ENRICHMENT AND RECOVERY OF RESISTANT MALTODEXTRIN FROM RICE STARCH BY ETHANOL PRECIPITATION AND ION - EXCHANGE CHROMATOGRAPHY

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ABSTRACT

Two processes are introduced as approaches for enriching and recovering resistant maltodextrin from rice starch-based pyrodextrin hydrolyzate: Ethanol precipitation and ion-exchange chromatography. For ethanol precipitation technology, the parameters include: Pyrodextrin solution concentration of 40%, rate of ethanol volume versus dry mass of pyrodextrin is 10 times (v/w), and time for separation precipitation of 5 hours. For the ion-exchange chromatography technology, it requires the strong acid cation exchange resins in Ca^{++} form with separation conditions including: Space velocity SV 0.25 - 0.50, initial pyrodextrin solution concentration of 30-40°Bx. The product recovery by ethanol precipitation provides a high separation yield, while the ion-exchange column yields less at higher purity. Both technologies can be considered applicable in practice.

Keywords: *Ethanol precipitation, ion exchange, pyrodextrin, resistant maltodextrin.*

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1. INTRODUCTION

Resistant starch as an excellent dietary fiber is an important part of the human diet and has been considered as an essential nutrient to humans, especially for obesity, diabetes, and cardiovascular diseases [1, 2]. Pyrolysis and enzymatic hydrolysis have been recognized as two principal steps in the process of producing a new type of soluble dietary fibers called resistant maltodextrins (RMD) [3, 4]. The pyrolysis is the first step and its product is pyrodextrin, which is used as the starting material for RMD production through intermediate stages of hydrolysis, purification, enrichment by separation, and drying [5, 6, 7]. The hydrolyzed product after saccharification and purification (filtration, decolorization, and demineralization) is a mixture of sugars, and oligosaccharides (dextrin, maltodextrin) with different molecular sizes or polymerization degrees (DP). According to previous research findings, the indigestible fraction (IDF) of this mixture is approximately 65-75%, the total dietary fiber content (TDF) is approximately 55 - 65%, and the DE is approximately 45 - 55% [8]. To produce a product with a higher level of IDF, TDF, as well as with a maltodextrin DE (≤ 20), it is necessary to remove sugars with polymerization degrees of DP1 and DP2, leaving only the components with DP > 3 [9, 10].

Two commonly used methods for sugar separation are ethanol precipitation and ion exchange chromatography. Precipitation is considered to be a simple unit operation. It is widely used to concentrate biomolecules, mainly proteins and sugars, in aqueous solutions [10, 11, 12]. Specifically, precipitation with ethanol has low capital costs and low operating costs. The precipitating agent can be recycled by simple distillation after the liquid and precipitated phases have separated, thereby reducing the

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environmental impact of the effluent. The ethanol fractionation method is based on the different solubility of oligosaccharides in ethanol solutions with different concentrations [13, 14, 15]. When performing chromatographic separation, the large sugar molecules will exit first, the double (DP2) and single sugars (DP1) will come out last. Therefore, for pyrodextrin solution mixtures, maltodextrin and dextrin will be recovered first, dextrose will be collected last [16 - 19].

The aim of this study was to determine the technological parameters for enriching and recovering rice starch-based resistant maltodextrin by removing low-molecular-weight sugar components in order to evaluate their industrial-scale applicability.

2. MATERIALS AND METHODS

2.1. Materials

Rice starch of the IR50404 variety was purchased from Hoa Phat Food Processing & Trading Company Limited (HCMC, Vietnam). After purification and drying at 105°C, the rice starch containing 93.9% carbohydrate, 1.03% protein, 0.22% lipid, and 5% moisture was used as a native starch sample. Acetic acid ($C_2H_4O_2$), NaOH and HCl, were purchased from Sigma-Aldrich Corp. The high purity α-amylase from *Bacillus licheniformis*, protease from *B. licheniformis*, amyloglucosidase from *Aspergillus niger*, total dietary fiber (TDF) kit K-TDFR-100A/KTDFR-200A 04/17, and K-GLUC (GOPOD Format) were purchased from Novozymes (Denmark) and used as received. Resin Lanlang® CS-1 is a strong acid cation resin in calcium form specified for glucose and fructose separation was manufactured and provided by Taiyuan Lanlang Technology Industry Corp (China RPD). Resin Purolite® PCR642K in polystyrene gel form is a strong acid cation resin in potassium form purchased by Purolite Ltd. (UK). Ambersep® 200 H also is a strong acid cation resin having sulphonic acid located in polystyrene matrix. This resin is manufactured by Rohm & Haas (USA) and provided by Megazymes Ltd. (Ireland).

