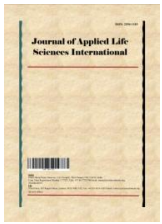

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Optimization in Preparing Sago Based Maltodextrin in a Twin Screw Extruder

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Authors' contributions

This work was carried out in collaboration among all authors. Author AA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors LR, SW and Tamrin managed the analyses of the study and the literature searches.

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ABSTRACT

Maltodextrins were prepared from sago starch in a twin screw extruder. A response surface design was used in the extrusion, which included the variations in the α-amylase (Termamyl 120L) concentrations of 12-24 g/kg dry starch, the moisture contents of 45-55%, and the screw speeds of 75-175 rpm. The product properties examined included dextrose equivalent (DE) values, and composition of malto-oligosaccharides of degree of polymerization 2 (DP2) to DP7 by using high performance liquid chromatography (HPLC). The result indicated that DE values ranged from 3.16 – 23.55 mg/g sample and the oligosaccharide component of DP5 was predominant in the samples. The possibility of using the products in food application was also discussed, including incorporating the maltodextrin in a food system as binder, bulking agent, flavor encapsulation, fat replacer, or other applications.

Keywords: Degree of polymerization; dextrose equivalent; malto-oligosaccharide; sago starch; twin screw extrusion.

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1. INTRODUCTION

Enzymatic and chemical degradation of starch result in the production of D-glucose, maltose, oligosaccharides and higher molecular weight saccharides. A lightly hydrolyzed starch is usually termed a maltodextrin and has a typical dextrose equivalent (DE) of less than 20 [1,2]. Different DE values of maltodextrin causes the differences in physicochemical and functional properties. Maltodextrin may be applied in food as binders, bulking agent, or flavor encapsulator. Fat-like mouth-feel property possessed by low DE maltodextrin is regarded unique, leading to development of many commercial fat replacer products [2]. The need to reduce fat and caloric intake in the diet has made low DE maltodextrins important food ingredients [3,4].

Maltodextrin have been developed commercially from starches of potato, corn, rice and tapioca [5]. Other reports have also been published on the preparation of maltodextrin from various origins including those from regular, waxy, high amylose corn starches and their hydroxypropyl derivatives, from oat flours and oat bran [6], and from sago starch [7,8,9]. All of those maltodextrins were produced with conventional method, such as by utilizing a jet cooker, which require a relatively long time [10,11]. However, there was apparently no yet study reported in the production of maltodextrin from sago starch using an extrusion method.

There may be advantages in using the extrusion method to produce sago maltodextrin. The advantages of extrusion are flexible and more efficient. It could produce various types of products by simply changing the raw material, extruder operating conditions and die design. It could also operate continuously, briefly at high temperatures, and a less water usage [12]. In the extrusion process, many substantial changes in the molecules, structures and physicochemical properties might have been occurred. These changes are largely dependent upon the extrusion conditions applied. Several studies have been reported on the extrusion effects on the properties and molecular changes of various starches [13,14,15].

The present study was carried out to produce maltodextrin by lightly hydrolyzing sago starch with a thermostable α–amylase in a twin screw extruder. The effects of the independent variables of α–amylase concentration, moisture content, and screw speed during liquefaction in the extruder on dextrose equivalent (DE) and malto-oligosaccharides composition were examined.

2. MATERIALS AND METHODS

2.1 Materials

The sample of sago starch (extracted from *Metroxylon sagu*) was obtained from the Village of Lamokula, Moramo, Southeast Sulawesi, Indonesia. The moisture content of the starch was 17.55% (wet basis), determined with thermo-gravimetric method. Termamyl 120L, a thermostable bacterial α–amylase from *Bacillus licheniformis*, with activity of 120 KNU/g, was used for partial hydrolysis. One KNU (Kilo Novo α–amylase Unit) was equivalent to the amount of enzyme which broke down 5.26 g starch per hour. Other reagents used in this experiment were of analytical grade unless otherwise specified.

