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DRY HYDROLYSIS OF CORN STARCH BY EXTRUSION PROCESS

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ABSTRACT

Dry hydrolysis (dextrinization) of corn starch at low moisture content by extrusion process was investigated. The effects of the extruder operating conditions (screw speed and temperature) on the dextrose equivalent (DE), mean residence time (t_m), residence time distribution (RTD), and specific mechanical energy (SME) were examined. Results showed that the extrusion process can be used to produce dextrin. The DE of dextrin was increased by increasing the screw speed and temperature. The range of t_m was 34-86 sec. The RTD was primarily affected by the screw speed and the flow pattern of the extrudates was close to plug flow. The extrusion process at low moisture content was a high energy consumption process, where the range of SME was 0.15-3.32 kW.h/kg. Dextrin with the highest DE (8.48) was obtained at 65 rpm and 135 °C where the t_m was 37 sec and the SME was 1.85 kW.h/kg.

Keywords: *dextrin, dextrinization, dextrose equivalent, dry acid hydrolysis, extrusion.*

INTRODUCTION

Dextrin is widely known as a derived product of starch and is extensively used in textile, food, brewing and pharmaceutical industries [1]. Dextrin can be obtained by different starch hydrolysis reactions such as enzymes hydrolysis, acids hydrolysis or their combination [2]. Each procedure has its own advantages and disadvantages. For example, using amylase as a hydrolytic enzyme will involve frequent handling of starch slurries; require relatively long reaction time and drying process of the output product [3].

On the other hand, using acid hydrolysis method requires less time but an excessive amount of by-products are formed. However, in an industrial scale, most of the dextrin is produced using dry acid hydrolysis method in which starch is hydrolyzed under low moisture conditions and high temperatures. This method is mainly chosen rather than the wet hydrolysis method because of its higher yield and it does not require drying process. The most common acid used for the dry hydrolysis reaction is the hydrochloric acid because of its high hydrolytic capability, low price, readily available and easy to be removed by evaporation process. Also, it has been reported that during dry hydrolysis process some amount of the hydrochloric acid is evaporated, therefore it is not necessary to neutralize the final product [4].

Dextrinator is the general name of the equipment used to carry out the hydrolysis process either using enzymes or acids. Two models are commonly available, the first is a steam or oil jacketed kettle which uses a rotating arm to agitate the starch. This dextrinator type is known as the “Hagen” cooker. The second dextrinator model is a rotary heated drum. Continuous dextrinator model is also available, where a thin layer of acidified starch is transported using a moving belt through heated ovens at a uniform speed [5]. By using conventional dextrinization process, white dextrin can be produced in the presence of acid at low temperature range (79-120 °C) in a short time (3-8 hours) and yellow dextrin can be produced at higher temperature range (150-220 °C) in a longer time (8-18 hours) [6]. The use of an extruder as a continuous reactor has been suggested as a novel approach to overcome many drawbacks of the conventional hydrolysis method [7]. The extrusion technology uses a combination of high-temperature, compression and shear stress in order to produce a product in a short-time with the advantage of high flexibility and the absence of effluents [8]. A number of researchers [7, 9, 10, 11, 12] have investigated several extrusion methods in pre-gelatinization, liquefaction and hydrolysis of starches using enzymes as hydrolytic catalyzing agents.

However, there are no reports found on the use of extrusion process to dextrinize acidified corn starch at low moisture content.The physicochemical properties of starch during extrusion have been reviewed by Lai and Kokini [13]. They reported that complete gelatinization did not occur during extrusion of starch at low moisture content. Furthermore, they also underlined that the starch granules were softer and became more mobile due to melting by extrusion at high temperature (150-170 °C). During starch extrusion process, X-ray diffraction revealed that the intensity of shear forces promotes a mechanical disruption of the molecular bonds of starch which resulted in a partial or complete destruction on the crystalline structure of starch. This was a strong indication that starch fragmentation can be achieved by an extrusion process [13]. In addition, the high specific mechanical energy (SME) can be used as an indicator of shear forces action on the disrupting molecular bonds of starch during extrusion process. Also, Klinger et al. [14] emphasized that the hydrogen bonds between neighboring starch polymers in starch structure can be broken during extrusion at high shear forces and temperatures. During the extrusion process, the hydrolysis reaction occurs as the material transported through the extruder barrel in certain time. Thus, the mean residence time is an important factor which represents the time in which the material is exposed to the heat, shear forces and chemical reactions inside the extruder barrel.

The main objective of this study was to investigate the dry acid hydroysis (dextrinization) of corn starch by extrusion process to produce dextrin. The degree of dextrinization process was quantified by the value of the dextrose equivalent. The effect of screw speed and roasting temperature on the dextrose equivalent, mean residence time, residence time distribution and specific mechanical energy were also determined during the extrusion process. Also, the relationships between the dextrose equivalent as influenced by the mean residence time and specific mechanical energy were analyzed.

MATERIALS AND METHODS

Materials

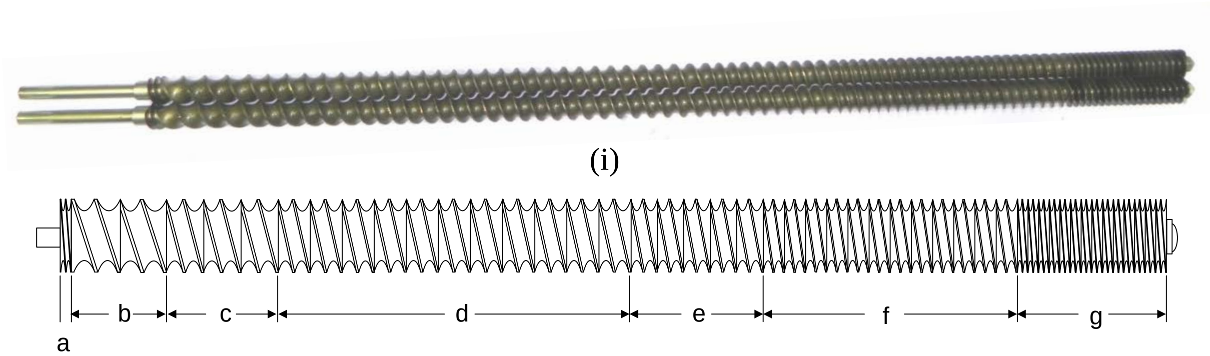
Food grade normal corn starch was purchased from ARASCO Ltd-Saudi Arabia, and used in this study. Analysis of its properties indicated that the moisture content was 13% wet basis, the pH of 10% starch suspension was 4.2, and the particle size was less than 200 microns. The dextrinization process was catalyzed by hydrochloric acid (HCl) (Panreac Quimica, Spain). All reagents used in the determination of dextrose equivalent (DE) were analytical grade. The standard for the reducing sugar (dextrose) was D (+) glucose anhydrous (Avonchem Inc. Cheshire-UK).

Feed preparation

Corn starch powder was mixed thoroughly with HCl (0.2 M) (2 starch : 1 acid) using dough mixer (Tian Shuai–Taiwan, model TS-201) for 20 min at 110 rpm. The mixture was equilibrated at room temperature for 48 hours as thin layers of 1 cm thickness on trays. The moisture content of the feed material was 11.26±0.19 %. After the equilibration process, the size of feed material was uniformed by grinding (Lab Size Disc Mill, Type DFH-48, Glen Creston, Stanmore-England) and sieving by a 200 μm pore mesh. Prior to extrusion, the feed was stored at 4 °C in sealed plastic bags.