2.2. Pyrodextrin preparation and enzymatic hydrolysis

The overall process of resistant pyrodextrin production was explained as follows: Six kilograms of rice starch (3 - 5% moisture) were put into a rotary mixer (Vietnam), 600 ml of 1% HCl solution was sprayed with compressed air while rotating the mixer, and after being uniformized through an overnight equilibration at room temperature, the sample was dried at 105°C in a rotary roaster (Vietnam) for 6 hours until its moisture content was 3 - 5%. The dextrinization process was then carried out in the same equipment by raising the temperature to 170°C for approximately 90 minutes. The counting of the heating time started when the sample temperature reached the target temperature, and the heating stopped when the whiteness of the starch reached 65 - 67. The pyrodextrin was hydrolyzed by two steps with two enzymes for liquefaction and saccharification. Deionized water was added to 5 kilograms of pyrodextrin, the substrate concentration was 33 - 40%, and pH 5.8 was adjusted with 20% sodium hydroxide before adding 0.4% by weight of a solution of -amylase (Termamyl SC, Novozymes A/S, Denmark, activity 2860 IU/mL) to liquefy at 95-97°C for 60 minutes.. The saccharification was performed using amyloglucosidase (AMG 300 L, Novozymes, Denmark, activity 518 U/mL) at a concentration of 0.2%, pH 4.5, and temperature of 60°C for 5 hours. Most of the solution was refined through conventional processes such as decoloring and filtration with activated charcoal and deionization with ion exchange resin. Clear liquid products after liquefaction, saccharification, and purification had an index of dextrose equivalent (DE) of 54 - 57, a soluble solid concentration (Brix degree) of 29 - 31°Bx, a content of indigestible fraction (IDF) of 64 - 67% (db), a protein content < 0.6% (db), a lipid content < 0.04% (db), and a color of 65 - 70 [8].

2.3. Ethanol precipitation of hydrolyzed pyrodextrin solution

The ethanol precipitation was done following Woo and Moon with minor modifications [10]. Take 25 mL of pyrodextrin hydrolysate and add

96% ethanol by volume fractions of 5, 7, 10, and 15 times the mass of soluble solids in the dextrin solution (12 g). Stir well and then leave the mixture at laboratory conditions for 4 hours to form a precipitate. Remove all the liquid from the top by decanting to another beaker. Use warm water at 60°C to redissolve the precipitate and then vacuum concentrate to remove excess ethanol. Adjust the volume to 25 mL exactly equal to the original volume. Measure the concentration in °Bx to calculate the recovery yield (rate of precipitated fraction) and determine the level of indigestibility. Precipitation incubations of 1, 3, 5, 10 and 15 hours were used to determine the appropriate precipitation time. To determine the appropriate hydrolysate concentration, pyrodextrin solutions with 20, 30, 40 and 50% (°Bx) were tested. The recovery of maltodextrin by ethanol precipitation at the scale of 5 l each batch was carried out in a 30 liters device with parameters derived from a small-scale test.