2.2 Extrusion Trials

A modification of the method of Veen et al. [16] was used in the extrusion trial. The 40 mm APV Baker twin screw extruder, with a total barrel length of 1000 mm (25 L/D), was configured to provide two sections; Section 1 to gelatinize, and Section 2 to liquefy the starch. A circular die was used at the exit. Screw configuration is shown in Fig. 1.

To initiate gelatinization, the starch, plus an amount of water to give a moisture of 40%, were fed into section 1, which was heated to 69°C, and then 100°C (Fig. 1). The feed rate at 5 kg/h was kept constants during the extrusion run. To liquefy the gelatinized starch, the α–amylase solution was introduced in Section 2, where the temperature was kept constant at 90°C. Concentration of enzyme and total moisture content in this section were varied by dissolving the enzyme in phosphate buffer (pH 6.5). Ca^{++} was added to the enzyme solution at 150 mg/kg starch.

Concentration of α–amylase used were 12, 18, and 24 g/kg dry starch; total moisture content were 45, 50, and 55%; and screw speed of the extruder varied between 75, 125, and 175 rpm.

The extrudates were started to be collected after no visible drift in the % torque at least 5 min observed. The extruder parameters such as

melting temperature, screw speed, and %torque were automatically recorded. The liquefied extruded samples were directly put in a solid CO_2, then vacuum-dried at 70°C for 12 h, milled and sieved through an 80-mesh size screen, deposited in a closed plastic container, and stored in the freezer for further analysis.

2.3 Average Residence Time

Average residence time was determined according to the tracer technique of Komolprasert and Ofoli [17]. The values were recorded in the absence of enzyme at only 3 different conditions of moisture and screw speed (45% - 75 rpm; 50% - 125 rpm; and 55% - 175 rpm) due to time constrains. Extrudates were collected after constant torque reading and uniform feed rate. Red 40 food color was dissolved in water, and injected at the enzyme feed port in section 3. The extrudates were collected at intervals of 20s until the dye was no longer present. The extrudates were then dried in a vacuum oven at 80°C for 12 h, resulting in a moisture of less than 5%. Each extrudate was ground and sieved through an 80-mesh screen. The redness of the ground sample was measured to obtain the Hunter 'a Value' against the reference pink tile (standardized as L=75.2; a=11.3; b=8.7) using a Hunterlab colorimeter. The 'a-value' was then plotted against the elapsed time.

The approximate average residence time 't-avg' was calculated, as shown in Equation (1).

$$t-avg = \frac{\sum_{i=1}^{\infty} ti(ai-ao)\Delta ti}{\sum_{i=1}^{\infty} (ai-ao)\Delta ti} \quad (1)$$

Where *ai* was the redness value for the colored extrudate at discrete time values *ti*, and *ao* was the redness value for the base extrudate

2.4 Determination of Dextrose Equivalent

Reducing sugar content of the liquefied starch was determined with Somogyi-Nelson method. The samples (10 mg) were dissolved with 0.5 mL dimethyl sulfoxide (DMSO), boiled for 5 min, and diluted with distilled water to give a total volume of 10 mL. Then µL of the solution was added with 0.990 mL water, mixed with 1 mL Somogyi reagent, and boiled for 20 min. The mixture was cooled, mixed with 1 mL Nelson reagent, and diluted with 2 mL of deionized distilled water. The absorbance of the mixture was measured at 660 nm. A standard curve was constructed using anhydrous-glucose (Sigma Chemical Co.)

