

# Effects of the extrusion and enzymatic extrusion treatment on the apparent viscosity of degerminated maize grits

Mingming Qi<sup>1</sup>, Dongliang Zhang<sup>1,2</sup>, Chengqian Jin<sup>1,2</sup>, Shanfeng Chen<sup>1,2</sup>,  
Hongjun Li<sup>1,2</sup>, Chengye Ma<sup>1,2\*</sup>

(1. School of Agricultural Engineering and Food Science, Shandong University of Technology, Zibo 255000, Shandong, China;

2. Key Laboratory of Shandong Provincial Universities for Technologies in Functional Agricultural Products, Zibo 255000, Shandong, China)

**Abstract:** Apparent viscosity is an important parameter for glucose syrup production. which is greatly affected by particle size and concentration of samples. In order to analyze the factors influencing the apparent viscosity, the particle size distribution, steady shear flow behavior, temperature, and time sweep test of native degerminated maize grits (NDMG), extruded degerminated maize grits (EDMG), and enzymatically extruded degerminated maize grits (EEDMG) with different particle sizes (passed through 20, 40, 60, 80, and 100 mesh sieves) and concentrations (water-sample ratios: 2:1, 3:1, 5:1, 10:1, and 20:1) were investigated. All samples passed through different mesh sieves showed a gradient and a relatively concentrated distribution, and slurries had typical shear-thinning properties. Apparent viscosity increased with increasing particle size and concentration. The lowest apparent viscosity was attained from the samples obtained at 100+ mesh and 20:1 water-sample ratio. Moreover, the sample with a 20:1 water-sample ratio showed the most stable apparent viscosity in the temperature sweep test. In the time sweep test, the power law equation with high determination coefficients ( $R^2=0.9446, 0.9382$ ) and low root mean square error (RMSE=0.0002) had the best fit to the experimental data of the EDMG and EEDMG samples passed through 100+ mesh. Overall, the lower apparent viscosities of the EDMG and EEDMG samples obtained at 100+ mesh and 20:1 water-sample ratio can improve the activity and accessibility of enzymes for glucose syrup production. This study provides critical insight into decreased apparent viscosity and expands the uses of EDMG and EEDMG in the glucose syrup sector.

**Keywords:** degerminated maize grits, enzymatic extrusion, apparent viscosity, particle size

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## 1 Introduction

Maize is one of the world's most important crops and is a major source of food, feed, renewable energy, and industrial materials<sup>[1]</sup>. Glucose syrup is a valuable product from the deep processing of maize that is used in food and beverage industries owing to its physicochemical properties<sup>[2,3]</sup>. In industrial applications, starch is hydrolyzed by acid to produce glucose syrup<sup>[4]</sup>. However, this method is energy-intensive, difficult to monitor, and requires the use of non-corrosive materials as reaction vessels<sup>[5]</sup>. Compared with acid hydrolysis, enzymatic hydrolysis has many advantages, such as easy control and mild reaction conditions<sup>[6]</sup>. Commercially, glucose syrup is obtained from maize starch through enzymatic hydrolysis

in four steps, namely, gelatinization, liquefaction, saccharification, and refinement<sup>[7,8]</sup>.

Since the 1940s, high-temperature and short-time extrusion cooking treatments have been applied in the food industry<sup>[9–11]</sup>. The properties of extrudates depend on variables in the extrusion process, including temperature, moisture, screw speed, and feed rate<sup>[12]</sup>. The addition of enzymes during the extrusion process could be a prospective new pretreatment for starch degradation<sup>[12]</sup>. Compared with the usage of enzymatic hydrolysis to prepare glucose syrup, enzymatic extrusion pretreatment eliminates the traditional high-temperature gelatinization and liquefaction and has a simple process, short treatment time (from 36–40 h to 12 h), and low energy input<sup>[13]</sup>. Furthermore, the enzymatic extrusion of maize increases the efficiency of the saccharification process and reduces the tendency of starch recrystallization<sup>[14]</sup>. Syrup production processes, such as mixing, conveying, stirring, heating, and cooling, are all strongly influenced by rheological properties (e.g., apparent viscosity)<sup>[15,16]</sup>. Previous studies have shown that the continuous production of glucose syrup is difficult to achieve owing to the high water absorption and poor flowability of degerminated maize grit (DMG) starch, which tends to sink to the bottom of the tank after settling. Measuring the rheological properties of starch paste or slurry helps select the appropriate parameters for the saccharification process in glucose syrup production.