2.4. Ion-Exchange chromatography for separation

The column is made of SU304 stainless steel with 2 shells, 46 mm inner diameter, 1009 mm long. Hot water supply system was provided by a thermostat (JSRC-13C, JS Research, Korea) and a built-in pump in the device. Deionized water for column washing, elution was supplied with the Lead Fluid BT102S pump with rpm or mL/min indication. The system consists of 3 columns that can be operated independently or connected by valves and heat-resistant plastic pipes. The first column uses Lanlang® CS-1 resin which is a strong acid cation exchange resin in the form of Ca^{++}, the second column is filled with Purolite® PCR642K resin in the form cation K^+ and the third column uses Ambersep® 200 H resin which also is a strong acid cation exchanger in the H^+ form. The column was filled with resin to a height of about 90 cm (measured after column washing). The volume of filled resin was about 1,500 ml. A pyrodextrin solution of 40% concentration was injected into the column at a flow rate of 0.5 bed volume (BV) (equal to space velocity SV = 0.5), i.e. 0.75 l/hours or 12.5 ml/min. After finishing loading the sugar solution into the column, use deionized distilled water at 60°C to elute. The fractionation was maintained at a flow rate of 13 ml/min, maintained for 90 min. Sampling every 5 minutes, for a total of 18 times in 90 minutes. The analytical parameters include: Dextrose equivalent (DE), indigestible fraction content (IDF), concentration of dextrose sugar (DP1), maltose sugar (DP2). Expressed in terms of C_i/C_0 where i is the type of component *i* and C_0 is the initial concentration of component *i* in the sugar solution.

Determination of space velocity (SP) by filling 3 columns with the same resin which has been determined from the above-mentioned experiment. Maintain the same condition of 3 columns, only the difference in fluid flow rate is 0.25; 0.50 and 0.75 BV respectively 6.25 mL/min; 12.5 mL/min and 18.75 mL/min. Determine the column feedstock concentration with the difference in soluble solids concentrations of the solution: 30, 40 and 50°Bx. Flow rate 0.5 BV, sample collection every 5 min, and sample aliquots were stored and analyzed in the same manner as described above.

2.5. Indigestible fraction (IDF) and total dietary fiber (TDF)

The indigestible fraction (IDF) contents of the treated starch samples were determined according to the AOAC Official Method 2001.03 with a slight modification [20, 21]. This method was described in our previous study [6, 7, 8]. The TDF content of pyrodextrin or starch was determined according to the AOAC method 991.43 (AOAC, 2003) with some improvements at the HPLC sugar determination stage [22]. Accordingly, the K-MASUG kit (Novozymes, Denmark) was used to determine the total sugars DP1 and DP2 (3 types of sugars: Maltose, sucrose and glucose).

2.6. Physicochemical properties

The pH value of starch in the mixture was determined by taking 25 g of starch (or pyrodextrin) and mixing it with 50 ml of deionized water. Determination of dextrose equivalent (DE) was according to Vietnam National Standard TCVN 10376: 2014 on "Starch Hydrolysis Products - Determination of Reducing Power and Dextrose

Equivalent - Lane and Eynon Constant Title Method". Measure the whiteness of starch or pyrodextrin directly with the Kett C-300 whiteness meter (Kett Electric Laboratory) which has a range of 0 to 100 with 0 representing the darkest, 100 representing the whitest. To measure liquid color, first prepare a pyrodextrin solution with 10% dry matter, i.e., 10°Bx. Then filter through filter paper and then measure optical density at 420 nm and 720 nm using quartz cuvettes 1 cm thick. The difference between two ODs (absolute value) multiplied by 10 is considered as the color of the pyrodextrin [3, 4].

2.7. Statistical analysis

One-way analysis of variance was performed with Minitab software version 16. The least significant differences for comparison of means were computed at $p < 0.05$. All analyses were performed at least in triplicate, and the experimental results were expressed as mean ± standard deviation.

3. RESULTS AND DISCUSSION

3.1. Determination of separation parameters by ethanol precipitation

3.1.1. Ethanol concentration

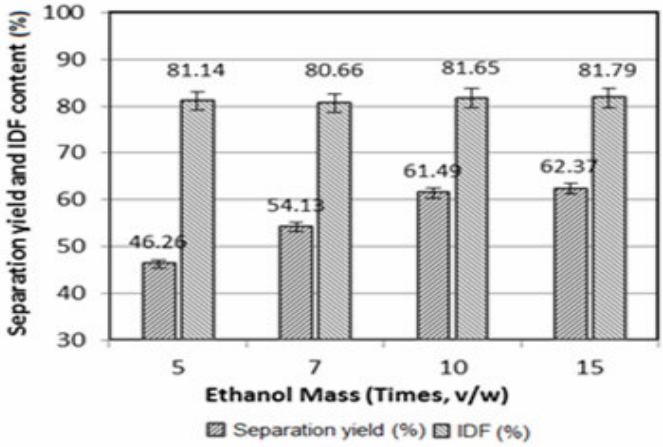

Figure 1. Change in the separation yield and indigestible fraction content (IDF) depends on ethanol concentration