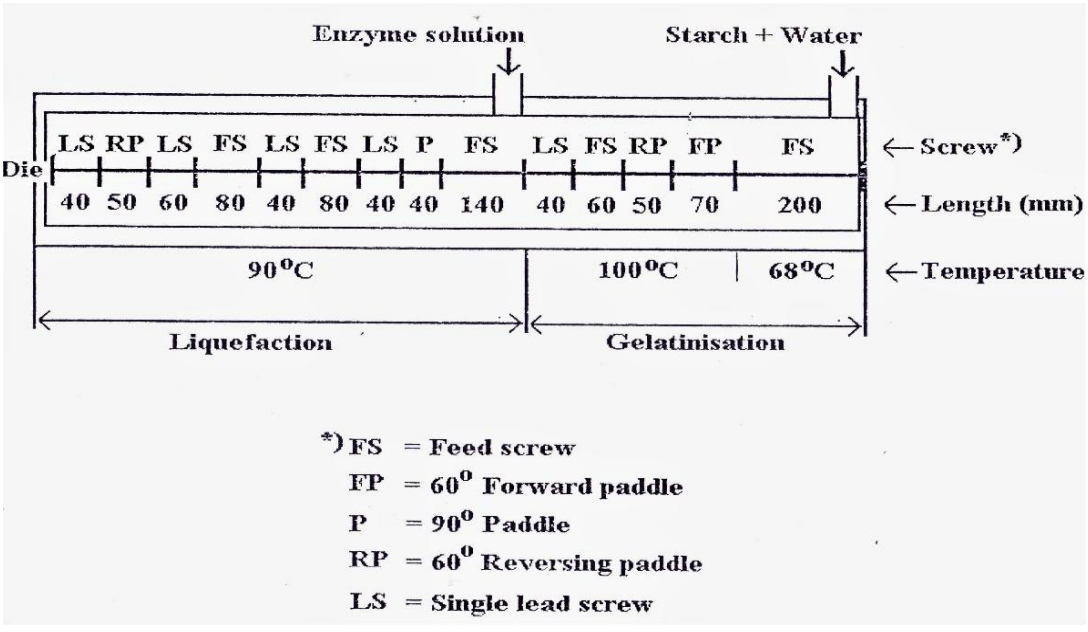

Fig. 1. Screw configuration employed in the experiment of maltodextrin prepared from sago starch in the twin screw extruder

Degree of hydrolysis was expressed as dextrose equivalent (DE), and calculated according to Hofman et al. [18], as shown in Equation (2).

$$[DE] = \frac{\%RS}{\%DS} x100 \quad (2)$$

Where RS was reducing sugar content, and DS was dry substance of the samples.

2.5 Analysis of Malto-oligosaccharides Composition with HPLC

The liquefied samples (10 mg) were solubilized with 10 mL of H_2O in a 4-mL screw-cap test tube by heating the dispersion for 5 min in a boiling water bath. An aliquot (20 µL) of the sample solution was injected into a two column HPLC system, using H_2O as the eluent at a flow rate of 0.6 mL/min. The waters liquid chromatographic system was used, including an automatic sample injection module (WISP model 712), a pump (model 510), and a differentiation refractometer (model 410). The software used for the acquisition, storage and processing of data was the Millenium 2010 Chromatoghraphy Manager. The two column, Aminex Carbohydrate HPX-42A (BioRad) with dimensions of 300 x 7.8 mm, were connected in series, and maintained at 80°C. Peak identification and concentration of each malto-oligosaccharide were determined by using individually maltose (DP2), DP3, DP4, DP5, DP6, and DP7 (from Sigma Chemical Co.) as standards.

2.6 Experimental Design and Statistical Analysis

A response surface method with the Box-Behnken design was employed in collecting the data. Fifteen conditions of experiments were carried out in a random order, including three replications at the center points.

Combination of the coded and process variables are listed in Table 1. The process variables were coded using the following formulas:

X_1 = (α-amylase – 18)/6 in g/kg dry starch.
X_2 = (Moisture – 50)/5 in %
X_3 = (Screw speed -125)/50 in rpm

A second order polynomial model, which may contain linear, interaction, and quadratic terms, was fitted; and three-dimensional surface curve was generated using the Minitab 18 statistical program.