Extrusion process

A co-rotating twin screw extruder (Model ZPT-32HT Zenix Industrial Co., Ltd.-Taiwan) was used to dextrinize the acidified corn starch. The extruder screw was 32 mm in diameter and the die plate was not mounted since the extruder was used as a continuous reactor to accomplish the roasting process. Each screw shaft was configured by the two right hand screw type elements where each screw type has a different pitch and length. The screw elements arrangement can be illustrated by Fig. 1. The barrel length was 110 cm and consisted of ten sections where each section was 11 cm long. The temperature of each section was controlled using a heating and cooling system. Heating was provided by electrical induction heater elements and steady state temperature was maintained by circulating cooling water which passes through the barrel. The temperature of each section was controlled and monitored using a digital control panel. The extruder was driven by a direct current motor (model LSK 114 VL21-Leroy Somer-French) coupled with a speed reducer (Model Zambello2-Lendinara-Italy) with a ratio of 3:1. The screw speed was controlled and the loading ampere was monitored.



a. 14/14*; 1 element
b. 50/50; 2 elements
c. 42/42; 3 elements
d. 35/35; 11 elements
e. 28/28; 5 elements
f. 21/21; 12 elements
g. 14/14; 14 elements

(ii)

Fig. 1: (i) Photo of the twin screws used in this study, (ii) illustration of the arrangement of the screw elements, not to scale, *screw element pitch (mm)/length (mm).

Preliminary experiments were conducted to identify the suitable range of the operating condition of the extruder. Based on that, the effects of three extrusion temperatures (125, 130, and 135 °C) and five different screw speeds (35, 45, 55, 65 and 70 rpm) on the dextrinization process were investigated. A steady state of extrusion temperature was achieved when the difference between the set temperature value and the real temperature of the material before the exit from the extruder was ± 1 °C over 10 minutes of operation. A constant feeding rate was attained and maintained at 32.56±0.53 gm/min using a twin screw feeder.

Dextrose equivalent (DE)

As preparation for DE analysis, three samples from the extruder were collected in plastic containers. The samples were milled by an electrical grinder (Moulinex-France) and sieved using a 200 μm powder siever. Prior to the DE analysis, the samples were kept in a tightly sealed container and stored at 4 °C. DE is defined as reducing sugars, expressed as dextrose and calculated as a percentage of the dry substance [15]. In this study, the reducing sugar content was assessed by a spectrophotometric method (modification of Nelson-Somogyi method [16]) using a spectrophotometer (UV/Vis spectrophotometer UNICO SQ-2800, Dayton, USA) at λ 529 nm. The moisture levels were confirmed for the determination of dry substance of samples by oven drying method [17]. The DE was calculated using the following equation:

$$DE = \frac{\%\ \text{reducing sugar x } 100}{\%\ \text{dry substance}} \qquad (1)$$

The DE analysis was performed in triplicate and the average and standard deviation were reported.

Mean residence time (t_m) and residence time distribution (RTD)

The mean residence time was determined using a stimulus response technique as described by Guzman-Tello and Cheftel [18] using Malachite green dye (C.I. 42000, The British Drug Houses L.td., England). The tracer (200 mg) made from dye mixed feed material was introduced into the entrance of the extruder at time zero when a steady state of desired temperature and screw speed was attained. Samples were collected every 15 seconds until no dye color was recognized visually in the output product over a period of 300 seconds. For dye extraction, one gram of sample was dissolved into 19 ml of distilled water and stirred using a vortex mixer (Velp Scientifica, Italy) at 2000 rpm. The extract was centrifuged (Heraeus Christ Gmbh centrifuge Type UJ3) at 3500 rpm for 25 minutes then the supernatant was pipetted for analysis. The concentration of dye in the supernatant was determined by absorbance at λ 529 nm using a UV/Vis spectrophotometer (UNICO SQ-2800, Dayton, USA). Triplicate experimental runs were performed and the

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average was reported. The residence time distribution of material in the extruder was evaluated using the E (t) curve which shows the variation of the tracer concentration at the exit. The area under the curve was normalized by dividing the concentration values by the total area under the curve, giving the E(t) values which is represented by the following equation [19].

$$E(t) \cong \frac{c_i}{\sum_0^\infty c_i \Delta t_i} \quad (2)$$

Where c_i is the dye concentration at time t.
The accumulated quantity of tracer at the exit was represented by the F (t) curve and given by equation 3 [19].

$$F(t) \cong \frac{\sum_{i=0}^{t} c_i \ \Delta t_i}{\sum_{i=0}^{\infty} c_i \Delta t_i} \quad (3)$$

Then the mean residence time (t_m) was calculated by the following equation [19]:

$$t_m \equiv \int_0^t t \ \mathrm{E(t)\,dt} \cong \frac{\sum_{i=0}^{\infty} t_i \, c_i \ \Delta t_i}{\sum_{i=0}^{\infty} c_i \Delta t_i} \quad (4)$$

The dimensionless parameter θ was defined for the ease of comparison of residence time distribution curves as:

$$\theta = \mathrm{t} / \mathrm{t_m} \quad (5)$$

Then, the F (t) function was normalized as follows:

$$F(t) = F\ (\theta) \quad (6)$$

Specific mechanical energy (SME)

SME is defined as the total input of mechanical energy per unit mass of the outcome product [20]. The extruder was calibrated under unloading condition to obtain the calibration factor (α). Under steady state conditions, the SME was calculated using the following equation [21]:

$$\mathrm{SME} = \frac{\alpha \ \mathrm{I \ V \ n}}{1000 \, \dot{m}_{\mathrm{out}}} \quad (7)$$

Where
SME = specific mechanical energy [kW.h/kg]
α = calibration constant [0.0049 rpm^{-1}]
I = extruder loading at a certain operating condition [ampere]
V= voltage input of extruder motor [380 Volt]
n = screw speed [rpm]
$\dot{m}_{out}$ = mean of mass flow rate [kg/h].

Experimental design and statistical analysis

Preliminary experiments were conducted to identify the suitable range of roasting temperatures and screw speeds. It was noticed by DE analysis that the starch hydrolysis was not detected at any temperatures less than 120 °C and screw speeds less than 25 rpm. On the other hand, at roasting temperatures beyond 140 °C and screw speed of 50 rpm, melting occurred and some glossy brown agglomerates were formed during the extrusion process. Investigation on the DE value of the melted material from the extrusion at a temperature of 140 °C showed the DE value was 2.91±0.38 which was lower than the DE at 135 °C. These preliminary results suggested that starch hydrolysis using acid as a hydrolytic agent and roasted by extrusion only occurred in a narrow temperature range. Thus, the primary experiment treatments included three levels of roasting temperatures (125, 130, and 135 °C) and five different screw speeds (35, 45, 55, 65 and 70 rpm). A full factorial experimental design was performed to evaluate the effect of different treatments on DE, t_m, and SME. Analysis of variance (ANOVAs) was carried out to analyze the obtained data using SAS 9.1.3 Service Pack 4 (SAS Institute Inc., Cary, USA), in order to determine the variance differences between the parameters. Tukey multiple range tests were performed for further statistical analysis to determine the significance level between the different treatments.

Non linear regression analysis (step wise method) was employed to find out the relationships between the independent variables (screw speed and roasting temperature) and the dependent variables (DE, t_m, and SME) using SPSS 11.0 (SPSS. 2001. SPSS Statistical Package for Windows Ver.11.0. Chicago: SPSS, Inc.), where the following generalized regression model used was:

$$\begin{aligned} Y = a_0 + a_1 n + a_2 T + a_3 n^2 + a_4 T^2 + a_5 n^3 + a_6 T^3 \\ + a_7 n\, T + a_8 n\, T^2 + a_9 n\, T^3 \\ + a_{10} n^2 T + a_{11} n^2 T^2 + a_{12} n^2 T^3 \\ + a_{13} n^3 T + a_{14} n^3 T^2 + a_{15} n^3 T^3 \end{aligned} \quad (8)$$

where Y is the response, n is the screw speed and T is the roasting temperature. The response surface plots were generated from the regression equations by Matlab version 7.9.0.529-R2009b (The Mathworks[TM]).

RESULTS AND DISCUSSIONS

The average and standard deviation of DE, t_m, and SME at different operating conditions are reported in Table 1. Indeed, the results revealed that the extrusion process can be used to produce dextrin with DE value up to 8.48 depending on screw speed and temperature. The Anova analysis indicated that the effect of screw speed, roasting temperature and the interaction between these two variables were statistically significant for all dependent variables ($p \leq 0.05$). Tukey multiple range tests showed the significance level between different treatments which were indicated by different superscript letter(s) for each value of the dependent variables. Parameters of the regression equations for DE, t_m and SME are shown in Table 2, where all the independent variables are significant ($p \leq 0.05$).