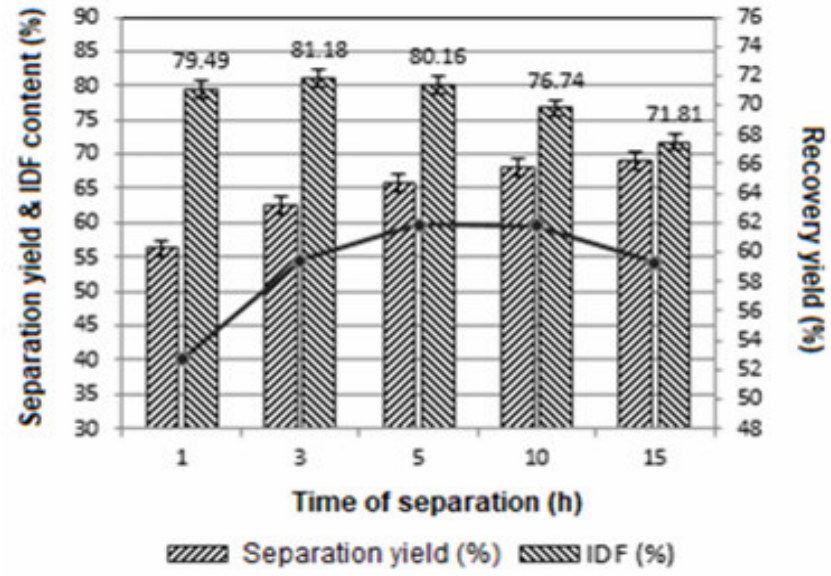

Figure 2. Change in separation yield and indigestible fraction content (IDF) depends on precipitation time

The appropriate ethanol concentration can be determined using two criteria: Separation yield and indigestible fraction (IDF) content. The separation yield was expressed as a percentage of the ratio of the soluble solid mass of the solution after precipitation to that before separation. The initial hydrolyzed pyrodextrin solution had a concentration of 30°Bx, and the ethanol concentration was 96 degrees in all experimental formulas. The results of the determination of separation yield and IDF content of resistant maltodextrin with different ethanol ratios are shown in figure 1. The data show that separation yield increases with increasing ethanol ratios. The highest separation yield was obtained at a 10 : 1 ratio and did not increase significantly at 15 : 1. The contents of IDF in the formulations were all very high and did not differ significantly. From this result, the recommended ethanol ratio is 10:1. Results of this study are completely consistent with many other published results because, as is known, ethanol precipitation is one of the most widely used methods for preparing polysaccharides and sugars, in which ethanol concentration significantly affects the precipitate yield and is usually set at high concentration of 70 - 80% [10 - 15].

3.1.2. Separation time

The separation yield of resistant maltodextrin was determined at various time intervals ranging from 1 to 15 hours. The precipitation parameters were the concentration of solute (30°Bx), ethanol (96 degrees), and the ratio 10 : 1. The results are shown in figure 2. The data shows that the separation efficiency at 1 hours of precipitation is the lowest (56.34%), increasing to 62.48% and 65.70% upon precipitation for 3 and 5 hours, respectively. When the precipitation time was increased to 10 and 15 hours, the separation yield increased significantly, gaining 68.13% and 69.11%, respectively. As a result, the separation yield increases with precipitation time and reaches a maximum after 10 hours. The difference between 10 hours and 15 hours was negligible. In contrast to the separation yield, the content of IDF decreased gradually from 3 hours to 15 hours of precipitation, which decreased markedly after 10 hours. The IDF recovery yield is the separation yield multiplied by IDF, so the value corresponding to each precipitation time is also shown in figure 2. The results show that the recovery yield peaked at 5 hours of separation. Based on these findings, the optimal time to separate the resistant maltodextrin was 5 hours. If the precipitation time is increased, although there is an increase in the separation yield, the product is not beneficial in terms of the recovery yield on the indigestible fraction.