3. RESULTS AND DISCUSSION

3.1 Average Residence Time

The time distribution as measured by the redness ('a-value') of the extrudates is shown in Fig. 2. The average residence time ('t-avg') was calculated at only 3 different conditions of moisture and screw speed (55% - 175 rpm; 50% - 125 rpm; and 45% -75 rpm), due to the time and resources constrains. The values obtained were 149, 169, and 224s, respectively. After the

Table 1. The coded and actual units of the independent variables employed in the extrusion of sago starch

Run No.	Coded			Actual Unit		
	X_1	X_2	X_3	X_1 (α-amylase) (g/kg dry starch)	X_2 (moisture) (%)	X_3 (screw speed) (rpm)
1	-1	0	-1	12	50	75
2	-1	0	1	12	50	175
3	-1	-1	0	12	45	125
4	-1	1	0	12	55	125
5	0	-1	1	18	45	175
6	0	-1	-1	18	45	75
7	0	1	-1	18	55	75
8	0	1	1	18	55	175
9	0	0	0	18	50	125
10	0	0	0	18	50	125
11	0	0	0	18	50	125
12	1	0	1	24	50	175
13	1	-1	0	24	45	125
14	1	1	0	24	55	125
15	1	0	-1	24	50	75

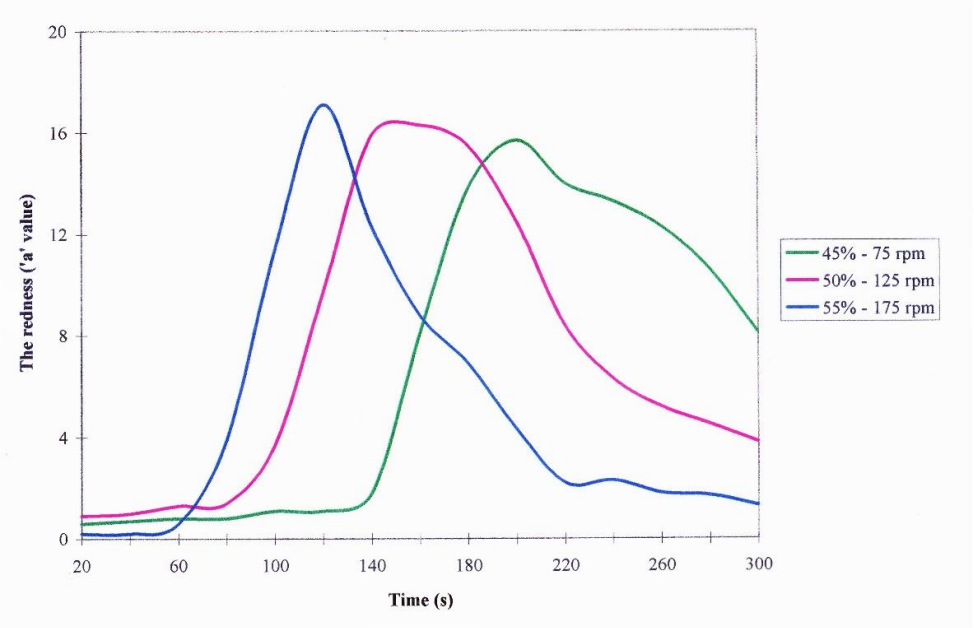

Fig. 2. Residence time distribution of the extrudates processed at various moistures and screw speeds of 55%-175 rpm, 50%-125 rpm, and 45%-75 rpm

dye was introduced into the extruder, the first visual trace of the color was observed at approximately 60, 80, and 140s, respectively and reached its peak at approximately 120, 140, and 200s, respectively. The size of the peak was broader with increasing residence time. The values obtained may give an approximate indication of the effective reaction time of the enzymatic starch hydrolysis occurring inside the extruder barrel for its following experiment in the preparation of maltodextrin.

3.2 Dextrose Equivalent (DE)

Dextrose equivalent of the liquefied sago starch samples varied widely from 3.16 to 23.55 mg/g sample (Table 2). Although they were not statistically significant, the quadratic effect of screw speed and the interaction effect of enzyme concentration and moisture content tended to influence DE value, as shown in Table 3.