Table 1: The average and standard deviation of DE, t_m, SME as affected by screw speed (n) and roasting temperature (T)

Parameter	T [˚C]	n [rpm]					
		Feed	35.2±0.3	44.9±0.2	55.1±0.2	64.8±0.5	70.3±0.3
DE	-	$0.25\pm0.00^{a*}$	-	-	-	-	-
	125.5±0.8	-	0.21 ± 0.18^{a}	2.75 ± 0.26^{bc}	3.75 ± 0.58^{def}	3.88 ± 0.44^{dgh}	5.40 ± 1.33^{i}
	130.3±0.6	-	1.50 ± 0.91^{j}	2.78 ± 0.66^{bk}	3.54 ± 0.60^{ceghkl}	4.30 ± 1.07^{fl}	5.41 ± 0.98^{i}
	134.7±0.6	-	7.71 ± 1.29^{mno}	7.00 ± 0.93^{mp}	6.76 ± 0.71^{p}	8.48 ± 1.04^{nq}	8.29 ± 1.27^{oq}
t_m [sec]	125.5±0.8	-	85.2 ± 0.5^{ab}	67.3 ± 2.8^{c}	45.7 ± 0.0^{d}	34.7 ± 1.1^{ef}	34.8 ± 0.1^{eg}
	130.3±0.6	-	86.2 ± 1.0^{a}	71.1 ± 0.5^{h}	55.9 ± 1.4^{i}	34.7 ± 0.2^{fg}	36.4 ± 1.3^{j}
	134.7±0.6	-	84.5 ± 2.3^{bk}	83.9 ± 0.1^{k}	54.9 ± 0.3^{i}	37.4 ± 0.2^{j}	41.0 ± 0.6^{l}
SME [kW.h/kg]	125.5±0.8	-	0.15 ± 0.02^{ab}	0.29 ± 0.02^{acde}	0.76 ± 0.04^{f}	1.19 ± 0.08^{g}	2.13 ± 0.08^{h}
	130.3±0.6	-	0.20 ± 0.05^{bci}	0.43 ± 0.07^{dj}	0.95 ± 0.04^{k}	1.73 ± 0.15^{l}	2.30 ± 0.17^{m}
	134.7±0.6	-	0.33 ± 0.04^{eij}	0.64 ± 0.02^{f}	1.10 ± 0.08^{gk}	1.85 ± 0.09^{l}	3.32 ± 0.20^{n}

*values followed by same letter(s) are not statistically different (p>0.05) in both direction either rows or columns for each parameter

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Table 2: The regression models for DE, t_m and SME based on the independent variables: screw speed (n) and roasting temperature (T)

Dependent variable	Independent variables*	Coefficient	Sig.	R^2
DE	Intercept	307.46	0.001	0.82
	T^3	3.16×10^{-4}	0.000	
	n^3	1.46×10^{-4}	0.008	
	T^2	-0.59×10^{-1}	0.000	
	$T\ n^3$	-1.04×10^{-6}	0.013	
t_m	Intercept	173.92	0.000	0.94
[sec]	n	-2.93	0.000	
	$T^2\ n^2$	7.85×10^{-7}	0.000	
SME	Intercept	0.695	0.000	0.95
[kW.h/kg]	$T^3\ n^3$	0.90×10^{-11}	0.000	
	$T\ n^2$	-0.8×10^{-5}	0.000	

* units of n and T are rpm and °C, respectively

Dextrinization of corn starch

As a product of hydrolyzed starch, the amount of dextrin is quantified by its dextrose equivalent (DE) value, which can be used as indicator of the degree of hydrolysis. During starch hydrolysis several degraded starch products are formed such as dextrin, olygomer and glucose [22]. The DE value of pure glucose is 100 which represents a complete transformation of raw starch to dextrose (i.e. D-glucose), while the DE value of pure maltose is about 50. As emphasized by Zapsalis and Beck [15], DE can be used to quantify the amount of degraded starch which represents the amount of starch that has been cleaved by mechanical action and heat treatment.

The DE values listed in Table 1 indicate that the extrusion process was effective to hydrolyze starch. It can be observed that the DE of the feed was 0.25; meanwhile the DE range of the treated samples was 0.21-8.48. It should be noticed that the DE of the feed was not significantly different from that of the extrudates at the lowest screw speed (35 rpm) and temperature (125 °C). This was probably due to the mechanical and thermal energies (attributed to the shear forces and temperature effects) at this operating condition were not enough to break the starch granules. It was reported by Lai and Kokini [23] that during an extrusion process, gelatinization, melting and fragmentation occurred when starches were subjected to high temperatures and shear forces; thereby forming smaller starch components.

Results showed that the extrusion process can be applied to accomplish hydrolysis reaction and can be an alternative method to produce dextrin in a continuous process. Conventionally, dextrin is produced in a batch reactor where acidified starch is roasted in a heated stainless steel vessel. The vessel is equipped with an agitator and the setting temperature is in the range of 110-120 °C for 2 to 4 hours. Handoko [24] reported that dextrin with DE value of 4.44 was obtained from conventional dry hydrolysis method using feed which was made from a mix of hydrochloric acid (0.04 M) with starch at a ratio of 2:3 respectively, where the roasting temperature was 110 °C and the heating time was 50 min.

Based on the DE values, the dextrin obtained in this study was comparable to that reported by other investigators although they used a combination of extrusion and enzymes to hydrolyze starch. For example, it has been reported that the DE range was 3.5-4.5 for barley extrudates [25], 2.7-6.4 for corn-soybean extrudates [9], and 0.3-10.4 for sago starch extrudates [7]. According to Zapsalis and Beck [15] the hydrolyzed starch having DE ≥ 20 are classified as glucose syrups when post hydrolysis starch products are purified and concentrated. Since the DE values of the dextrin obtained in this study were less then 10, it can be classified as low DE dextrin products. It has been reported that low DE dextrins can be used as vehicles for carrying flavors since they have little taste while offering an increase on the cohesiveness and thickness to several flavors and other foodstuffs [15]. Goldberg and Williams [26] classified the usage of low DE dextrins as follow: dextrin with DE values ranging from 1 to 5 can be used as fillers in milk or fat based product such as yoghurt, ice cream and margarine, while dextrin with DE values in the range of 9 to 12 can be used in sugar free cheesecake filling products.

The DE of the dextrin obtained by the extrusion process was significantly affected by both temperature and screw speed (Table 1). Specifically at temperatures of 125 and 130 °C, increasing the screw speed resulted in an increase on the DE values. Increasing the screw speed represents a higher shear rate which causes more shear forces resulting in a higher degradation of the starch. Similar phenomenon was reported by Lai and Kokini [23] where they observed that increasing the screw speed increased the effect of shear forces between the sample and screw; hence long chain of starch polymer can be fragmented into shorter chain. It was noticed that at high temperature (135 °C) increasing the screw speed from 35 to 70 rpm resulted in a little increase on the DE values, however at temperatures of 125 and 130 °C the effect of screw speed was more significant. On other words, the effect of shear forces at high temperature (135 °C) was less than that at low temperatures (125 and 130 °C). The statistical analysis also indicated that increasing the temperature from 125 to 130 °C at some screw speeds treatments did not significantly affect the DE values. Generally, it can be stated that both temperatures (125 and 130 °C) gave almost similar thermal degradation effects on the starch. Increasing the temperature from 125 to 135 °C resulted in a significant increase on the DE values. This indicates that temperature of 135 °C was effective in the dextrinization of starch using the extrusion process. As previously reported that during preliminary experiments, the feed extrusion at roasting temperature of 140 °C and screw speed of 50 rpm resulted in low DE (2.91±0.38) where it was lower than the DE at temperature of 135 °C. This could be due to repolymerization process at higher temperatures (above 135 °C). It has been reported by Tomasik [27] that during dextrinization process, the amount of reducing sugar increases as the temperature increases to reach a maximum value at a certain temperature then decreases as the temperature increases further. Moreover, Evans and Wurzburg [5] reported that the reducing sugar content of the dextrin at higher temperature decreased due to a complex repolymerization reaction of low molecular weight compounds. The maximum temperature in which the highest DE dextrin can be produced depends on the method of dextrinization.