3.1.3. Concentration of hydrolyzed pyrodextrin solution

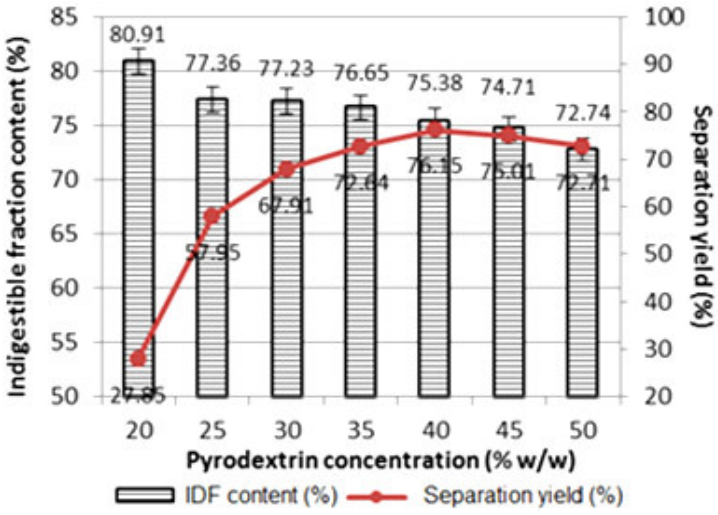

Figure 3. Change in the separation yield and indigestible fraction content depends on concentration of pyrodextrin solution

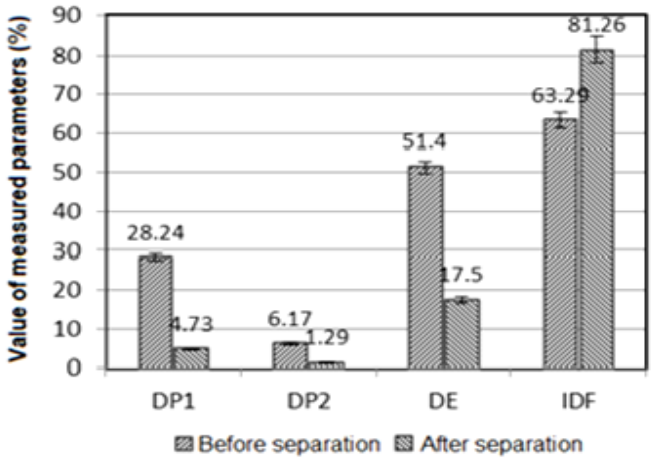

Figure 4. Content of glucose (DP1) and maltose (DP2), DE, and IDF of pyrodextrin solution before and after the ethanol separation

The results of the determination of separation yield for precipitated pyrodextrin solutions with different soluble solid concentrations are presented in figure 3. The precipitation parameters used were ethanol 96 degrees with a ratio of 10 : 1 (v/w) and a precipitation separation time of 5 hours. The data show that separation yield increases with concentration (°Bx), specifically increasing in the range of 20 - 40°Bx, reaching the maximum value at 40°Bx. However, separation yield tends to decrease gradually at 45 - 50°Bx. The IDF contents were all high and decreased gradually with the pyrodextrin concentration. The IDF recovery yield at 40 and 45°Bx was the highest. From this result, the most suitable pyrodextrin concentration for maltodextrin separation is 40 - 45% (in Brix degrees).

3.1.4. Maltodextrin separation by ethanol precipitation on semi-pilot scale

The purpose of this experiment is to verify the sugar separation parameters obtained from three small-scale laboratory experiments. The process of sugar separation was at the scale of 5000 mL of initial solution having 40°Bx, the ethanol ratio was 10 times v/w (20 liters of ethanol at 96°), and the precipitation time was 5 hours. In order to compare the important components of the solution before and after the sugar separation by precipitation method, some parameters such as glucose concentration (DP1), maltose (DP2), indigestible content (IDF) and dextrose equivalent (DE) were determined, and the results are shown in figure 4. The data show that the purified hydrolysate's DP1 (glucose) decreased from 28.24% to 4.73% and the DP2 (maltose) decreased from 6 to 17% to 1.29%. Because DP1 and DP2 were mostly removed from the precipitated solution, there were main components with a polymerization degree of DP3 (maltotetraose) or higher in the precipitate. For this result, the DE value of 51.4 was down to 17.5 after separation. The most interesting effect is the increase in IDF content, which increased from 63.29% before separation to 81.26% in the product after separation. As a result, the ethanol precipitation technique can be used to enrich resistant maltodextrin by increasing the IDF of pyrodextrin solution.