The DE values of the products might be predicted by controlling the variables in the extrusion process, using the following regression model:

$$DE = -55.2456 + 2.2058X_1 + 3.5625\ X_2 - 0.2860X3 + 0.0212\ X_1^2 - 0.0403X_2^2 + 0.0009X_3^2 - 0.0533X_1X_2 + 0.0007X_1X_3 + 0.0018X_2X_3$$

Where X_1, X_2, X_3 were α–amylase concentration, moisture content and screw speed, respectively. The value of R^2 indicated that 95% of the variation in DE values was explained by the model.

Fig. 3 shows the effect of enzyme concentration and moisture content on DE values. A lower moisture content in the liquefaction process during extrusion resulted in a higher DE value, while enzyme concentration seemed to have a little influence. Perhaps, the higher water content would have reduced the total concentration of enzyme leading to an early inactivation of the enzyme in the extruder, and consequently a lower degree of hydrolysis. Veen et al. [16] also found that the reduced water content during gelatinization gave a higher DE value in the subsequent liquefaction.

DE values of extrusion hydrolysates reported in the literature varied widely from less than 1 [19] to about 36 [20]. This was largely dependent upon the extrusion conditions employed, particularly enzyme concentration and effective reaction time in the barrel [21].

DE was indeed an empirical approximate measurements of reducing sugar content, and thus an indication of starch hydrolysis [22]. Often, the starch hydrolysates consisted of

complex mixtures of molecular species ranging from glucose to long polymeric chains (linear and branched). This would lead to differences in the physical and functional properties of the products, even from those having the same DE value. Therefore, it seems that the saccharide composition of a hydrolysate, particularly the oligosaccharides, would have more importance in determining its physical and functional properties.

Table 2. Dextrose equivalent (DE) values and malto-oligosaccharide compositions of the liquefied sago starch

Run No[a].	DE[b]	Malto-oligosaccharide[b]					
		DP2	DP3	DP4	DP5	DP6	DP7
1	11.93	8.53	7.67	3.19	10.61	1.18	1.28
2	16.77	8.88	8.58	3.70	13.88	1.41	1.41
3	12.55	8.82	7.91	3.55	11.70	1.60	1.48
4	3.16	2.79	2.97	1.73	5.05	1.19	0.00
5	20.57	10.86	10.14	4.67	14.76	1.92	1.82
6	17.54	8.68	8.29	4.39	13.34	1.95	1.71
7	5.14	4.27	4.04	1.95	6.80	0.86	0.23
8	9.95	4.87	5.27	2.41	9.32	1.27	0.98
9	12.11	6.53	6.89	3.13	12.36	1.22	0.89
10	11.49	5.92	6.18	2.63	10.92	1.18	0.83
11	12.40	6.98	7.36	3.28	12.49	1.27	1.19
12	18.61	8.49	8.48	3.91	13.97	1.73	1.28
13	23.55	14.33	11.04	4.32	12.09	1.78	1.81
14	7.76	4.56	4.24	2.30	6.91	1.09	0.93
15	12.97	5.48	5.60	2.64	9.80	1.45	0.86

[a]*Samples with different Run No were prepared by using the process variables described in Table 1.*
[b]*DE is Dextrose Equivalent, DP1 to DP7 are malto-oligosaccharide concentrations in mg per g sample*