The effect of temperature and screw speed on the DE value can be predicted by the empirical relation as shown in Table 2. Fig. 2 illustrates the effect of both screw speed and temperature on the DE value where the DE increased as both the screw speed and temperature were increased. During the extrusion process, specifically at low roasting temperature; it can be seen clearly that the increase of DE was more affected by the increase of screw speed. As the temperature increased, the effect of screw speed on the DE value was proportional to the temperature. The figure suggested that higher DE can be obtained by increasing both the screw speed and temperature; however energy consumption must be taken into consideration when choosing the most suitable operating conditions for dextrin production as discussed in the section of Specific Mechanical Energy (SME).

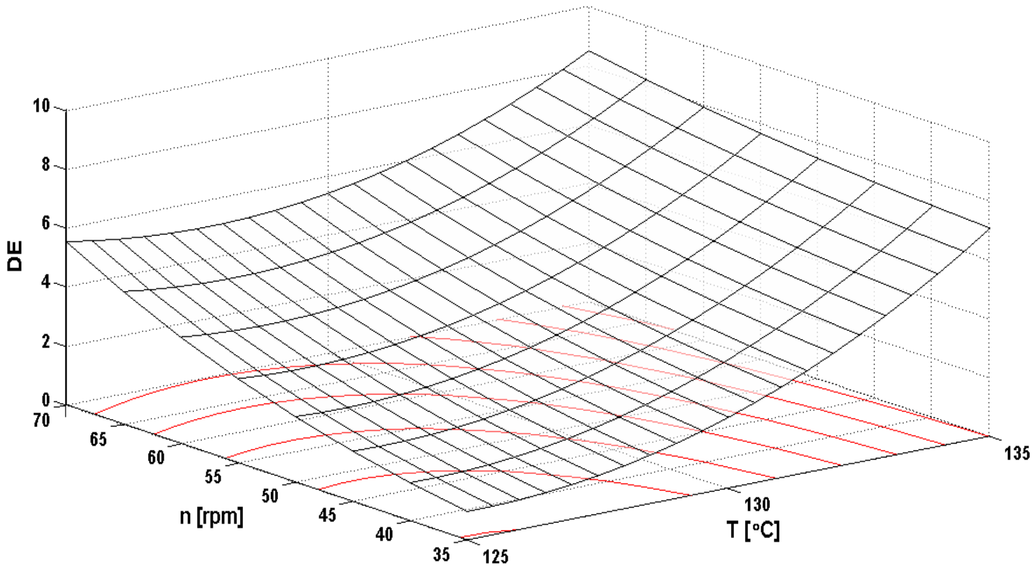

Fig. 2. Effect of screw speed and roasting temperature on the dextrose equivalence value

Mean residence time (t_m) and residence time distribution (RTD)

The t_m and RTD are important factors to be considered during an extrusion process. The t_m corresponds to the mean duration time while RTD relates to the distribution pattern of the material transiting through the extruder barrel. The t_m represents the effective reaction time where the feed material is exposed to the heat, shear forces and other chemical reactions occurring during the extrusion process. For this reason, analyzing the effect of screw speed and roasting temperature on the t_m and RTD are important, as well the relationship between DE and t_m. Table 1 indicates that the effect of screw speed on the t_m was statistically significant in most cases. The range of t_m during the extrusion process was 34.7-86.2 sec. This is a very short time if compared with the conventional method in which the dextrinization process takes about 1-4 hours [27]. In general, increasing the screw speed decreased the t_m at all processing temperatures as shown in Fig. 3. The decreased of t_m as the screw speed increased was also observed by some researchers [28, 29, 30]. Valle et al. [28] also reported that increasing screw speed at constant dry feed rate decreased the fraction filled in the screw and reduced the t_m. This was also emphasized by Unlu and Faller [30] who mentioned that the decrease of the barrel fill was the reason for the decrease in the t_m when the screw speed was increased at a constant feed rate. The effect of temperature on the t_m was significant in some cases as shown in Table 1. In some cases the t_m increased slightly by increasing temperature as shown in Fig. 3. That might be due to the softening and partial melting of some starch granules at high temperature which increased the t_m, as reported also by Lai and Kokini [13]. It can be noticed that there was a strong relationship between the t_m and the independent variables (screw speed and roasting temperature) as shown in Table 2 where the R^2 was 0.94. By using this empirical correlation, a surface response plot was generated (Fig. 4) to illustrate the effect of both screw speed and roasting temperature on the t_m where the effect of screw speed was more important than the temperature.

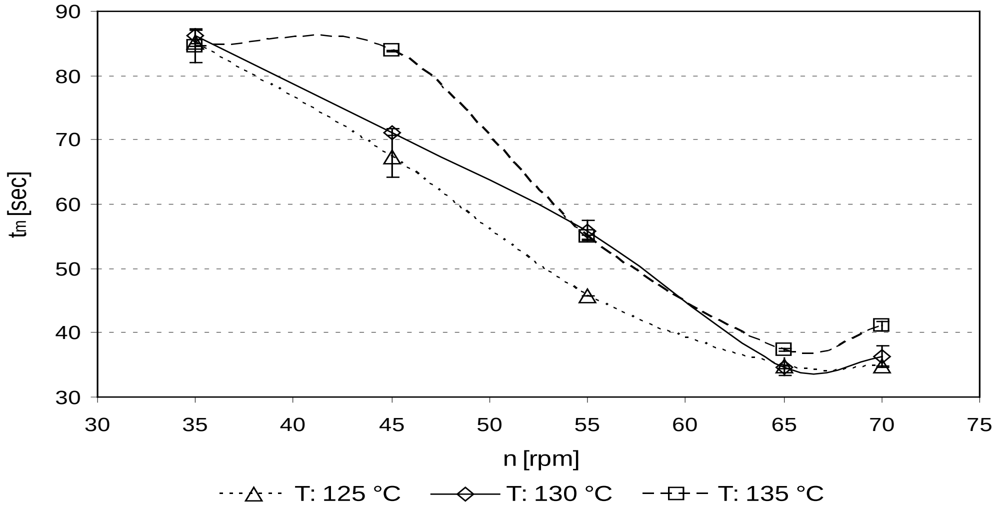

Fig. 3: Mean residence time of acidified corn starch at different screw speed and roasting temperature during the extrusion process, vertical error bars represent 95% confidence interval.

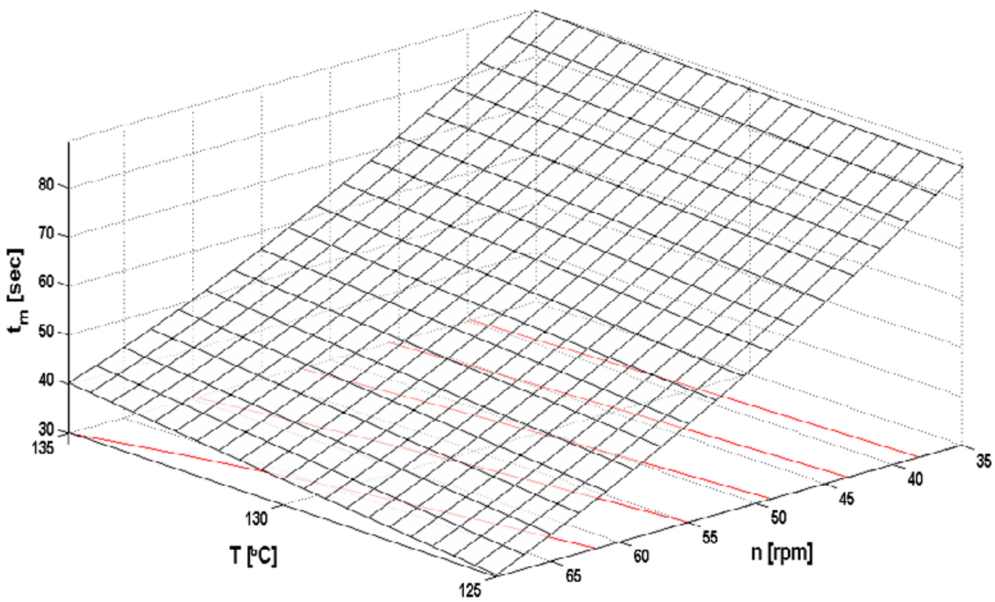

Fig. 4: Effect of screw speed and roasting temperature on the mean residence time of acidified corn starch during the extrusion process.

