Chemical Compositions Changes during Hot Extrusion at Various Barrel Temperatures for Porang (Amorphophallus Oncophyllus) Tuber Flour Refining

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Abstract. Porang tuber flour has unique physicochemical and biological properties that promote its uses for various applications, such as in food, drink, drug and cosmetics. These unique properties are mainly due to the presence of glucomannan in the flour. The current methods to manufacture high quality porang tuber flour are the wet and dry methods. Although wet methods may result in high purity porang tuber flour, they are very tedious, time consuming and require high manufacturing cost. This research aimed to develop an efficient dry method to produce high quality porang tuber flour by implementing hot extrusion process. Porang tuber flour with 20% (w/w) moisture was extruded in a single screw extruder by operating 150 rpm screw speed at various barrel temperatures. Changes in carbohydrates and other components were observed to find the best operating temperature. The results showed that starch and soluble sugar were the most affected components, while glucomannan was stable at low barrel temperatures and only changed its composition at 160 °C. The content of fat and ash were found to be less affected by extrusion than that of protein. The best barrel temperature was 140 °C where highest glucomannan content in the porang tuber flour was obtained.

1. Introduction

Porang flour, commonly referred to as konjac glucomannan, is a β-1,4 linked D-mannose, and D-glucose associated through the C-3 of D-mannose and D-glucose with an approximate degree of branching of 8%. The molar ratio of mannose and glucose in this biopolymer is about 1.6:1 [1]. The chain has a 5 – 10% acetyl group substitutes located at every 9 to 19 sugar units, which is responsible to the solubility and gelling properties [2]. With respect to its specific rheological and gelling properties, Porang flour can be used as a gelling agent, thickener, emulsifier, and stabilizer in various foods, drinks and cosmetic products [4]. Literature survey revealed Porang flour can be derived from the tubers of Amorphophallus plants, which belongs to the Aracea family. The *Amorphophallus oncophyllus* has been reported to be the most essential source due to its higher glucomannan content and sustainable availability [7]. Freshly harvested porang tuber contains about 8 to 10% (w/w) of glucomannan [2]. The flour, which constitutes 60 – 80% of the dried root tuber, is obtained by a dry milling process of thin tuber chips. The crude porang tuber flour may contain glucomannan in the range of 50 to 70% (w/w) [8], whereas purified porang tuber flour should have glucomannan content of no less than 90% (w/w). The impurities trapped in the crude porang tuber flour particles are usually coming from the tissue space [4], which mainly consists of starch, lipid, protein and ash [2]. Starch is
the main impurity that seriously affects the purity and quality of glucomannan, such as reducing viscosity and increasing turbidity [9].

The common processing methods to obtain high purity porang flour are by dry or wet processing, which can split the starch from the glucomannan granules. The dry processing method involves milling of dried porang tuber chips into crude porang flour, which is followed by separation using wind-sifting [10]. Porang flour obtained using this method is usually of low purity and is therefore sold as a low price food commodity [11]. Wet methods, which employ acid or enzymatic hydrolysis [12, 13], dialysis and ethanol precipitation [14], washing with water, aqueous alcohol [15] and benzene-ethanol solution [16], high energy centrifugation [8] or a combination of the aforementioned methods, may produce high purity of glucomannan. However, these methods are very tedious, time consuming and require higher operating cost [4]. Regard to the abovementioned facts, it is clear that the development of more efficient technologies are needed to substitute the existing porang flour refining technology.

The development of dry processing method is interesting because it offers simplicity and low operating cost. The high-speed grinder can be used to disintegrate the softer starch granules and peripheral corn fiber cells into very small, ash-like, particles. During this process, the glucomannan granules are usually not broken up because they are harder. Due to the difference in size and weight, glucomannan could be separated from starch particles by cyclonic separation and sifting [11]. To obtain konjac flour with good physical and chemical properties, the browning, case hardening and gelatinization must be avoided during dry processing methods [17]. The browning and case hardening may affect the whiteness of konjac flour, while gelatinization may lead to the serious reduction of viscosity. Porang tuber flour may undergo gelatinization at 68.5 – 77.6 °C with enthalpy of gelatinization of 16.6 J/g dry starch [18]. At present, the traditionally manufactured porang flour often exhibits such drawbacks: discolorization, excessive sulfur content and low viscosity. This study aims to investigate the effect of temperature and reaction time on the swelling power and water solubility of gadung starch during heat moisture treatment process. The results of this study are expected to be one of probing parameters in searching the possible utilizations of gadung starch in the food industry.

