Contents List available at VOLKSON PRESS

New Materials and Intelligent Manufacturing (ICNMIM)

Journal Homepage: https://topicsonchemeng.org.my/

ISBN: 978-1-948012-12-6

HIGH AMYLOSE PREPARATION

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ARTICLE DETAILS

Article History:

Received 26 June 2018
Accepted 2 July 2018
Available online 1 August 2018

ABSTRACT

The basic properties of corn starch and a new method of corn starch preparation are introduced. After corn is soaked, it is coarsely crushed. After separating embryos, crude starch milk is obtained. The crude starch milk is finely crushed, and proteins and fibers are separated. A starch milk is obtained, and an alkali solution is added to the starch milk to form a starch solution. The temperature of the starch solution is 40°C. to 60°C., the starch solution is filtered to obtain amylose and amylopectin, and the amylose is dissolved. In alkaline solution, while amylopectin is not easily soluble, amylose and amylopectin are separated by filtration. The amylose and amylopectin are directly separated during the process of preparing corn starch by the process of separating the amylose and amylopectin through the different solubility of amylose and amylopectin in hot water, 18.91% of the upper layer was centrifuged for the first time. The product was mainly long amylose.

KEYWORDS

Corn starch;branching enzyme;amylose;amylopectin;filtering.

1. INTRODUCTION

Corn starch has good degradability and can be directly or combined with other substances into starch base material and used as degradation material. However, ordinary corn starch contains 70-90 wt% of amylopectin. The strength of amylopectin is small, and the film-forming performance is poor, which seriously affects the performance of the degradable materials formed by normal corn starch directly or in combination with other substances. Amylose has the advantages of high strength, good mold ability, oil resistance, etc. It is necessary to separate these amyloses from raw materials for further processing.

Both amylose and amylopectin are glucose high polymers, but they have different structures and properties: Amylose is a glucose polymer with glucopyranose linked only by α-1,4-glycosidic linkages and has a molecular weight of about 5 Million, with good film forming properties, good strength and other properties, and soluble in hot water does not become a paste; amylopectin is the main chain of α-1,4-glycosidic bond, and α-1,6-glycosidic bond The glucose polymer as a branching point has a molecular weight of between 1 million and 6 million and a high content in food starch. Amylopectin is insoluble in cold water and forms a paste with hot water. Therefore, amylose and amylopectin can be separated using different properties in hot water.

First, the corn is crushed after soaking, and the starch is separated to obtain crude starch milk. It is preferable to use corn having an amylose content of 10 wt% to 30 wt% as a raw material, such corn has a high yield and a wide range of sources, and the content of amylose in the corn is more preferably 20 wt% to 30 wt%. The corn is preferably air-dried or quick-frozen corn. First, the corn is soaked to break the network structure of the proteins in the corn, increase the permeability of the corn grain epidermis, passivate the germ, so that the germ, protein, fiber, etc. Separated from starch. According to the present invention, the sulfuric acid is preferably used for soaking. The concentration of the sulfurous acid is preferably 0.1 wt% to 0.3 wt%, and more preferably 0.2 wt% to 0.25 wt%. The soaking time is preferably 30 h to 60 h, more preferably 36 h. ~ 55h; The soaking temperature is preferably 45°C to 55°C, more preferably 49°C to 53°C. During the soaking process, sulfite can destroy the reticular structure of corn granule protein and facilitate the washing and separation of starch. The soaked corn is subjected to a coarse grinding well known to those skilled in the art, and the purpose of the coarse grinding is to grind the corn and separate the embryos. The temperature of the coarse grinding is preferably 40°C to 60°C, and the particle size of the crude starch milk obtained after the rough grinding is preferably 20 mesh to 60 mesh. The coarsely milled mixture was filtered to separate the embryos.

The coarse grinding of the raw starch is continued, and the purpose of the fine grinding is to separate the proteins and fibers from the starch. The temperature of the fine grinding is preferably 40°Cto 60°C, and the particle size of the starch milk obtained after the fine grinding is preferably 150 mesh to 300 mesh. The finely milled mixture is subjected to filtration well known to those skilled in the art to separate proteins and fibers to obtain starch milk containing amylose and amylopectin.

After the starch milk is obtained, an alkali solution is added to the starch milk to obtain a starch solution, and the temperature of the starch solution is 40°C to 60°C. Amylose is dissolved in a solution at a higher temperature, while amylopectin is insoluble, so that the amylose can be separated from amylopectin by filtration. According to the present invention, the alkaline solution is preferably a sodium hydroxide solution or a potassium hydroxide solution, more preferably a potassium hydroxide solution. The amount of the alkaline solution is preferably 0.1% to 10% by weight of the starch milk, more preferably 1% to 10%, and most preferably 1% to 4%. The temperature of the starch solution is 40°C to 60°C, more preferably 45°C to 55°C, and the pH is preferably 10 to 12.