To reduce the deficiencies in this field and consider the impact of apparent viscosity on syrup production, DMGs were extruded with and without thermostable  $\alpha$ -amylase. Particle size and apparent

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**Biographies:** **Mingming Qi**, PhD candidate, research interest: storage and processing of agricultural products, Email: [2663281307@qq.com](mailto:2663281307@qq.com); **Dongliang Zhang**, PhD, Assistant Professor, research interest: storage and processing of agricultural products, Email: [zytazhang@hotmail.com](mailto:zytazhang@hotmail.com); **Chengqian Jin**, PhD, Professor, research interest: field crop planting and harvesting mechanization and intelligent technology research and product development, Email: [412114402@qq.com](mailto:412114402@qq.com); **Shanfeng Chen**, PhD, Professor, research interest: storage and processing of agricultural products, Email: [csf72@126.com](mailto:csf72@126.com); **Hongjun Li**, PhD, Professor, research interest: storage and processing of agricultural products, Email: [Hongjunli1351@hotmail.com](mailto:Hongjunli1351@hotmail.com).

**\*Corresponding author:** **Chengye Ma**, PhD, Professor, research interest: storage and processing of agricultural products. School of Agricultural Engineering and Food Science, Shandong University of Technology, Zibo 255000, Shandong, China. Tel: +86-533-2786382-88. Email: [mcycn2002@163.com](mailto:mcycn2002@163.com).

viscosity were detected, and the influences of extrusion and enzymatic extrusion on the parameters of the rheological model were investigated. Ultimately, these results provide optimal particle size and concentration of samples in order to improve the glucose syrup production efficiency.

## 2 Materials and methods

### 2.1 Materials

DMGs (12.46% moisture content, 67% starch content) were purchased from Tianjin Baodi Maize Factory (Tianjin, China). Thermostable  $\alpha$ -amylase (purity is larger than 99%, from *Bacillus licheniformis*, 20 000 U/mL) was supplied by Boli Bioproducts Co., Ltd. (Jiangsu, China). All other chemicals and solvents used in this study were analytical grade.

### 2.2 Methods

#### 2.2.1 Material preparation

A single screw extruder (designed by the Key Laboratory of Shandong Provincial Universities for Technologies in Functional Agricultural Products) with a length-to-diameter ratio of 17.4 was used to fulfill the extrusion test. The main parameters of the extrusion process were set as follows: the speed of screw rotation was 110 r/min, the temperature of the mixing zone was 40°C, the compression zone was kept at 50°C, the die zone was kept at 60°C, the moisture content was adjusted to 30%, and the die has a circular orifice with 12 mm diameter.

Enzymatically extruded degerminated maize grits (EEDMG) were obtained by adding 10 U/g thermostable  $\alpha$ -amylase to DMG before extrusion. Extruded degerminated maize grits (EDMG) were obtained without the enzyme. The extrudates were air-dried at room temperature to a constant weight and ground and passed through 20, 40, 60, 80, and 100 mesh sieves. The extrudates with particle sizes of 20-40, 40-60, 60-80, 80-100, and 100+ mesh were labeled as 20-40, 40-60, 60-80, 80-100, and 100+, respectively.

#### 2.2.2 Particle size distribution

The particle size distributions of three samples were determined using a laser diffraction particle size analyzer with a wet dispersion unit (Mastersizer 2000, Malvern Instrument, Ltd., Malvern, UK) according to the method described by Wei et al.<sup>[17]</sup> with slight modifications. After background measurement, the sample was added to the sample cell rendering an obscuration of 10%-20%. The solution was uniformly mixed by stirring and ultrasonic vibration. After the measurement was completed, the instrument automatically gave the particle size distribution curve and characteristic values. Each sample was measured at least three times.

#### 2.2.3 Rheological measurements

The rheological properties of the three samples were determined using a Malvern Kinexus Rotational Rheometer (Malvern Instrument Ltd., Malvern, England) equipped with a coaxial cylinder with inner and outer diameters of 25.0 and 27.5 mm, respectively. The gap between the measuring rotor and cylinder bottom was 0.1 mm. The sample was placed in the annular space between the inner rotor and outer cylinder for measurement. The three different test programs provided different results.

##### 1. Steady shear test

2 treatments were used to determine the stable shear properties of the samples. The first was to prepare DMG, EDMG, and EEDMG samples with different particle sizes (20-40, 40-60, 60-80, 80-100, and 100+ mesh) and the same concentration (10:1 water-sample ratio, v/w), and the second was to prepare DMG, EDMG, and EEDMG samples with the same particle size (100+ mesh) and different concentrations (2:1, 3:1, 5:1, 10:1, and 20:1 water-sample

ratios, v/w).