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The DE value was affected by the t_m and temperature and was modeled as shown in Eq. 9 where all the constants are significant.

$$DE = 445.63 + 4.03x10^{-4} T^3 - 1.03\, t_m - 7.84x10^{-2} T^2 + 7.50x10^{-3} T\, t_m ;\ R^2 = 0.92 \quad (9)$$

For more emphasis; Fig. 5 indicates that the DE decreased as the t_m increased. This was due to the more shear forces at higher screw speed where the t_m was low. Also, it can be noticed that the DE was more sensitive to the t_m at low temperature (125 °C) where the slope of the curve increased as the temperature decreased. This was an indication that the hydrolysis reaction at low temperature (125 °C) was affected primarily by the shear forces rather than that at high temperature (135 °C) where the reaction was affected by the temperature rather than the shear forces. Lai and Kokini [13] stated that generally during an extrusion process, an increase in the screw speed decreases the t_m, consequently the shear forces between the material within the extruder and the material will increase. Also it has been reported by Plunkett and Ainsworth [31] that during extrusion at low temperature and high screw speed starchy product was more abrasive, and then as the temperature increased the extruded material became more fluid and less abrasive in nature.

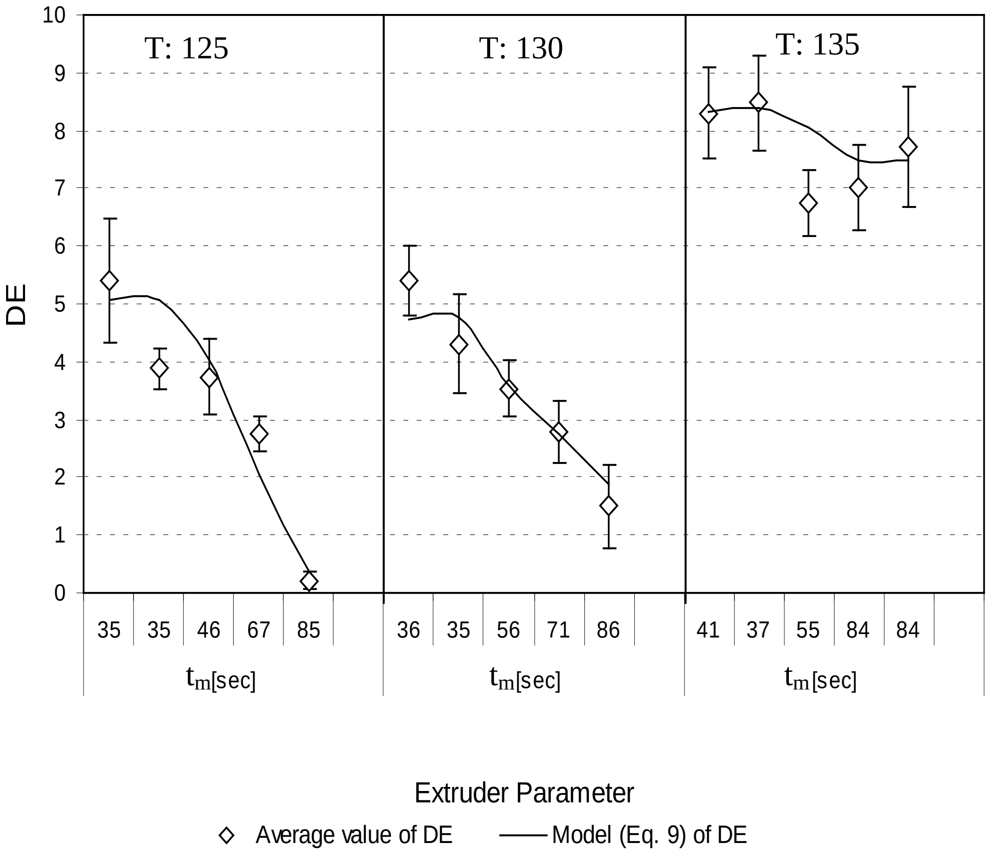

Fig. 5: Effect of mean residence time on the dextrose equivalence of dextrin during extrusion process, vertical error bars represent 95% confidence interval.

The exit age distribution (E(t)) curve of the extruded acidified starch at the extreme roasting temperature and screw speed is shown in Fig. 6. It can be seen that by decreasing the screw speed from 70 to 35 rpm the E(t) curve shifted to the right. In general, the exit age distribution curve was very similar in shape except at high temperature (135 °C) and high screw speed (70 rpm) where tailed curve was observed. This could be an indication of observed partially melted starch granules during the extrusion process which also noticed by Lai and Kokini [13].

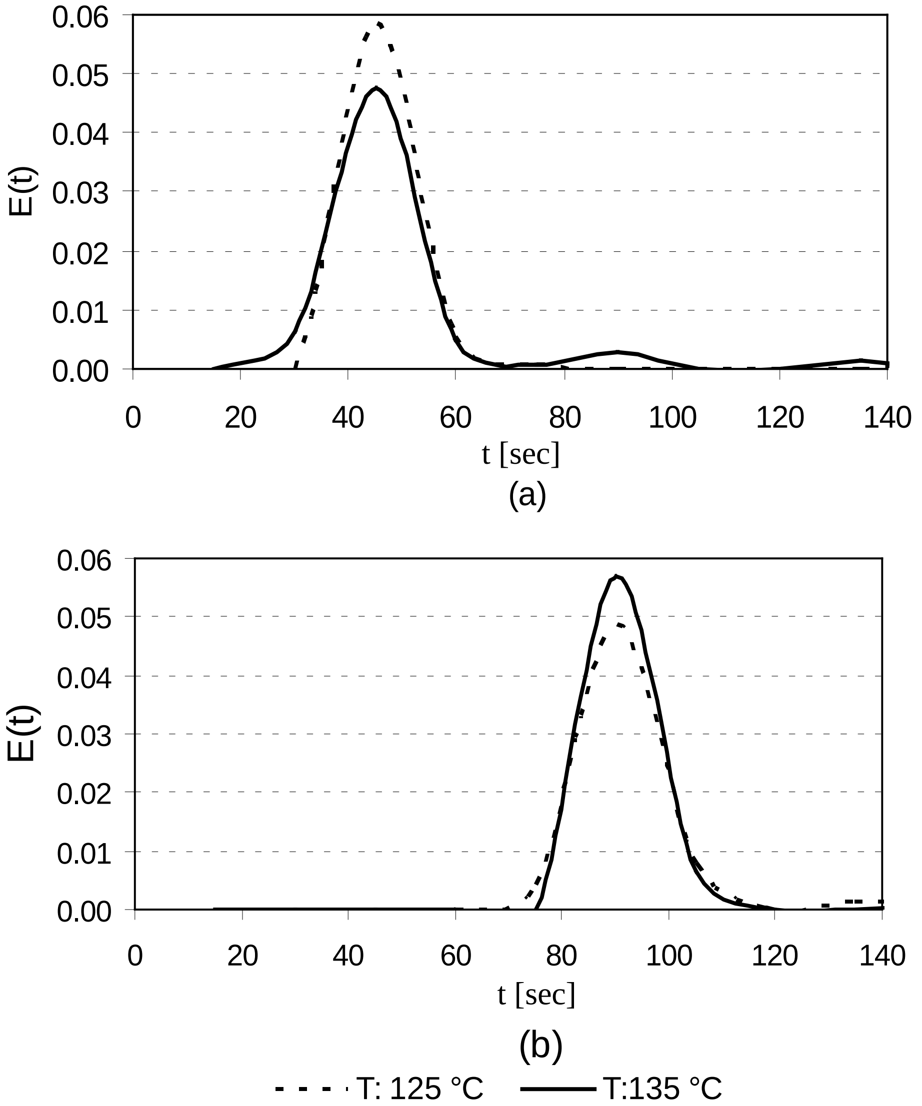

Fig. 6: Exit age distribution (E(t)) of acidified corn starch during extrusion process at the extreme operating conditions where (a) at screw speed of 70 rpm, and (b) at screw speed of 35 rpm.