The extrusion process is an efficient continuous process, which distinctively incorporates some unit operations, viz: mixing, shearing, heating, pumping, forming, and sizing. This process is a high-temperature, short-time, versatile, and modern unit operation that convert materials, usually in a granular or powdered form, into specific products with enhanced textural and structural attributes [19]. Extrusion cooking has been used for processing of starch as well as proteinaceous materials for a long time [20]. Low-moisture extrusion, which provided high temperatures and shear rates, enhanced degradation of starch and the formation of dextrins [21]. The total residence time in the extruder should typically be in the range 2–5 min. In this period, the starch must be cooked (gelatinized) and the modification reaction taken to the desired extent of completion [22]. Generally, the gelatinization of high amylose starch requires high processing temperatures than that for low amylose starch, which imposes changes on reaction conditions. The typical process temperatures for low amylose starch are about 70–80 °C. Those for high (>50%) amylose starch can range 120–140 °C [22]. With amylose content of 19.9%, porang tuber flour falls under normal starch category [18]. Degradation of starch via extrusion process followed by wind-sifting will be likely one of the best combined processes to refine porang tuber flour. Some extrusion parameters such as barrel temperature, feed rate, screw speed, moisture content and die geometry have been reported to greatly affect the extrusion performance [23-24]. This research aims to study the changes in chemical compositions during hot extrusion of porang tuber flour under various barrel temperatures, where no previous studies have ever been reported.

2. Materials and Method

2.1. Materials

The material used in this research was crude porang tuber flour purchased from PT. Prima Agung Sejahtera-Surabaya (Indonesia) with average particle size of 80 mesh. All of chemicals and reagents
used for analysis were of analytical grade (purity ≥ 98% w/w) and procured from Sigma Aldrich through an authorized chemicals distributor in Semarang-Indonesia. All the chemicals were directly used without prior treatments.

2.2. Porang flour extrusion
The porang tuber flour extrusion was carried out in a single screw extruder (Figure 1). The extruder design was similar to the C.W. Brabender Model 2802. The feed was conditioned to 20% (w/w) moisture content by spraying distilled water as a fine mist onto starch in a Patterson V-blender. The extruder was operated at constant screw speed of 150 rpm under steady state condition. Accomplishment of steady state condition was determined by a constant torque required to turn the screw and by a regular extrusion rate. The feed zone barrel temperature was 80 °C, and the compression and die zone temperatures were varied from 80 °C to 160 °C and were controlled by electrical resistance band heaters. Prior to analyzes, the product was frozen in liquid nitrogen and then immediately ground in a Waring Blender followed by finer comminution in a coffee grinder. Since some analyses were conducted several weeks after the extrusion process, the samples were dried for preservation. The starch and soluble sugar was separated from glucomannan by wind-sifting method.

Figure 1. The typical cross sectional of single screw extruder [25]

2.3. Analytical methods
Starch content of crude porang tuber flour was determined by acid hydrolysis method [26]. The Kjeldahl method was used to obtain the total nitrogen content for protein determination [27]. The protein content was then calculated by applying the nitrogen conversion factor of 5.7, as proposed by the U.S. Food Chemicals Codex (FCC) and European Commission [10]. The moisture, ash, fat and dietary fiber contents were determined according to AOAC Method 934.01, 920.153, 948.15 and 991.43, respectively [28]. The reducing sugar of the sample was determined using phenol-sulfuric acid colorimetry [29]. Glucomannan content was calculated based on the reducing sugar content of the hydrolyzate and porang flour sample solution following the method previously used by Chua et al. [30]. Soluble sugars were determined by High Performance Liquid Chromatography (HPLC) according to Berrios et al. [31].

3. Results and Discussion

3.1. The proximate compositions of porang tuber flour
The proximate compositions of crude porang tuber flour used in this study are tabulated in Table 1. Although the main component of the flour is glucomannan, the flour also contains considerable amount of protein and starch, which theoretically will affect flour quality. It is clear that the crude
porang tuber flour meets the standard of porang tuber flour set by Food and Nutrition Board Committee on Food Chemicals Codex [32]. The glucomannan content in the crude porang tuber flour was within the range of glucomannan content of porang flour produced in Thailand [8]. However, the value was far below the glucomannan content in crude porang tuber flour manufactured in China [15]. Further, Xu et al. [15] also reported that no fat content was detected in crude porang tuber flour used in their study. Coincidentally, the fat content found in this study was very close to that of crude porang tuber flour from Japan [2].