Viscosity flow curves were obtained at 45°C after 5 min of temperature equilibration at the operating shear rates of 50-500 s<sup>-1</sup>. The flow behaviors of the samples were described by the fluid power law equation of the Ostwald-de Waele law<sup>[18]</sup> as shown in Equation (1).

$$\eta = K\gamma^{n-1} \quad (1)$$

where,  $\eta$  is the apparent viscosity, Pa·s;  $\gamma$  is the shear rate, s<sup>-1</sup>;  $K$  is the consistency coefficient, Pa·s <sup>$n$</sup> ;  $n$  is the flow behavior index that reflects the closeness to Newtonian flow.

##### 2. Temperature sweep test

Temperature sweep test was performed at the fixed shear rate of 200 s<sup>-1</sup> and the shear stress of 1 Pa over the temperature range of 5°C-25°C at a scan rate of 5°C/min. The rheological properties of the flow curve were recorded as apparent viscosity versus temperature. After heating treatment, the samples were immediately cooled down to 25°C.

##### 3. Time sweep test

A time sweep test was performed to study the variation in apparent viscosity with increasing test time (1800 s) at the constant temperature of 45°C and the shear rate of 200 s<sup>-1</sup>. Apparent viscosity was calculated using the power law model as shown in Equation (2)<sup>[18]</sup>.

$$\eta = Kt^{m-1} \quad (2)$$

where,  $t$  is the testing time, s;  $K$  is the consistency coefficient, Pa·s <sup>$n$</sup> ;  $m$  is the flow behavior index.

Root mean square error (RMSE) was used to detect the differences between the experimental data and model estimates to evaluate the performance of the power law model. RMSE was calculated using Equation (3).

$$\text{RMSE} = \left[ \frac{1}{N} \sum_{i=1}^N (\eta_{\text{exp},i} - \eta_{\text{pre},i})^2 \right]^{\frac{1}{2}} \quad (3)$$

where,  $\eta_{\text{exp},i}$  is the experimental apparent viscosity,  $\eta_{\text{pre},i}$  is the predicted apparent viscosity, and  $N$  is the number of model parameters.

### 2.3 Statistical analysis

Significant differences between values were analyzed by one-way ANOVA and Duncan's post-hoc multiple range test ( $p < 0.05$ ) using the SPSS software version 17.0 (SPSS Inc., USA).

## 3 Results and discussion

### 3.1 Particle size distribution analysis

The particle size of a material has a direct influence on its rheological properties<sup>[19]</sup>. Parameters related to particle size are listed in Table 1. These parameters were used to further investigate the relationship between the particle size and apparent viscosity of the samples. D10, D50, and D90 are the maximum diameters of the 10%, 50%, and 90% of the total volume of particles<sup>[20]</sup>. D[4, 3] is the volume-weighted mean diameter that is more sensitive to large particles, and D[3, 2] is the surface-weighted mean diameter influenced by small particles. The D[4, 3] and D[3, 2] values of the three samples varied significantly in the ascending order, (100+)<(80-100)<(60-80)<(40-60)<(20-40) ( $p < 0.05$ ), which exhibited a gradient and relatively concentrated distribution. The D10, D50, and D90 values showed the same trend as D[4, 3] and D[3, 2]. The D10, D50, and D90 values of extruded degerminated maize grits (EDMG) and enzymatically extruded degerminated maize grits

(EEDMG) were higher than those of native degerminated maize grits (NDMG) for the 60-80, 80-90, and 100+ mesh samples. This result indicates that extrusion and enzymatic extrusion treatments

could significantly ( $p < 0.05$ ) increase the particle size at relatively small particle sizes owing to the extrusion expansion effect, which could facilitate the reaction of the enzyme with the starch substrate.