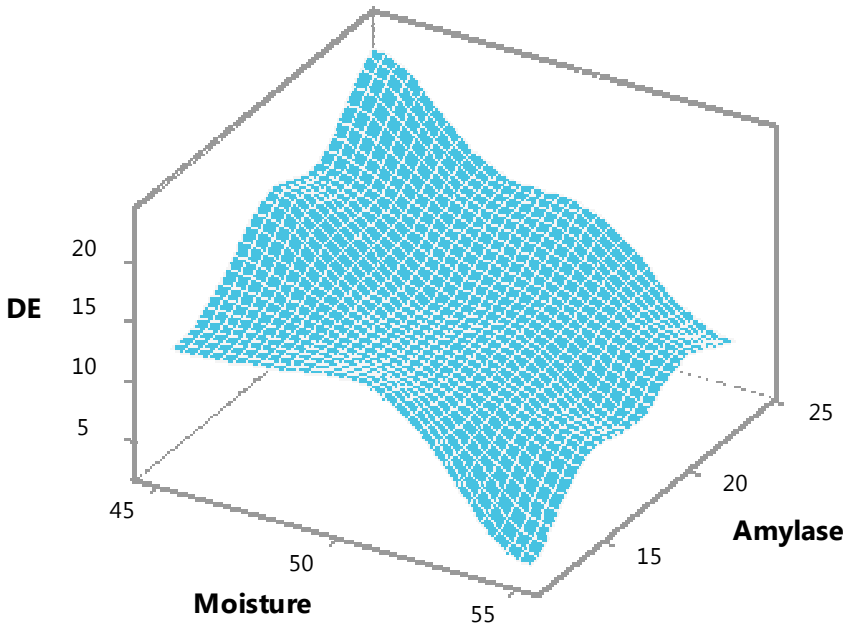

Fig. 3. The influence of enzyme concentration (X_1 in g/kg dry starch) and moisture content (X_2 in % wet basis) during extrusion on DE values

Table 3. The R^2 value and regression coefficients for dextrose equivalent (DE) and malto-oligosaccharide contents of the liquefied extruded sago starch

Dependent variables	Intercept values	R^2	Independent variables[a]	Regression coefficient	p-level	Significance[b]
DE	-55.25	0.95	X_1X_2	-0.0533	0.189	ns
			X_3^2	0.0009	0.089	ns
DP2	37.66	0.85	X_1X_2	-0.0312	0.377	ns
DP3	-24.76	0.90	X_3^2	0.0002	0.387	ns
DP4	13.53	0.93	X_3^2	0.0001	0.144	ns
DP5	-144.48	0.96	X_2	7.0380	0.015	*
			X_2^2	-0.0802	0.008	**
DP6	19.00	0.94	X_3	-0.0357	0.072	ns
DP7	19.91	0.87	X_3^2	-0.0512	0.223	ns

[a]X_1 = feed moisture (%); X_2 = melt temperature (^oC); and X_3 = Screw speed (rpm).
Only independent variables having the lowest p-level value from statistical analysis were selected for presentation in this Table.
*[b]ns = not significant; * = p < 0.05; ** = p < 0.01*

Table 4. Summary of correlation coefficients of dextrose equivalent (DE) and degree of polymerization (DP) of the liquefied sago starch

	DE	Malto-oligosaccharide					
		DP2	DP3	DP4	DP5	DP6	DP7
DE	-	0.92	0.96	0.94	0.86	0.85	0.90

3.3 Malto-oligosaccharides Profiles of the Liquefied Starch

In all runs, the components of DP1 through to DP7 were well separated. DP was degree of polymerization, with DP1, DP2, DP3, DP4, DP5, DP6, and DP7 representing respectively the component of glucose, maltose, malto-triose, malto-tetrose, malto-pentose, malto-hexose, and malto-heptose. Chromatograms of selected samples of Run No. 4 and 5, respectively having DE of 3.16 and 20.57 mg/g sample are shown in Fig. 4; and the malto-oligosaccharide composition of the liquefied samples is given in Table 2.

Statistical analysis, as presented in Table 3, indicated that the effect of extrusion conditions employed was significant on the composition of DP5 ($p<0.05$), but not significant on the composition of maltose, DP3, DP4, DP6, and DP7. The composition of malto-oligosaccharides was proportionally related to DE, which was indicated by their positive correlation (Table 4).

It appears that the average proportions of the various malto-oligosaccharides (DP2 to DP7), when calculated for the three replications in the center points, were similar to those calculated from the whole 15 runs. This suggested that different process variables used in the extrusion, which gave a wide range of DE, resulted in quite similar relative proportions of oligosaccharides.