**Table 1.** The proximate composition of crude porang tuber flour (g/100g).

| Components    | This work | Xu *et al.* [15] | Takigami [2] | CODEX [32] |
|---------------|-----------|------------------|--------------|------------|
| Glucomannan   | 50.62     | 74.13            |              | ≥ 75.0     |
| Starch        | 21.62     | 8.89             | 82.6         | ≤ 8.0      |
| Soluble sugar | 4.27      | 2.47             |              | ≤ 5.0      |
| Protein       | 6.53      | 3.67             | 2.2          | -          |
| Fat           | 2.02      | 0                | 2.3          |            |
| Ash           | 4.84      | 4.26             | 5.2          |            |
| Fiber         | 0.44      | 0                | 0.5          |            |
| Moisture      | 9.67      | 9.5              | 7.2          | ≤ 15.0     |

Although the protein content fell within the range of that determined by Food and Nutrition Board Committee on Food Chemicals Codex [32], the values is the highest among the protein content data shown in Table 1. The starch content was also very high indicating that a more extensive starch removal is needed to obtain high purity porang tuber flour. Ash, the total amount of inorganic composition, is a critical parameter for determining quality. In the food production, including glucomannan, ash is strictly essential to be kept in a controlled range; otherwise, the material may be categorized as contaminated or disqualified. The differences in chemical composition of porang flour may be influenced by ecological environment, growing condition and harvest time [33]. In addition, the use of fertilizers, shades, planting densities and the type of production process also may significantly affect the chemical composition of porang flour [34].

### 3.2. Effect of barrel temperature on chemical compositions of extruded porang tuber flour

Extrusion cooking has been used for processing of starchy as well as proteinaceous materials for a long time [21]. Macromolecular degradation during extrusion is known to occur as a function of temperature, feed moisture and screw speed [35]. The effect of barrel temperatures on the chemical composition of porang tuber flour is presented in Table 2.

**Table 2.** The chemical composition of crude porang tuber flour extruded at various barrel temperatures (g/100g).

| Components    | Raw   | 80°C | 100°C | 120°C | 140°C | 160°C |
|---------------|-------|------|-------|-------|-------|-------|
| Glucomannan   | 50.62 | 50.62| 50.62 | 50.62 | 50.62 | 47.97 |
| Starch        | 21.62 | 17.65| 15.27 | 10.77 | 5.58  | 5.49  |
| Soluble sugar | 4.27  | 8.24 | 10.62 | 15.12 | 20.21 | 23.04 |
| Protein       | 6.53  | 4.57 | 4.38  | 4.09  | 3.34  | 3.92  |
| Fat           | 2.02  | 2.00 | 1.99  | 1.98  | 1.56  | 1.52  |
| Ash           | 4.84  | 4.47 | 4.29  | 3.92  | 3.69  | 3.68  |
| Fiber         | 0.44  | 0.44 | 0.44  | 0.44  | 0.44  | 0.44  |
| Moisture      | 9.67  | 9.67 | 9.62  | 9.67  | 9.57  | 9.56  |