**Table 1** Diameters of NDMG, EDMG, and EEDMG ( $\mu\text{m}$ )

Sample	Particle size range/mesh	D10	D50	D90	D[4, 3]	D[3, 2]
NDMG	20-40	632.73±9.68 <sup>a</sup>	946.44±13.22 <sup>a</sup>	1407.96±13.46 <sup>a</sup>	988.57±12.19 <sup>a</sup>	901.53±12.34 <sup>a</sup>
	40-60	322.61±6.31 <sup>d</sup>	491.86±11.65 <sup>d</sup>	746.37±24.30 <sup>c</sup>	516.85±12.42 <sup>d</sup>	466.66±9.65 <sup>c</sup>
	60-80	164.89±2.39 <sup>f</sup>	254.79±1.39 <sup>f</sup>	383.33±6.81 <sup>d</sup>	264.42±4.69 <sup>f</sup>	201.39±36.22 <sup>e</sup>
	80-100	136.01±0.16 <sup>e</sup>	199.34±0.34 <sup>b</sup>	291.81±0.93 <sup>c</sup>	207.96±0.48 <sup>b</sup>	190.94±0.34 <sup>c</sup>
	100+	17.62±0.08 <sup>g</sup>	102.82±1.42 <sup>j</sup>	205.65±4.78 <sup>e</sup>	109.44±1.58 <sup>g</sup>	50.16±0.42 <sup>e</sup>
EDMG	20-40	584.06±4.69 <sup>c</sup>	845.81±8.32 <sup>c</sup>	1329.21±12.53 <sup>b</sup>	898.89±8.26 <sup>c</sup>	803.62±7.35 <sup>b</sup>
	40-60	318.10±5.78 <sup>d</sup>	488.37±8.34 <sup>d</sup>	742.63±12.66 <sup>c</sup>	512.79±8.84 <sup>d</sup>	462.27±8.10 <sup>c</sup>
	60-80	196.72±1.47 <sup>e</sup>	284.04±3.69 <sup>e</sup>	410.25±13.64 <sup>d</sup>	295.66±5.09 <sup>e</sup>	272.89±2.36 <sup>d</sup>
	80-100	155.16±0.53 <sup>f</sup>	220.51±0.58 <sup>e</sup>	313.20±0.53 <sup>c</sup>	228.69±0.54 <sup>e</sup>	212.53±0.58 <sup>c</sup>
	100+	75.11±0.99 <sup>b</sup>	146.56±1.42 <sup>j</sup>	255.65±2.04 <sup>f</sup>	156.62±1.46 <sup>g</sup>	115.31±0.86 <sup>f</sup>
EEDMG	20-40	559.75±4.22 <sup>b</sup>	865.99±5.34 <sup>b</sup>	1357.14±4.59 <sup>b</sup>	918.45±4.81 <sup>b</sup>	821.37±5.14 <sup>b</sup>
	40-60	313.26±9.68 <sup>d</sup>	482.13±15.51 <sup>d</sup>	735.82±26.83 <sup>c</sup>	506.84±17.23 <sup>d</sup>	456.16±14.77 <sup>c</sup>
	60-80	195.67±3.24 <sup>e</sup>	282.25±1.85 <sup>e</sup>	408.78±13.20 <sup>d</sup>	294.21±3.74 <sup>e</sup>	271.41±1.61 <sup>d</sup>
	80-100	155.04±3.31 <sup>f</sup>	222.29±1.12 <sup>e</sup>	318.13±3.19 <sup>c</sup>	230.82±0.55 <sup>e</sup>	213.82±1.83 <sup>c</sup>
	100+	78.29±0.92 <sup>b</sup>	148.16±1.38 <sup>g</sup>	256.55±2.35 <sup>f</sup>	158.60±1.48 <sup>g</sup>	121.64±1.09 <sup>f</sup>

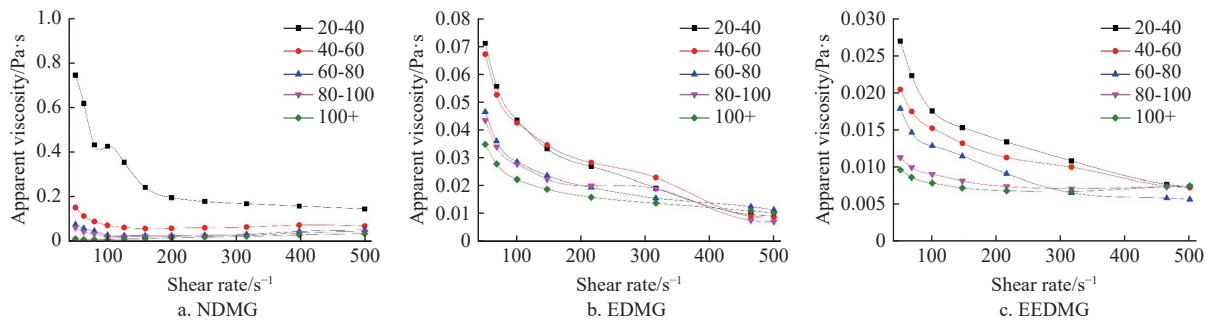
Note: Results are expressed as mean±standard deviation; values with different letters within a column indicate significant differences between means ( $p < 0.05$ ). NDMG, native degerminated maize grits; EDMG, extruded degerminated maize grits; EEDMG, enzymatically extruded degerminated maize grits; D10, D50, and D90 are the maximum diameters of the 10%, 50%, and 90% (total volume) of particles; D[4, 3] is the volume-weighted mean diameter; D[3, 2] is the surface-weighted mean diameter.

