly digestible starch (RDS), slowly digestible starch (SDS) and resistant starch (RS). RDS is known as rapidly and completely digested in the small intestine, while SDS is considered as slowly but completely digested in the small intestine. RS has been defined as the sum of starch and products of starch degradation not absorbed by small intestine of healthy individuals in the framework of EURESTA (Kendall et al., 2004). When digested, RS induces less starch being converted to glucose resulting in decreasing glycemic index. It potentially influences on diabetes and energy intake provides fermentable carbohydrates for colonic bacteria and makes short fatty chain fatty acid beneficial for the colon and physiology. However, major RS types, like native starch granules (RS2) and retrograded or recrystallized starch (RS3) may be partially or completely destroyed under processing treatment. It is beneficial that RS2 mainly originates from high amylose maize which could retain a higher RS level after processing. For this reason, it is commonly regarded as an essential source of foodstuff with higher RS standards (Htoon et al., 2009).

Here, we designed a study, for the first time, to combine high amylose maize flour with extrusion methodology and to explore the effect of extrusion treatment on physicochemical properties. In particular, extruded high amylose maize flour with different moisture content has been characterized in the context of significant gap of degree of gelatinization, and *in vitro* digestion. The main parameters studied included morphology, thermal properties, pasting characteristics, crystal structure, solubility and swelling power. In addition, digestibility was also determined to find out the quantitative change of RDS, SDS, RS. A comparison was made between normal and high amylose maize flour subjected to extrusion treatment. It provided statistical evidence to evaluate a possibility of using high amylose maize flour as a raw material to produce HAMF-based puffed foodstuff with nutritionally higher RS levels.

2. Materials and methods

2.1. Materials

High amylose maize and normal maize cultivars were provided by the Maize Biology and Genetic Breeding Laboratory in Northwest Arid Areas, Ministry of Agriculture, Northwest A&F University. Maize kernels were subjected to peel and germ removal using an LG-36 peeling machine (Qufu Lin Gong Machinery Co, Shan dong Province, China), and were subsequently ground to flour using a DF-35 continuous milling machine (Wenling Linda Machinery Co, Zhejiang Province, China) and passed through a mesh with 100 μm pores for analysis. High amylose maize flour with moisture content 12.47% was extruded to make extruded high amylose maize flour (EHAMF)-I and high amylose maize flour with moisture content 19.00% was treated under the same extrusion conditions to make extruded high amylose maize flour (EHAMF)-II. They were produced to ensure an adequate gap of degree of gelatinization (the ratio of the amount of gelatinized starch to the amount of total starch) with a co-rotating twin screw extruder (DS32-II, Saixin Machinery Co, Shan dong Province, China) using a feed rate of 225 g/min, temperature of 150 °C, and screw rotational speed of 225 rpm. Extruded normal maize flour (ENMF) was also attained at the same extrusion conditions.

Pancreatin from porcine pancreas (Cat. No. P-1625, activity 3 × USP/g) was purchased from Sigma–Aldrich Chemical Company (St. Louis, Mo, USA). Amyloglucosidase (EC 3.2.1.3, 3260 Units/mL) and glucose oxidase-peroxidase assay kit (Cat. No. K-GLUC) were acquired from Megazyme (Megazyme International Ireland Ltd., Bray, Ireland).

2.2. Chemical composition analysis

Moisture, ash, fiber and lipid content were determined according to the protocols (AOAC, 1995). Protein content was measured using an automatic protein analyzer (Kjeltec 8400, Foss Co, Germany) with nitrogen conversion factor of 6.25. Total starch content was calculated using the HCl hydrolysis using a 3,5-Dinitrosalicylic acid (DNS) colorimetric method (Guoquan and Xiuling, 2002). Amylose content was determined with a dual wavelength iodine binding technique (Zhu et al., 2008). Amylose–iodine complex formation was used to determine the degree of gelatinization (Juansang et al., 2012).

2.3. Granular morphology

The morphological features of maize flour were observed with a Hitachi S-3400N field emission scanning electron microscope (Hitachi Ltd, Tokyo, Japan) equipped with Quartz PCI digital image acquisition software (Quartz Imaging Corp., Vancouver, BC, Canada). The flour samples were loaded on a metal plate with double-sided adhesive tape, coating with gold: palladium (60:40) using a Polaron SC500 sputter coater (Quorum Technologies, East Sussex, UK), and examined at 15.0 kV accelerating voltage and 500–3000 times magnification.

2.4. Thermal properties

Gelatinization characteristics of maize flour were measured using a differential scanning calorimeter (Q2000 DSC, TA Instruments, New Castles, DE). Each flour sample (about 3 mg dry weight) with excess water (1:3) was heated from 30 °C to 150 °C at 10 °C/min rate in sealed aluminum pans, using an empty pan as the reference. The sample was analyzed in triplicate and the data were calculated by Universal Analysis 2000 (TA Instruments, New Castles, DE).

2.5. Pasting properties

The pasting properties of maize flours were measured with a Rapid Visco-Analyser (RVA-SUPER 3, Newport Scientific,

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