The flow pattern of material through the extruder barrel can be represented by the cumulative age distribution curve as shown by F(θ) versus θ curve (Fig. 7). The cumulative age distribution curve was normal in shape indicating that the flow pattern of feed material during the extrusion process was closed to a plug flow type then deviated slightly by increasing the screw speed. According to Fogler [32] flow deviation from a plug flow type to a laminar plug flow type is indicated by decreasing slope in the cumulative age distribution curve. In addition, ideal curve of plug flow type begins from θ=1, while ideal laminar flow type begins from θ=0.5 and ideal continuous stirred tank reactor begins from θ=0 [32]. It was observed that increasing the screw speed from 35 to 70 rpm resulted in a decrease on the slope of the curve, thus the flow of the material was close to laminar flow type. Also, it can be observed in Fig. 7 at the highest screw speed (70 rpm), increasing the temperature from 125 to 135 °C shifted the curve away from θ close to 1 toward 0.5. Komolprasert and Ofoli [33] mentioned that the flow of material in a short length reactor or short residence time approaches to a plug flow type. It should be noticed that in this study the feeding rate was maintained constant. As reported by Ainsworth et al. [34] and Yeh and Hwang [35] that increasing the screw speed at feeding with a constant rate in a twin screw extrusion resulted in a decrease in the degree of fill in the extruder barrel and as a result the flow deviated from plug flow type, which in this study can be seen as tailed curve at screw speed of 70 rpm (Fig. 6).

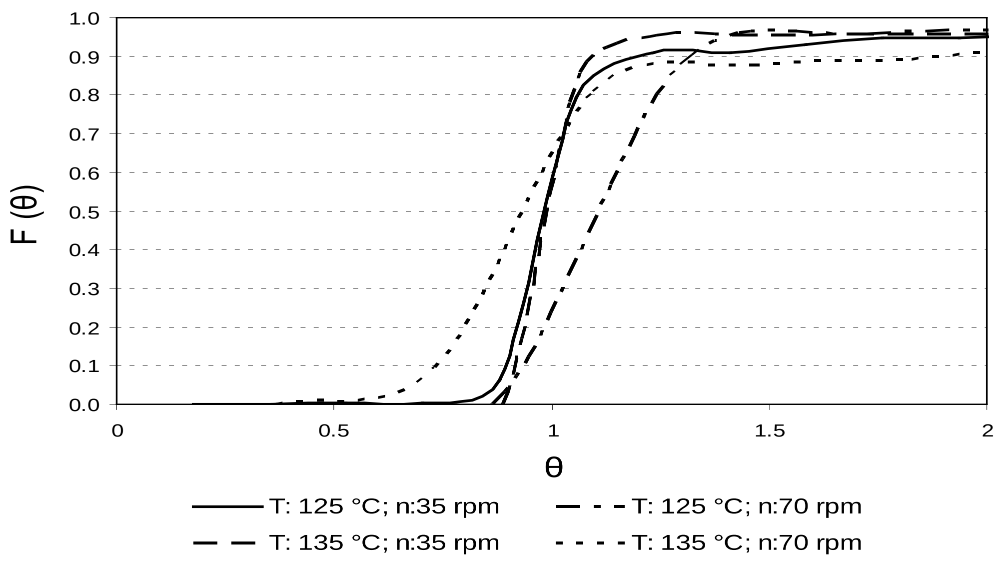

Fig. 7: Cumulative age distribution of acidified corn starch during extrusion process at the extreme operating conditions.

Specific mechanical energy (SME)

The SME is an important factor to evaluate the extrusion process in terms of the operating cost to produce a unit mass of a certain product. Both shear forces and thermal energy may break down the long chain molecules of starch to obtain new shorter chain molecules [36]. The required SME for dextrinization of starch at different operating condition is shown in Table 1. It indicates that the extrusion process was a high energy consumption process. The range of the SME was 0.15-3.32 kW.h/kg. The lowest SME occurred at the lowest screw speed and temperature. By increasing the screw speed and temperature, the SME increased drastically and reached the highest level at the highest screw speed and highest roasting temperature. Generally, extruding feed with high moisture content requires lower SME because water acts as a lubricating and plasticizing agent, therefore the viscosity and mechanical energy dissipation during extrusion is reduced [37]. For the purpose of comparison, Akdogan [38] reported that the extrusion of rice starch which had moisture content of 57, 60 and 65% w.b. at screw speed of 275 rpm and temperature of 90 °C required 0.07, 0.038, and 0.039 kW.h/kg, respectively. Seker and Hanna [39] reported an increase of moisture content from 40 to 70% reduced the SME from 0.773 to 0.347 kW.h/kg during the extrusion of blend starch, NaOH, and SMTP at screw speed of 140 rpm and temperature of 95 °C. Also, low SME range (0.0879-0.115 kW.h/kg) was reported by Meng et al. [40] who extruded chickpea flour based snack product which had moisture contents of 16-18%. Akdogan [38] emphasized that at the same temperature a decrease on the moisture content could be attributed to a higher SME. In addition to that, higher moisture content corresponds to a lower melt viscosity hence less torque is required to work the material in the screw channels. Therefore, it can be stated that dextrinization of acidified corn starch at low moisture content required higher SME if compared with other extrusion processes.

Statistical analysis indicated that the SME was significantly affected by the screw speed and temperature during the extrusion process as shown in Table 1 ($p \leq 0.05$). The SME was more noticeable at the highest screw speed. Actually increasing the screw speed was attributed to a higher SME as reported by some researchers [20, 38, 39]. Also, Table 1 shows that the SME increased as the temperature increased. During the extrusion process, it was observed that at screw speed of 70 rpm and roasting temperature of 135 °C, some glossy brown agglomerates were noticed in the extrudates. This can be an indication of a repolymerization process where the long starch polymer chain was fragmented due to high shear forces (high SME) and high temperature and then some of the material that spent more time in the extruder melted and started to re-polymerize again. In general, similar phenomenon was mentioned by Likimani et al. [9] where the SME increased with an increase on the barrel temperature when a mix of corn-soybean flour was extruded in the presence of amylase enzyme.

A high regression correlation (R^2= 0.95) was obtained for the model of SME as affected by the screw speed and roasting temperature. The surface response plot (Fig. 8) illustrates the effect of both screw speed and temperature on the SME where the effect of temperature on the SME was more pronounced at the high screw speed. The effect of SME on the degradation of starch is shown in Fig. 9. The mechanical energy applied to the starch during the extrusion process broke down the starch granules. This was indicated by increasing the DE values as the SME increased. This phenomenon was also reported by Xie et al. [41] who observed that an exponential decrease in the molecular weight of starch when the specific mechanical energy input was increased. Fig. 9. also shows that at high temperature (135 °C) the SME did not significantly affect the DE, but the DE was more affected by increasing the temperature from 130 to 135 °C.