**3.2 Steady rheological properties**

**3.2.1 Influence of particle size on apparent viscosity**

Apparent viscosity is an important parameter when evaluating the fluidity or workability of DMG slurries. The flow curves of the three kinds of samples with various particle sizes are presented in Figure 1. All samples exhibited non-Newtonian pseudoplastic flow behaviors, that is, their apparent viscosity decreased with the increase in shear rate, and a constant apparent viscosity was

obtained at higher shear rates<sup>[21]</sup>. The particle size of the sample had a remarkable influence on the apparent viscosity of the slurry. The apparent viscosity of NDMG decreased from 0.74 to 0.01 Pa·s at 50 s<sup>-1</sup> to from 0.15 to 0.03 Pa·s at 500 s<sup>-1</sup>. The results showed that the sample with a smaller particle size had lower apparent viscosity and weaker shear-shinning behavior because the increased specific area of the slurries led to better dispersion and higher solubility of smaller granules and less friction between granules.



Note: 20-40, 40-60, 60-80, 80-100, and 100+ are the different particle sizes of degerminate maize grits, units in mesh. NDMG, native degerminated maize grits; EDMG, extruded degerminated maize grits; EEDMG, enzymatically extruded degerminated maize grit, same below.

Figure 1 Influence of particle size (10% concentration) on the apparent viscosity of three samples at 45°C

The main purpose of liquefaction was partial hydrolysis and loss in viscosity. Based on Figures 1b and 1c, the apparent viscosities of EDMG and EEDMG remarkably decreased from 0.070 to 0.009 Pa·s at 50 s<sup>-1</sup> to from 0.009 to 0.005 Pa·s at 500 s<sup>-1</sup>, indicating that extrusion and enzymatic extrusion were effective for reducing viscosity and simplifying the process of glucose syrup production. Extrusion and thermostable  $\alpha$ -amylase ruptured the starch granules, destroying the organized granule structure completely or partially; breaking amylopectin; producing dextrin, maltose, glucose, and oligosaccharide; and resulting in a rapid decrease in apparent viscosity<sup>[22,23]</sup>. Although the thermostable  $\alpha$ -amylase was slightly inactivated by high shear force and pressure in the extruder, extrusion could disrupt the starch structure and enable

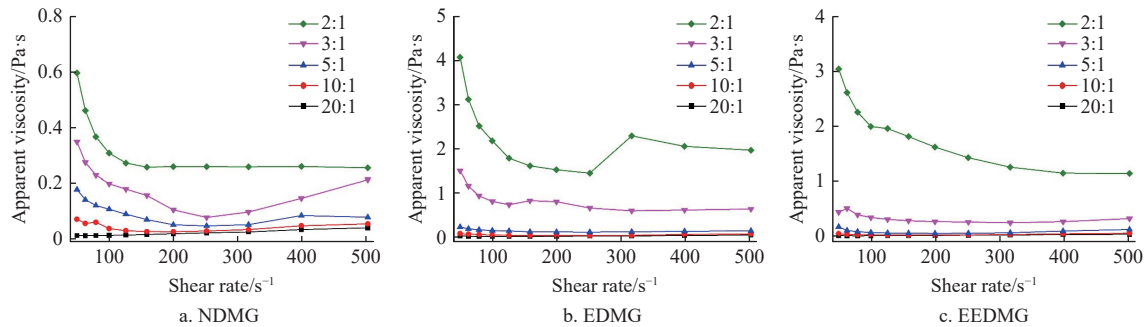
thermostable  $\alpha$ -amylase to penetrate more easily into the starch granules<sup>[24]</sup>. Furthermore, reducing the particle size can effectively reduce the viscosity of maize grits; a lower apparent viscosity can reduce energy consumption and improve enzyme activity in syrup production<sup>[25]</sup>.

**3.2.2 Influence of sample concentration on apparent viscosity**

The apparent viscosities of the samples with different concentrations (water-sample ratios: 2:1, 3:1, 5:1, 10:1, and 20:1; v/w) were plotted against shear rate in the range of 50-500 s<sup>-1</sup> as shown in Figure 2, and the data were fitted using the Ostwald-De Waele power law equation. Relevant parameters are presented in Table 2. The flow curves showed the shear-thinning or pseudo-plastic behaviors of the samples (apparent viscosity decreased with

increasing shear rate). The sample with the water-sample ratio of 20:1 had  $n > 1$  and thus can be classified as a dilatant fluid<sup>[26]</sup>. However, when the water-sample ratios were 10:1, 5:1, 3:1, and 2:1 (the sample concentration increased),  $n$  considerably decreased to 0.384, 0.386, and 0.573 for NDMG, EDMG, and EEDMG, respectively. This outcome is attributed to the increase in sample concentration, because the entanglement of macromolecules, such as starch, and the friction between molecules increased ( $p < 0.05$ ), which resulted in an increase in apparent viscosity<sup>[27]</sup>. The apparent

viscosity of NDMG (0.012 Pa·s) was much lower than those of EDMG (0.025 Pa·s) and EEDMG (0.017 Pa·s) when the water-sample ratio was 20:1. The lower apparent viscosity of NDMG might have resulted from the leaching of a small amount of amylose and the weak intermolecular interactions during the shear rate test; in comparison, the higher apparent viscosity of EEDMG, where molecular flexibility was reduced, might be due to the decrease in relative molecular weight and the shortening of chain length caused by extrusion and enzymatic action<sup>[28,29]</sup>.