3.2. Determination of separation parameters by ion exchange chromatography

3.2.1. Selection of the ion-exchange resin

The results shown in figure 5 indicate that the strong acid cation exchange resin in the form of Ca^{++} (Lanlang CS-1) gives a lower concentration of DP1 and DP2 after separation than the other two resins, while owning a much higher concentration of high molecular weight sugars (95.36% versus 91.59% and 87.81%). The results in figure 6 also show the advantage of Lanlang CS-1 resin because this resin had the ability to obtain significantly higher content of IDF than the other two resins.

Among the 3 resins, the separation efficiency was in the following order: Lanlang CS-1 > Purolite PCR642K > Ambersep 200H. Therefore, Lanlang CS-1 resin produced and supplied by Lanlang Company was recommended to be used for further studies. However, Purolite PCR642K resin can also be used as an alternative if it has other advantages such as supply or durability.

The resin in Ca^{++} (alkaline earth metal) and K^{+} (alkaline metal) forms have also been proposed to be used for the purpose of sugar (fructose/glucose) separation in a number of foreign publications [16, 17]. However, a comparative study, especially compared with both H^{+}, has not been published at home or abroad [18, 19].

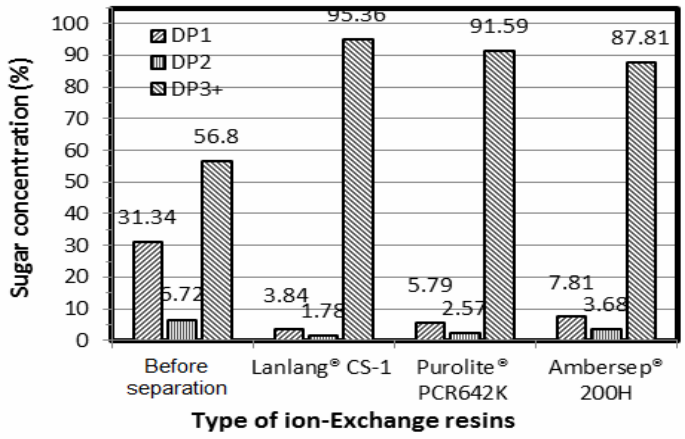

Figure 5. Comparison of concentration of sugar composition between 3 products from ion exchange chromatographic separation using 3 types of resin (40°Bx, S.V. 0,5)

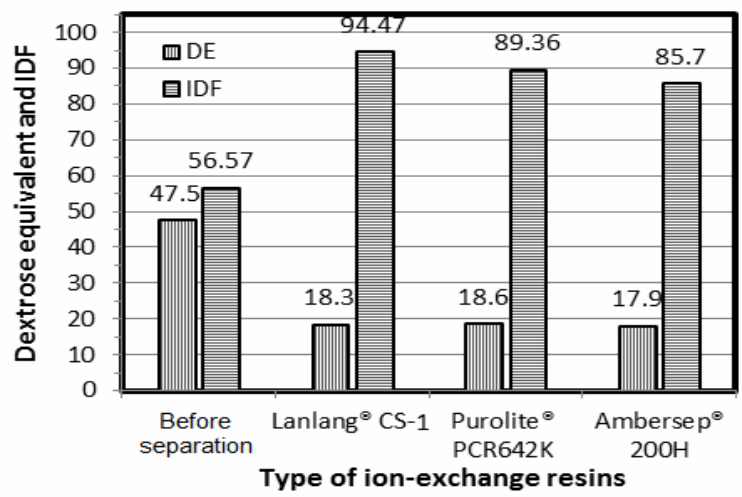

Figure 6. Comparison of indigestible content and dextrose equivalent (DE) between 3 products from ion exchange chromatographic separation using 3 types of resin (40°Bx, S.V. 0,5)

3.2.2. Determination of appropriate space velocity

In this experiment, a strong acid cation exchange resin in the form of Ca^{++} (Lanlang® CS-1) was used to separate the sugars. Separation conditions were maintained similar to the experiment mentioned in Section 3.2.1, only differing in the space velocity (SV): 0.25, 0.5, and 0.75 BV (bead volume). The analyzed parameters include glucose (DP1), maltose (DP2), and sugars with DP ≥ 3 (DP3+). Values are expressed as C_i/C_o as a percentage, i.e., the value of the C concentration at the i-th sampling time relative to the initial concentration. Only DE is expressed in

absolute value. In principle, it was only necessary to determine the DE value to calculate the fractionation stopping time to collect maltodextrin samples and thereby control the DE of the product after drying. This is based on the requirement that maltodextrin have a DE ≤ 20.