After the starch solution is obtained, the starch solution is filtered, the insoluble material obtained is pullulan, the filtrate is amylose solution, and the obtained filtrate is cooled and centrifuged to obtain amylose. In order to achieve a better separation effect, the pore size of the filter used in the filtration is preferably 0.6 μm to 1.5 μm. In order to separate high molecular weight amylose and low molecular weight amylose

2. MODEL AND MATERIAL PARAMETERS

The starch solution was filtered through a microfiltration membrane having a pore size of 0.6 μm to 1.5 μm to obtain amylose solution and amylopectin; The amylose solution was filtered through a microfiltration membrane having a pore size of less than 0.6 μm to obtain a high molecular weight amylose and a low molecular weight amylose.

In step 1, insoluble amylopectin is filtered off and the resulting filtrate is a solution of amylose. Continuing to separate the filtrate from the microfiltration membrane having a pore size of less than 0.6 μm, the high-molecular-weight amylose and the low-molecular-weight amylose can be separated. In order to increase the separation effect between the high-molecular-weight amylose and the low-molecular-weight amylose, the pH of the filtrate obtained in Step 1 is preferably adjusted to 9 to 11 before Step 2 is performed. According to the present invention, the pH of the filtrate obtained in step 1 is preferably adjusted with hydrochloric acid. After step 1 and step 2 are twice filtered, amylopectin, high molecular weight amylose and low molecular weight amylose are all separated, and the resulting starches are of higher purity. Pullulan can be applied in the food industry Chain starch can be used in both the food industry and the chemical industry. It can also be used for the production of amylose with different molecular weights depending on production needs.

In order to increase the separation efficiency of amylose and amylopectin, it is preferable to add a debranching enzyme to the starch milk before the alkali solution is added to break the amylopectin, thereby making the amylose more soluble in the alkali solution. The addition of an alkali solution to the starch milk containing the debranching enzyme will inactivate the debranching enzyme and will not affect the subsequent process. According to this experiment, the debranching enzyme is preferably a pullulanase.

Pullulanase: Novozymes novel Novozym 26062 (EC 3.2.1.41), the enzyme preparation is non-transgenic, can be eaten, suitable temperature 63 °C, holding time 30-90 minutes. The pH is 3 to 6.5, preferably 3.5 to 5.5. Enzyme activity was 400 PUN/g, a clear brown liquid (L) with a density of approximately 1.20 g/ml. Degradation dosage reference data provided 0.9-1.2 kg/ton, equivalent to 1 microliter/gram of starch.

US National Starch Co., Ltd. made high-amylose starch by genetic modification with a content of 55% to 70%. Based on researches, many starch material modification studies were based on the selection of high amylose starch [1-4]. A previous researcher studied the performance of high amylose starch films [5]. Three starches with different linear contents were used as the main raw materials to prepare starch-based biodegradable films and their performances were compared. The results showed that with amylose content. With the increase, the tensile strength and elongation at break of the film increase the water resistance, and the comprehensive performance of the high amylose-based degradable film is obviously better than that of the conventional starch-based degradable film [6-8]. Select starch: 1, corn starch Changchun Dacheng Group, water 13.8%.

Corn starch aqueous suspension, 2% concentration, kept at the gelatinization temperature or slightly higher than the gelatinization temperature, select 80 °C, during the separation to maintain the granular state, when the starch in the natural starch granules is easily soluble in hot water, at the same time. A solution with a lower viscosity was formed and the remaining amylopectin particles were removed by centrifugation, and the amylose in the supernatant was prepared by vacuum spray drying [9]. At 4000 rpm, 10 minutes centrifugalized starch emulsion, and the upper layer was stained with iodine invariant blue, which may be an energy gradient for separation, and could not counteract the hydrogen bonding forces between the chains [10-14].

Separation by sulphate method: 160°C pressurized heating, pH value controlled at 6.5~7.0, adding 13% magnesium sulfate, cooling at 90°C, spraying water to reduce the sulphate concentration to 10%, cold centrifuging at 20°C, precipitation as a straight chain Starch, magnesium sulfate was added to the mother liquor at a concentration of 13%, and amylopectin was obtained by centrifugation [15,16].