Note: 2:1, 3:1, 5:1, 10:1, and 20:1 are the water-sample ratios, v/w.

Figure 2 Influence of various concentrations on the apparent viscosity of the three samples at 45°C

**Table 2**  $K$  and  $n$  values of NDMG, EDMG, and EEDMG with various concentrations at the shear rate of 50-500 s<sup>-1</sup> at 45°C

Water-sample ratio (v/w)	NDMG		EDMG		EEDMG	
	$K$ /Pa·s <sup>a</sup>	$n$	$K$ (Pa·s <sup>a</sup> )	$n$	$K$ (Pa·s <sup>a</sup> )	$n$
20:1	0.001±0.000 <sup>e</sup>	1.557±0.001 <sup>a</sup>	0.005±0.000 <sup>e</sup>	1.316±0.001 <sup>a</sup>	0.004±0.001 <sup>e</sup>	1.293±0.003 <sup>a</sup>
10:1	0.455±0.001 <sup>d</sup>	0.849±0.001 <sup>b</sup>	0.619±0.001 <sup>d</sup>	0.746±0.000 <sup>b</sup>	0.151±0.001 <sup>d</sup>	0.678±0.001 <sup>b</sup>
5:1	1.330±0.002 <sup>c</sup>	0.450±0.001 <sup>c</sup>	1.415±0.001 <sup>c</sup>	0.641±0.001 <sup>c</sup>	0.346±0.002 <sup>c</sup>	0.657±0.001 <sup>c</sup>
3:1	3.110±0.002 <sup>b</sup>	0.405±0.002 <sup>d</sup>	6.168±0.001 <sup>b</sup>	0.521±0.001 <sup>d</sup>	2.085±0.001 <sup>b</sup>	0.621±0.001 <sup>d</sup>
2:1	5.851±0.001 <sup>a</sup>	0.384±0.001 <sup>e</sup>	43.918±0.002 <sup>a</sup>	0.386±0.001 <sup>e</sup>	15.263±0.003 <sup>a</sup>	0.573±0.002 <sup>e</sup>

Note: Results expressed as mean±standard deviation; values with different letters within a column indicate a significant difference between means ( $p < 0.05$ ).

3.2.3 Influence of temperature on apparent viscosity

DMG slurries with water-sample ratios of 2:1, 3:1, 5:1, 10:1, and 20:1 were used to investigate the effect of temperature on apparent viscosity. The apparent viscosities of the three samples at various concentrations at 25°C-95°C are shown in Figures 3a-3c.

The apparent viscosities (less than 1 Pa·s) of all NDMG slurries were somewhat reduced in the first stage at 25°C-75°C because of the accelerated molecular motion caused by constant heating and the insufficient energy available to melt starch crystals. When the temperature increased to 75°C-80°C, the apparent viscosity

increased dramatically at the slurries with ratios of 2:1, 3:1, and 5:1, which indicated the rapid swelling and gelatinization of starch granules. As the temperature continued to rise, the expansion and decomposition of starch granules also led to a continuous increase in apparent viscosity. At 90°C, the apparent viscosity of fully water-absorbing NDMG decreased because of the rupture of the swollen starch granules. Nevertheless, the apparent viscosities of the slurries with water-sample ratios of 10:1 and 20:1 slightly changed to 0.025-0.176 and 0.017-0.023 Pa·s, respectively, because of the low concentration.

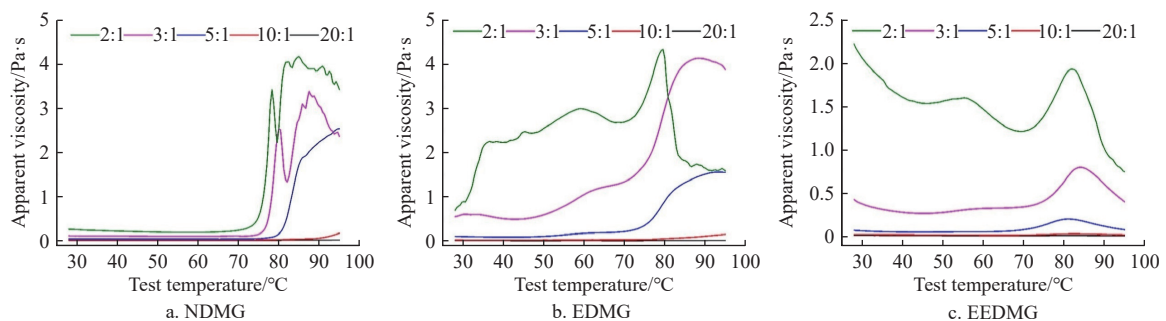


Figure 3 Influence of test temperature (25°C-95°C) on the apparent viscosity of the three samples at 200 s<sup>-1</sup>