The analysis results shown in figures 7 and 8 correspond to a flow rate or space velocity of 0.25, 0.5, and 0.75 BV. The data from figure 7 show that during the first 50 min, no DP1 sugar was eluted. At 55 minutes, glucose at a low concentration (10%) was determined. Maltose appeared earlier than glucose, as early as 35 minutes, but only in relatively low concentrations because maltose itself in the hydrolyzate is much lower than glucose. The more interesting component is the concentration of sugars with DP3+, which was found to occupy a large amount during the first 50 minutes of separation. These substances make up the indigestible component and the dietary fiber. The overlapping area of the DP3+ sugars with the DP1 and DP2 sugars was smaller than the corresponding area in figure 8, indicating that a low space velocity of 0.25 BV is likely to give a higher purity of DP3+ than that of 0.75. The 0.5 BV flow rate also gives the DP3+ separation as good as 0.25 BV (data not presented).

The DE values were 19.8 after 55 min of separation at SV of 0.25 BV, and DE 18.04 and DE 18.7 after 40 and 25 min of separation at SV 0.5 and SV 0.75, respectively. Thus, a small flow rate (SV 0.25) took longer to complete separation than a high SV, although the separation efficiency or the purity of the DP3+ components was lower due to the mixing of DP1 and DP2 at a larger level. From the above results, the SV 0.5 was proposed to be chosen because of the moderate separation time (40 minutes) and the high purity level.

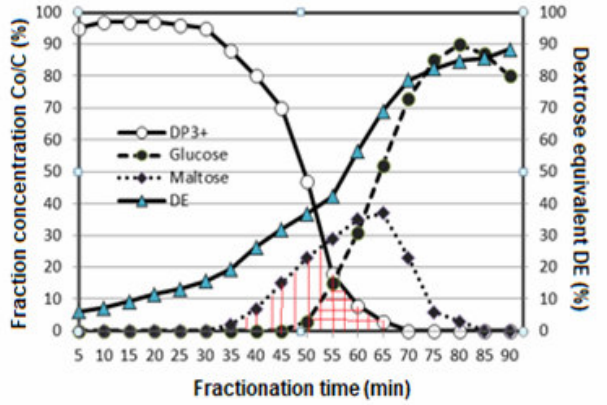

Figure 7. Separation profile of low MW fraction from pyrodextrin hydrolysate of 40°Bx, space verocity of 0.25 BV by Lanlang® CS-1 resin column

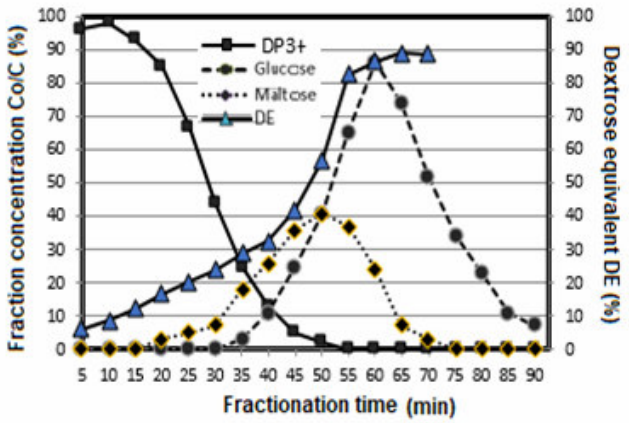

Figure 8. Separation profile of low MW fraction from pyrodextrin hydrolysate of 40°Bx, space verocity of 0.75 BV by Lanlang® CS-1 resin column