When the average oligosaccharides composition was calculated from 15 runs, the DP5 was seemingly predominant, followed by DP3 and DP2, as shown in Fig. 4. Composition of DP7 was the lowest amongst oligosaccharides, and was absent in Run No. 4 (Table 2). The predominance of DP5 was also observed in the extrusion hydrolysates of corn starch, as reported by Roussel et al. [23]; and Vasantan et al. [20] reported predominance of DP5 and DP6 in extruded barley starch. This similar phenomenon was apparently due to the action pattern of *Bacillus licheniformis* α–amylase employed in these studies. Maldonado and Lopez [10] had indicated that this *Bacillus licheniformis* α–amylase tended to hydrolyze the substrates in an endo-specific manner, preferentially at the fifth (1,4)-glucosidic bonds from the reducing end, resulting in a higher composition of DP5 component. It has been reported that the use of α–amylase from different species of *Bacillus*, such as *B. amyloliquefaciens* produced predominantly DP3 component [24].

In food application, the saccharide composition of the maltodextrins is particularly important in determining the sensory properties of the product. It has been claimed that a low-DE

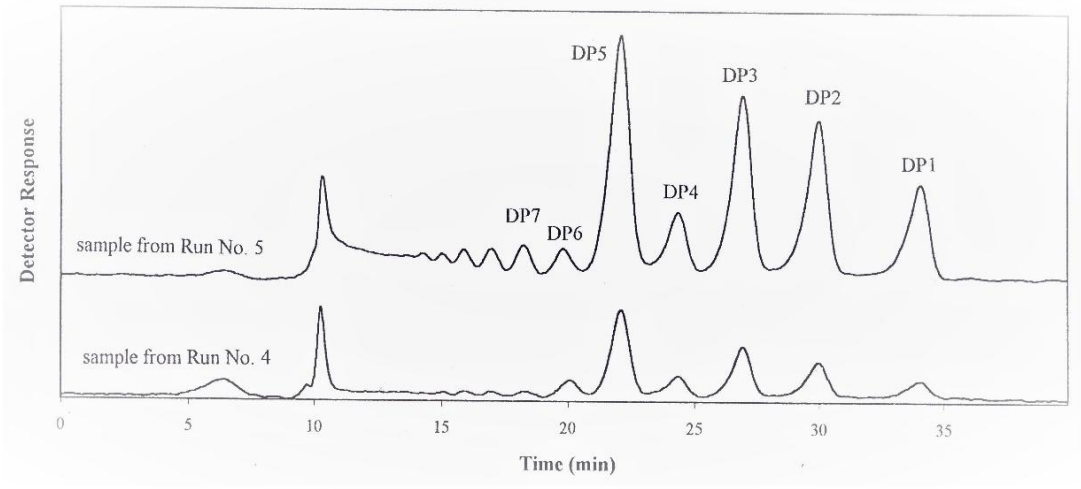

Fig. 4. Chromatogram of the samples processed with run No. 4 and 5, having a DE of 3.16 and 20.57 mg/g sample respectively

maltodextrin containing almost 98% malto-pentose and higher saccharides might provide a creamy, fat-like texture [3]. The information on the composition of saccharides is certainly useful if the maltodextrin is to be applied in a real food system, especially in fat replacement.

4. CONCLUSION

This study showed that the production of maltodextrin from sago starch by using the continuous process with a twin screw extruder is possible. The properties of the maltodextrins, particularly dextrose equivalent (DE) and malto-oligosaccharides composition varied, dependent upon the extrusion conditions applied. It was appeared that there was a possibility to produce a maltodextrin with a specific DE and malto-oligosaccharides composition by setting appropriate processing variables. Alterations in the conditions used here may be made to further improve the product properties.

This study surveyed major parameters which influenced the production of a useful maltodextrin. Optimization studies will be required to produce a sago maltodextrin which more closely resembles commercial maltodextrin in malto-oligosaccharides profile, DE, and other properties.

The maltodextrin produced may be incorporated in a food system as binder, bulking agent, flavor encapsulation, fat replacer, or other applications. However, it seems necessary to carry out some further studies, particularly in the areas of functional and sensory properties before any such applications.

With increasing demand on reduced fat foods, the development of low DE maltodextrin from sago starch as fat replacer may have a promising future. Certainly, a more thorough study is required for such a food application.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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