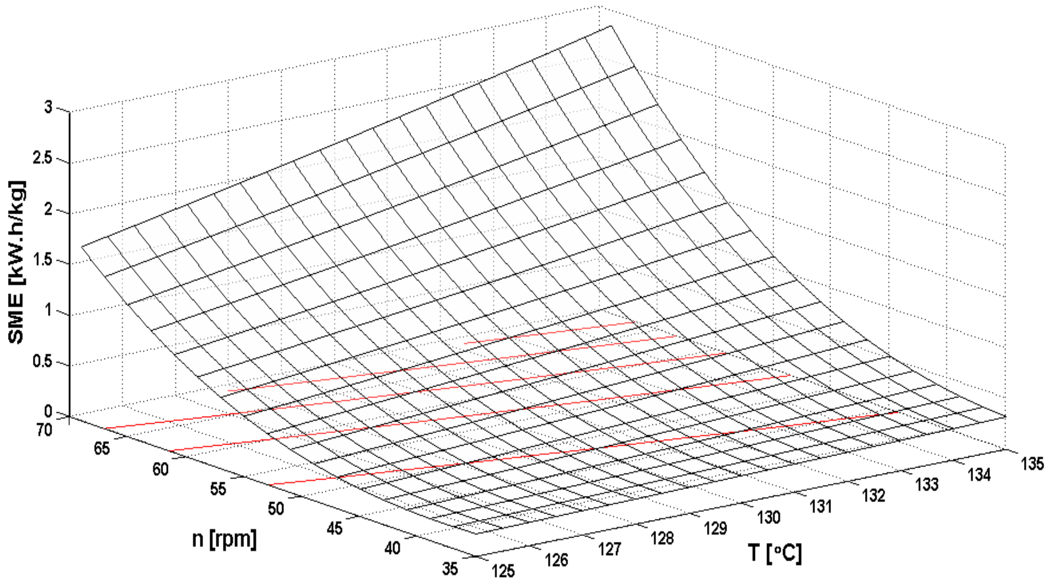

Fig. 8: Effect of screw speed and temperature on the SME requirement during extrusion process.

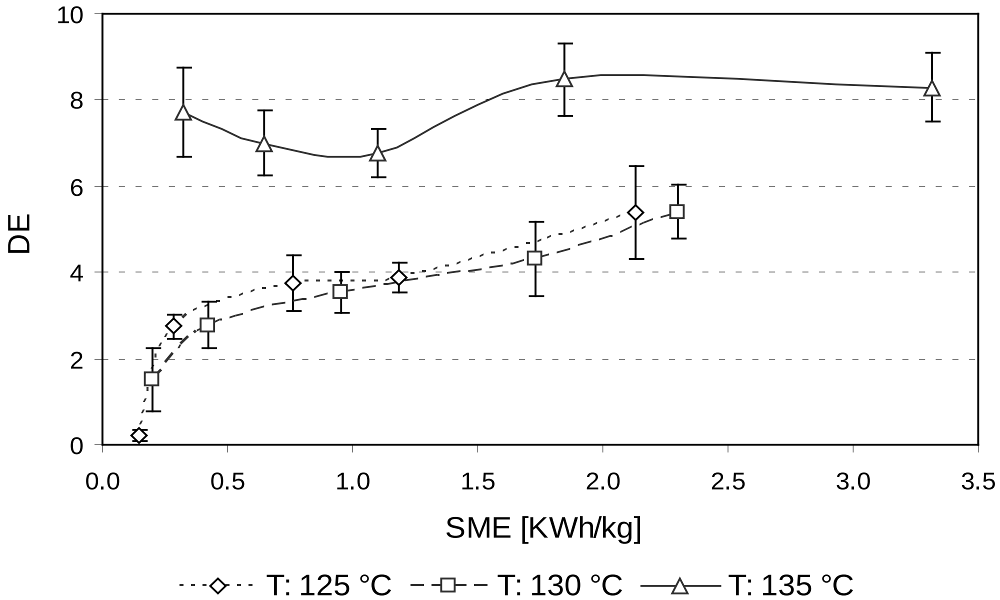

Fig. 9: Effect of specific mechanical energy on the dextrose equivalent of dextrin during extrusion process, vertical and horizontal error bars represent 95% confidence interval.

To summarize the effects of extrusion process during acid hydrolysis of corn starch in the production of dextrin, the extrusion parameters including the t_m, screw speed and SME on the dextrinization of acidified corn starch process at the best operating temperature (135 °C) are presented in Fig. 10. At this temperature the SME increased significantly however the DE was not significantly changed in most cases. Therefore, the lowest screw speed (35 rpm) and highest temperature (135 °C) can be considered as the most suitable operating condition to produce a high level DE dextrin (7.71) with low SME requirement (0.33 kW.h/kg). It should be noticed that these experiments were performed at a pilot scale; therefore for further study these results could be utilized in techno-economic feasibility study for industrial scale up and cost analysis. The characteristic and properties of dextrin produced by this process will be emphasized in another paper.

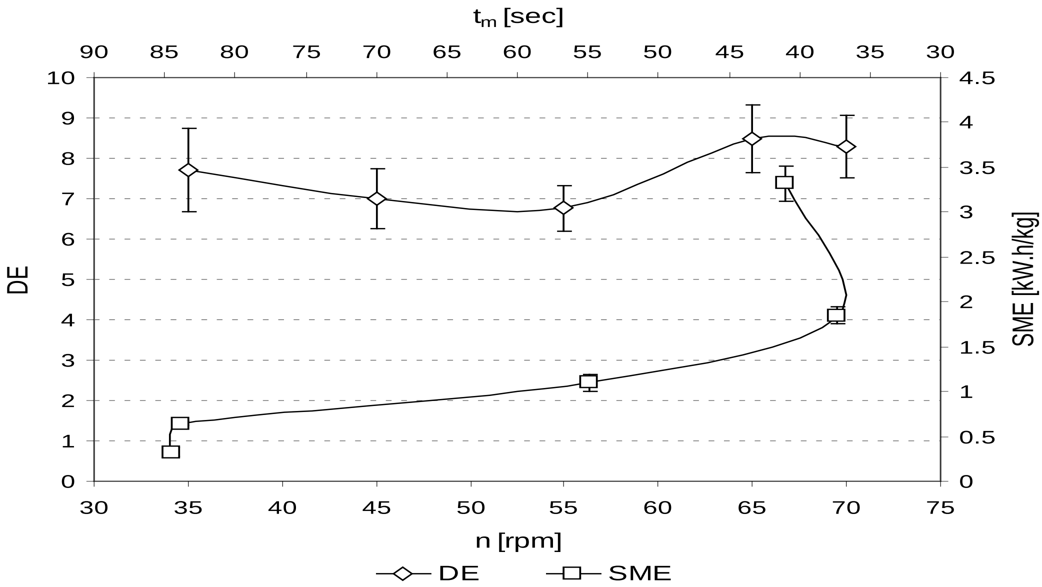

Fig. 10: Extrusion parameters at the best operating temperature (135 °C) as related to the dextrose equivalent of dextrin during extrusion process.

CONCLUSIONS

Extrusion process can be used to roast acidified corn starch to produce dextrin. The DE increased as the screw speed and temperature were increased. The t_m was significantly affected by screw speed and temperature. Increasing the screw speed and decreasing the temperature tended to decrease the t_m. The RTD curve suggested that the extrudates flow pattern was close to a plug flow. High SME requirement indicated that there were high shear forces contributed to the degradation of starch. The most suitable extrusion operating condition was obtained at a screw speed of 35 rpm and roasting temperature of 135 °C where the t_m was 84.5±2.3 sec, the SME was 0.33±0.04 kW.h/kg and the DE was 7.71±1.29. This work might be extended to obtain higher DE values using a multi step extrusion method to get a longer roasting time. Further studies are recommended regarding the high requirement of SME either by using different screw type and arrangement or using feed with high moisture content and acid concentration.

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REFERENCES

[1] Griffin, V.K., and Brooke, J.R. (1989) Production and size distribution of rice maltodextrins hydrolyzed from mille rice flour using heat-stable alpha-amylase. Journal of Food Science, 54, 19-193.

[2] Fullbrook, P.D. (1984) The enzymatic production of glucose syrups. In Dziedzic, S.Z., and Kearsley, M.W. (Eds.), Glucose Syrups: Science and Technology (Chapter 2, pp. 65-115). Elsevier Applied Science Publishers, London.

[3] Linko, P., Hakulin, S., and Linko, Y.Y. (1983) Extrusion cooking of barley starch for the production of glucose syrup and ethanol. Journal of Cereal Science, 1, 275-284.