Table 1: Experimental starch product number

Sample serial number	Sample name	Amylose content	Manufacturers
1#	Waxy starch	0.7%	Changchun Dacheng Group
2#	Common starch	28%	Changchun Dacheng Group
3#	High amylose	55%	National Starch
4#	Ordinary starch centrifugal upper layer		Preparation based on 2#

Different starch hot water extracts and starches were selected and formulated into 0.4% (w/w) starch DMSO solution. The elution time (t) at different concentrations (c) and the elution time (t0) of the DMSO solvent were measured at 25 ± 0.1 °C using a 0.8 mm Ubbelohde viscometer (Shenyang Xinbaishun Petrochemical Glass Instrument Factory). The specific viscosity (ηsp) and relative viscosity (ηr) are then calculated. Use the single-point formula to find (η), c=0.0004, and substitute the obtained (η) into the empirical equation of Mark-Houwink, ie, obtain the molecular weight M. In the formula, K=1.0*10-4, and α=0.7.

Thermal analysis tests were performed on a Perkin-Elmer Diamond DSC type instrument (PerkinElmer company, US) equipped with Pyris data analysis software, and the temperature was calibrated using metal In. The sample amount of 5 ~ 10mg, before use were dried in a vacuum oven 24 small test, sealed in aluminum pan. The temperature scanning program was set to: Warm up from room temperature to 210 °C, stay at 210 °C for 5 min to eliminate thermal history, then cool down to room temperature at a rate of 10 /min °C; then increase the temperature to 210 °C at the same rate, record the temperature rise curve; isothermal crystallization process The sample was analyzed at a constant temperature of 210°C for 3 minutes, rapidly cooled to room temperature at a cooling rate of 500°C/min, and the function of the enthalpy changes with time during the isothermal crystallization process. The experiment was conducted under the protection of N_2 gas.

3. RESULTS OF COMPARATIVE ANALYSIS

Figure 1 shows the starch granules after 80°C gelatinized starch and Figure 2-2 starch pellets after spray drying in the upper layer after centrifugation. It can be seen that the original starch granules collapsed, and the granule size was not uniform after gelatinized starch spraying, and the pellets were even after the upper starch sprayed after centrifugation, an order of magnitude different from the original starch. As the size of the aggregated state changes, its material properties will also change accordingly.

Product yield: 1, the initial centrifuge of the upper layer of 8.91%, the main products of long amylose; 2, enzymatic decomposition of the lower layer of the product, centrifuged again to obtain the upper product 49.15%, the main product of short amylose; 3. Residual product 27.5%, for the part of the short straight-chain pullulan; 4, the rest is loss.18.19% of the upper layer was centrifuged for the first time, and the product was mainly composed of long amylose.

Figure 1: The spray drying of maize starch gelatinization

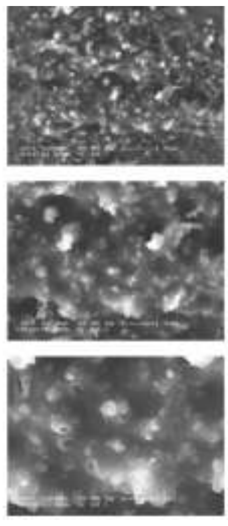

Figure 2: the spray drying centrifugal upper starch

Cite The Article: Hou Zhesheng, Kou Mengtian, Yin Jinghua (2018). High Amylose Preparation . *Topics in Chemical & Material Engineering* , 1(1) : 424-426.

Table 2: Different components of starch molecular weight

Sample serial number	Sample name	Product yield	Ubbelohde viscometer time (s)	Increased specific viscosity ηsp	Relative molecular weight
Control	Dimethyl sulfoxide		42.76		
1#	Common starch		69.24	0.63	$6.3*10^8$
4#	Ordinary starch centrifugal upper layer	8.91%	54.32	0.28	$1.98*10^8$

4. CONCLUSION

Developed a green, environmentally friendly and safe method for the preparation of amylose. The content of amylose with high content was 8.91%, high content of short amylose was 49.15%, and high content of amylopectin was 27.5%. The viscometric method was used to determine the viscosity average molecular weight of different components by more than an order of magnitude, which proved that this method is feasible. Spray drying of the separated components results in amylose granules that are one order of magnitude smaller than the original starch granules. The purity of the separation of corn starch components in this chapter needs to be improved. The separation of starch from the components can expand the application of starch in many aspects. The high-efficiency separation method needs further study.

5. ACKNOWLEDGMENTS

Fund Project：Significant Scientific and Technical Project in Jilin（20170201003GX）

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