The apparent viscosities of the EDMG slurries (Figure 3b) increased from 25°C to 75°C. The apparent viscosity of the EDMG slurry with the water-sample ratio of 2:1 was the highest at 75°C and then began to decrease at 85°C. Moreover, apparent viscosity

began to decrease when the ratio was 3:1. The apparent viscosities of the samples with ratios of 10:1 and 20:1 increased from 0.05 and 0.023 Pa·s at 25°C to 0.158 and 0.025 Pa·s at 95°C, respectively. When the starch granules were subjected to extrusion and enzymatic



extrusion, no intact starch granules could be found and the gelatinization degree was at least 90%<sup>[13]</sup>. After extrusion, the starch crystals were melted, gelatinized starch granules swelled causing the structure to be loose, and broken starch granules dispersed in the starch paste, all of which were responsible for the increase in the apparent viscosity of the EDMG slurries with increasing temperature.

The apparent viscosities of the EEDMG slurries (Figure 3c) decreased at the testing temperatures below 70°C. Apparent viscosity rose to the maximum at 85°C and then declined with the further increase in temperature. EEDMG was subjected to the frictional heat and conductive heat of the screw and barrel and also suffered from enzymatic degradation, which induced more damage to EEDMG than to NDMG and EDMG. The degradation of EEDMG produced a small amount of dextrin and oligosaccharides, increased the water absorption capacity and swelling of the starch, and resulted in a greater apparent viscosity at the same concentrations and low temperature compared with NDMG. The decrease in apparent viscosity after 80°C was caused by the weakening of intermolecular forces and the complete breakdown of the microcrystalline chains into fragments. Apparent viscosity

presented no remarkable difference for the EEDMG slurries with water–sample ratios of 10:1 and 20:1 at 25°C–95°C.

Therefore, we can conclude that at low slurry concentrations (10:1 and 20:1), the system’s stability (apparent viscosity) was not sensitive to temperature, which was favorable for the enzymatic reaction during saccharification.

### 3.2.4 Influence of testing time on apparent viscosity

The previous results demonstrated that particle size has a remarkable effect on apparent viscosity, that is, apparent viscosity decreased as the particle size decreased. Moreover, testing time had an influence on apparent viscosity as shown in Figure 4. The starch slurries were subjected to weak shearing forces. At the same particle size, the highest apparent viscosity was observed in EDMG, and the lowest apparent viscosity was found in NDMG. The increase in particle size might have led to an increase in fraction because granules are closer to each other; thus, apparent viscosity increased. Additionally, granules with small particle sizes tend to be distant from each other, resulting in low apparent viscosity. Curve modeling was achieved using the power law fluid Equation (2), and relevant parameters along with  $R^2$  and RMSE values are listed in Table 3.

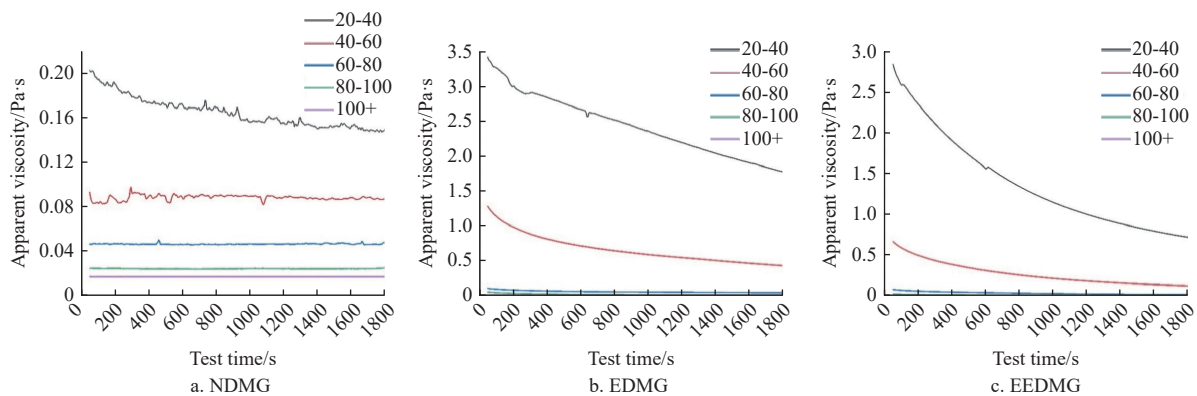


Figure 4 Influence of test time (50-1800 s, 45°C) on the apparent viscosity of the three samples at 200 s<sup>-1</sup>