3.2.3. Determination of concentration of initial pyrodextrin solution

This experiment maintained the same separation conditions as mentioned in Section 3.2.2. The difference was only in the concentration of the hydrolysate (30, 40, and 50°Bx). The aim was to choose which concentration was suitable for chromatographic separation. In theory, a high concentration is better for separation yield than a low concentration, but there may be some influence effects at high concentrations, such as the need for high temperatures to reduce viscosity. The results of fraction analysis at concentrations of 30°Bx and 50°Bx are shown in figures 9 and 10. The data showed that at the same flow rate (SV 0.5), lower concentrations (30°Bx) gave potential for better separation compared to solutions with high concentrations (50°Bx). This is reflected in the overlap area between the DP3+ concentration curve and the DP1 or DP2 curves. Compromising the benefits of separation capacity or DP3+ enrichment with separation yield, a concentration of 40% (40°Bx) was proposed as the choice. The results of product quality after sugar separation with separation parameters: pyrodextrin hydrolyzate 50°Bx, flow rate 0.5 BV, Lanlang® CS-1 resin, showed that the product quality parameters were satisfactory, such as DE < 20, high IDF content (94.47%), and very low concentration of low molecular weight sugars (3.84% glucose, 1.78% maltose).

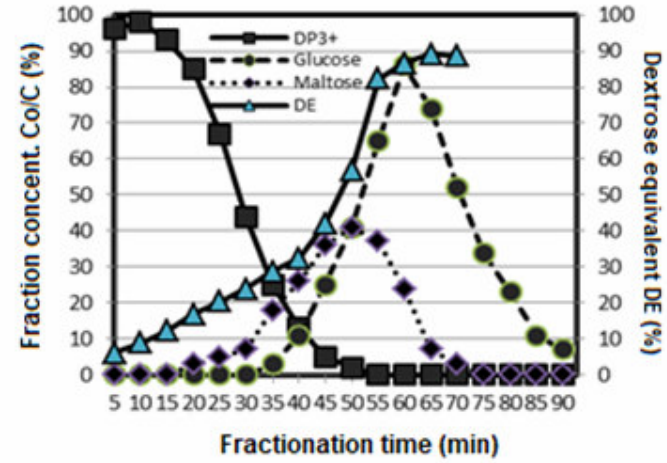

Figure 9. Separation profile of low molecular weight fraction from pyrodextrin hydrolysate of 30°Bx, space verocity of 0.5 BV by Lanlang® CS-1 resin column

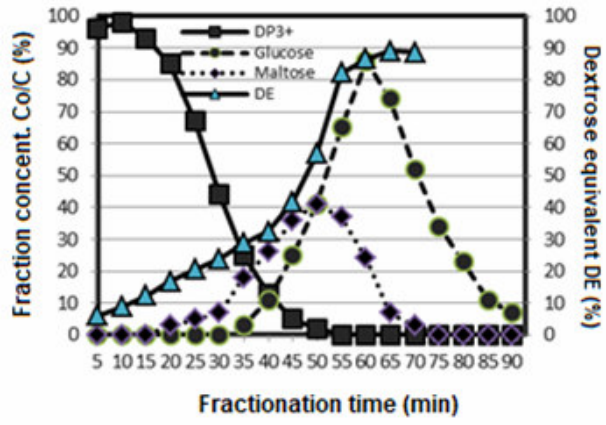

Figure 10. Separation profile of low molecular weight fraction from pyrodextrin hydrolysate of 50°Bx, space verocity of 0.5 BV by Lanlang® CS-1 resin column

4. CONCLUSION

Based on the results of this study, it can be concluded that it is possible to use ethanol precipitation and ion-exchange chomatography technology as well for enriching and recovering the indigestible fraction in pyrodextrin hydrolyzate. For the ethanol precipitation technology, the parameters include: Pyrodextrin solution concentration of 40%, rate of ethanol volume versus dry mass of pyrodextrin of 10 times (v/w), and time for separation precipitation of 5 hours. Strong acid cation exchange resins in Ca++ form are required for the ion-exchange chromatography technology, along with separation conditions such as space velocity SV 0.25 - 0.50 and an initial pyrodextrin solution concentration of 30 - 40°Bx. The product recovery by ethanol precipitation provides a high separation yield, while the ion-exchange column yields less at higher purity. It is necessary to improve and upgrade the technology and consider the econo-technical efficiencies to successfully choose the appropriate separation technology for production applications.

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