Glucomannan was not thermally degraded at temperatures of 140 °C or below, and its degradation was likely to begin at 160 °C. Starch started to degrade at 80 °C as observed by reduction of its content in the extruded porang tuber flour. This phenomenon is plausible because porang tuber flour
gelatinizes at 68.5 – 77.6 °C [18]. This process is accompanied by rupture of intermolecular bonds, resulting in rupture of starch grains [36]. Previous research on corn starch extrusion revealed that the extent of molecular degradation of starch increased with increasing barrel temperature [37]. Some hydrolysis might occur during extrusion of starch resulting in maltose, glucose and arabinose [38]. Indeed, the starch degradation took place and produced water soluble carbohydrates as indicated by increases in soluble sugar contents. Heat treatment of starch suspensions at high temperature and pressure may result in degradation of starch to lower molecular weight carbohydrates [39]. The formation of anhydroglucose end-chains has also been observed in extruded starch [40]. No fiber degradation was found in this study. However, severe extrusion-cooking of flours may also cause an apparent increase in dietary fiber due to the formation of amylase-resistant starch [41]. The fat content decreased slightly and was too small to be nutritionally significant, which was probably as a result of oxidation, hydrogenation, cis-trans isomerization or hydrogenation [42]. However, since the fat content is originally very low, then the oxidation of fatty acids during extrusion is minimal [43]. Therefore, Hagenimana et al. stated that the decrease in crude fat content occur through the lipids forming complexes with starch and protein in the extrusion process. Total protein changes very little during most extrusion operations [44]. Maillard reactions between ε-NH₂ groups of lysine residues and C=O groups of reducing sugars occur during extrusion particularly at high barrel temperature, low moisture, and high shear [45]. The ash content decreased gradually as the temperature increases. Less information is available regarding the bioavailability of minerals in food as reflected by ash content after extrusions [46]. Minerals are heat stable and unlikely to become lost in the steam distillate at the die [47, 48]. Therefore, the thermoplastic extrusion process did not reduce this nutrient [49]. A slight reduction in moisture content was also observed in this extrusion study. At high temperature the vapor pressure of the free moisture is also greater which would cause an increased rate of moisture flashing and puffing up on exit from the die [50].

During extrusion, the starch granules were disintegrated into tiny, ash-like, particles, while the glucomannan granules were generally not broken up as they were harder. The glucomannan, starch and soluble sugars granules differ in size, density and hardness, and thus can be separated by cyclonic separation and sifting [11]. The density and size of glucomannan particles are within 1,8120 kg/m³ and 177 microns (80 mesh), respectively [51]. Therefore, they are categorized as Group B powder following the Geldart classification of powders according to fluidization properties [52]. Its minimum bubbling velocity is approximately 1.8 m/s [53]. Therefore, applying superficial air velocity of about 4.5 m/s has already sufficient to separate glucomannan and the other smaller particles in a fluidized bed and cyclone systems. The chemical composition of extruded porang tuber flour after this pneumatic separation can be observed in Table 3.

### Table 3. The chemical composition of extruded crude porang tuber flour after wind-sifting (g/100g).

| Components   | Raw    | 80°C  | 100°C | 120°C | 140°C | 160°C |
|--------------|--------|-------|-------|-------|-------|-------|
| Glucomannan  | 50.62  | 55.99 | 57.74 | 60.89 | 65.37 | 64.38 |
| Starch       | 21.62  | 19.52 | 17.42 | 12.95 | 7.21  | 7.36  |
| Soluble sugar| 4.27   | 1.10  | 1.21  | 2.00  | 3.41  | 3.55  |
| Protein      | 6.53   | 5.06  | 4.99  | 4.91  | 4.31  | 4.45  |
| Fat          | 2.02   | 2.21  | 2.27  | 2.38  | 2.01  | 2.04  |
| Ash          | 4.84   | 4.94  | 4.89  | 4.71  | 4.76  | 4.93  |
| Fiber        | 0.44   | 0.49  | 0.50  | 0.53  | 0.57  | 0.59  |
| Moisture     | 9.67   | 10.70 | 10.97 | 11.63 | 12.36 | 12.81 |

Table 3 discloses the fact that some starch particles were separated from the extruded porang tuber flour. As expected, there were more soluble sugars separated from the flour most probably due to their low molecular weight. There dissipation of these components from porang tuber flour caused a significant increase in glucomannan and moisture content. Only a slight increase of fiber content was
observed. Adsorption of water vapor from the air by porang flour particles to achieve a specific equilibrium concentration can be the cause of moisture content increase [51].

4. Conclusion
A technologically efficient dry processing method to produce high quality porang tuber flour through hot extrusion process in a single screw extruder has been developed. With feed moisture of 20% (w/w) and screw speed of 150 rpm, changes in carbohydrates and other components at various barrel temperatures were observed. Starch and soluble sugar were the most affected components, while glucomannan was stable at low barrel temperatures and only changed its composition at 160 °C. The content of fat and ash were found to be less affected by extrusion than that of protein. The best barrel temperature was 140 °C where highest glucomannan content in the porang tuber flour of about 65.37% was obtained after wind-sifting.

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