Table 3 Fitting equations for the rheological properties of samples at different particle sizes over the time of 50-1800 s

Particle size range/mesh	NDMG			EDMG			EEDMG		
	Fitting equation	$R^2$	RMSE	Fitting equation	$R^2$	RMSE	Fitting equation	$R^2$	RMSE
20–40	$\eta=0.304 \cdot t^{-0.091}$	0.9671	0.0035	$\eta=9.068 \cdot t^{-0.202}$	0.8748	0.1571	$\eta=28.349 \cdot t^{-0.470}$	0.9231	0.2408
40–60	$\eta=0.094 \cdot t^{-0.012}$	0.0474	0.0085	$\eta=5.841 \cdot t^{-0.336}$	0.9692	0.0423	$\eta=9.738 \cdot t^{-0.559}$	0.9346	0.0571
60–80	$\eta=0.047 \cdot t^{-0.003}$	0.0520	0.0009	$\eta=0.325 \cdot t^{-0.271}$	0.9942	0.0013	$\eta=0.876 \cdot t^{-0.499}$	0.9130	0.0066
80–100	$\eta=0.025 \cdot t^{-0.002}$	0.1439	0.0003	$\eta=1.324 \cdot t^{-0.708}$	0.9737	0.0038	$\eta=0.432 \cdot t^{-0.625}$	0.9814	0.0014
100+	$\eta=0.017 \cdot t^{-0.0006}$	0.2163	0.0004	$\eta=0.008 \cdot t^{-0.146}$	0.9446	0.0002	$\eta=0.005 \cdot t^{-0.08}$	0.9382	0.0002

Note:  $R^2$ , determination coefficient; RSEM, root mean square error.

Figure 4a shows that the viscosity curves of the NDGM samples with particle sizes of 60-80, 80-100, and 100+ mesh were almost a straight line with  $k$  values of 0.047, 0.025, and 0.017 Pa·s, respectively, and the flow index  $m$  was above 0.99, which was close to the Newtonian fluid and indicates good fluidity.  $R^2$  was very low, except for the sample with 20-40 mesh, where  $R^2=0.9671$ .

The  $R^2$  of EDMG and EEDMG was 0.98, and RMSE ranged from 0.2408 to 0.0002, indicating that the predicted value and the experimental value fit well within the test time range from 50 s to 1800 s. The  $m$  values were less than 1, suggesting that all samples exhibited non-Newtonian pseudoplastic flow behaviors (Table 3). Moreover, the consistency coefficient  $k$  decreased whereas  $m$  increased when the particle size decreased. The  $m$  values of the EDMG and EEDMG with 100 mesh ranged from 0.854 to 0.92,

showing that the slurries were close to Newtonian fluid and had good flowability.

The RMSE values of all samples ranged from 0.0002 to 0.2408, confirming the good fit of the power law Equation (2) to the experimental values, especially for the sample with 100+ mesh, where the RMSE was close to zero, demonstrating that the model had the best fit for the experimental data.

## 4 Conclusions

Apparent viscosity is an important parameter of samples in the saccharification process for glucose syrup production. The rheological properties of NDMG, EDMG, and EEDMG were studied using a Malvern Kinexus rheometer. The three kinds of samples passed through different mesh sieves and showed a

gradient and relatively concentrated distribution. The slurries of these three samples had typical shear-thinning properties. In the shear rate test, the lowest apparent viscosity was obtained by the samples with 100+ mesh and a water-sample ratio of 20:1. Apparent viscosity varied greatly at the water-sample ratios of 2:1, 3:1, and 5:1 and was most stable at 20:1 in the temperature sweep test at 25°C–95°C (apparent viscosity was 0.023–0.025 Pa·s). In the time sweep test, the 100+ mesh samples with high  $R^2$  values (0.9446 and 0.9382) and low RMSE (0.0002) had the best fit to the experimental data for EDMG and EEDMG, respectively. Therefore, the particle size and concentration of samples could remarkably influence apparent viscosity, and the lower apparent viscosities of the EDMG and EEDMG samples with 100+ mesh and 20:1 water-sample ratio can improve activity and accessibility of enzyme and reduce the energy consumption of the glucose syrup production industry. This research provides positive guidance for the effective use of degerminated maize and product development.

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