[4] Kennedy, H.M., and Fischer, A.C. (1984) Starch and dextrin in prepared adhesives. In Whistler, R.L., Bemiller, J.N., and Paschall, E.F (Eds.), Starch: chemistry and technology. Academic Press, Orlando.

[5] Evans, R.B., and Wurzburg, O.B. (1967) Production and use of starch dextrin. In Wistler, R.L. and Paschall, E.F. (Eds.), Starch : Chemistry and technology (Chapter 11, pp. 254-276). Academic Press, London.

[6] Greenwood, C.T. (1967). The thermal degradation of starch. Advances in Carbohydrate Chemistry, 22, 483-515.
[7] Govindasamy, S., Campanella, O.H., and Oates, C.G. (1997) The single screw extruder as a bioreactor for sago starch hydrolysis. Journal of Food Chemistry, 60 (1), 1-11.
[8] Harper, J. (1992) Extrusion processing of starch. In Alexander, R.J., and Zobel, H.F. (Eds.), Developments in Carbohydrate Chemistry. A.A.C.C., USA.
[9] Likimani, T.A., Sofos, J.N., Maga, J.A., and Harper, J.M. (1991) Extrusion cooking of corn/soybean mix in presence of thermostable α-amylase. Journal of Food Science, 56(l), 99-108.
[10] Roussel, L., Vieille, A., Billet, I., and Cheftel, J.C. (1991) Sequential heat gelatinisation and enzymatic hydrolysis of corn starch in an extrusion reactor. Optimisation for a maximum dextrose. Lebensmittel-Wissenschaft und-Technologie, 24, 449-457.
[11] Grafelman, D.D., and Meagher, M.M. (1995) Liquefaction of starch by single-screw extruder and post-extrusion static-mixer reactor. Journal of Food Engineering, 24, 529-542.
[12] Baks, T., Kappen, F.H.J., Janssen, A.E.M., and Boom, R.M. (2008) Towards an optimal process for gelatinization and hydrolysis of highly concentrated starch-water mixture with alpha-amylase from B. licheniformis. Journal of Cereal Science, 47, 214-255.
[13] Lai, L.S., and Kokini, J.L. (1991) Physicochemical changes and rheological properties of starch during extrusion-a review. Biotechnology Progress, 7(3), 251-266.
[14] Klingler, R.W., Meuser, F., Niediek, E.A. (1986) Effect on the form of energy transfer on the structural and functional characteristics of starch. Starch/Starke, 38 (2), 40-44.
[15] Zapsalis, C., and Beck, R.A. (1986) Food chemistry and nutritional biochemistry (Chapter 6, pp. 351-355). John Wiley and Sons, Canada.
[16] Apriyantono, A., Fardiaz, D., Puspitasari, N.L., Yasni, S., and Budiyanto, S. (1989) Practical guidelines for food analysis. Center of Collaboration Research for Food and Nutrients, Bogor Agricultural University, Indonesia.
[17] AOAC. (1990) Official Methods of Analysis, 15th Edn. Association of Official Analytical Chemists, Washington D. C.
[18] Guzman-Tello, R., and Cheftel, J.C. (1987) Thiamine destruction during extrusion cooking as an indicator of the intensity of thermal processing. International Journal of Food Science and Technology, 22, 549-562.
[19] Levenspiel, O. (1972) Nonideal flow. In Levenspiel, O. (Ed.), Chemical reaction engineering (2nd ed., pp. 252–325). John Wiley and Sons, New York.
[20] Guan, J., and Hanna, M.A. (2006) Selected morphological and functional properties of extruded acetylated starch-cellulose foams. Bioresource Technology, 97, 1716-1726.
[21] Su, C.W., and Kong, M.S. (2007) Effects of soybean oil, cellulose, and SiO_2 addition on the lubrication and product properties of rice extrusion. Journal of Food Engineering, 78, 723-729.
[22] Garard, I.D. (1976) The introductory of food chemistry. Wesport: The AVI Publishing Company Inc.
[23] Lai, L.S., and Kokini, J.L. (1990) The effects of extrusion operating conditions on the online apparent viscosity of 98% amylopectin (Amioca) and 70% amylose (Hylon 7) corn starches during extrusion. Journal of Rheology, 34 (8), 1245-1266.
[24] Handoko, D.D. (2004) Study of water sorption isothermis of dextrin produced from *Maranta arundinaceae* at different hydrolysis level. Graduate student thesis (unpublished). Bogor Agricultural University, Bogor, Indonesia.
[25] Linko, P. (1992) Twin screw extrusion cooker as a bioreactor for starch processing. In Kokini, J.L., Chi-Tang Ho, and Karwe, M.V. (Eds.), Food Extrusion Science and Technology (pp. 33-344). Marcel Dekker, Inc., New York, USA.
[26] Goldberg, I., and Williams, R. (1991) Biotechnology and Food Ingredients (pp. 336-346). Van Nostrand Reinhold, New York, USA.
[27] Tomasik, P. (1989) The thermal decomposition of carbohydrates. Advance of Carbohydrate Chemistry and Biochemistry, 47, 279-343.
[28] Valle, G.D., Barres, C., Plewa, J., Tayeb, J., and Vergnes, B. (1993). Computer simulation of starchy products transformation by twin screw extrusion. Journal of Food Engineering, 19, 1-31.
[29] Chaudhary, A.L., Miller, M., Torley, P.J., Sopade, P.A., and Halley, P.J. (2008) Amylose content and chemical modification effects on the extrusion of thermoplastic starch maize. Carbohydrate Polymers, 74, 907-913.
[30] Unlu, E., and Faller, J.F. (2002) RTD in twin-screw food extrusion. Journal of Food Engineering, 53, 115-131.
[31] Plunkett, A., and Ainsworth, P. (2007) The influence of barrel temperature and screw speed on the retention of L-ascorbic acid in an extruded rice based snack. Journal of Food Engineering, 78, 1127-1133.

[32] Fogler, H.S. (2008) Distributions of residence times for chemical reactors. In Fogler, H.S. (Ed.), Elements of chemical reaction engineering (Chapter 13, pp. 867-944). Prentice-Hall, Englewood Cliffs, New Jersey.

[33] Komolprasert, V., and Ofoli, R.Y. (1991) A dispersion model for predicting the extent of starch liquefaction by Bacillus licheniformis α-amylase during reactive extrusion. Biotechnology and Bioengineering, 37, 681-690.

[34] Ainsworth, P., Ibanoglu, S., and Hayes, G.D. (1997) Influence of process variables on mean residence time distribution and flow patterns of tarhana in a twin-screw extruder. Journal of Food Engineering, 32, 101-108.

[35] Yeh, A.I., and Hwang, S.J. (1992) Effect of screw profile on extrusion cooking of wheat flour by a twin screw extruder. International Journal of Food Science and Technology, 27, 557-563.

[36] Miladinov, V.D., and Hanna, M.A. (1999) Physical and molecular properties of starch acetates extruded with water and ethanol. Industrial Engineering Chemical Research, 38 (10), 3892–3897.

[37] Ilo, S., Tomschik, U., Berghofer, E., and Mundigler, N. (1996) The effect of extrusion operating conditions on the apparent viscosity and the properties of extrudates in twin-screw extrusion cooking of maize grits. Lebensmittel-Wissenschaft und-Technologie, 29, 593-598.

[38] Akdogan, H. (1996) Pressure, torque, and energy responses of twin screw extruder at high moisture content. Food Research International, 29 (5-6), 423-429.

[39] Seker, M., and Hanna, M.A. (2005) Cross-linking starch at various moisture contents by phosphate substitution in an extruder. Carbohydrate Polymers, 59, 541-544.

[40] Meng, X., Threinen, D., Hansen, M., and Driedger., D. (2010) Effects of extrusion conditions on system parameters and physical properties of a chickpea flour-based snack. Food Research International, 43, 650-658.

[41] Xie, F., Yu, L., Liu, H., and Chen, L. (2006) Starch modification using reactive extrusion. Starch/Stärke, 58 (3-4), 131-139.