



Rheological properties of dilute aqueous solutions of cassava starch

Li-ming Che^a, Dong Li^{a,*}, Li-jun Wang^b, Necati Özkan^c, Xiao Dong Chen^{a,*}, Zhi-huai Mao^a

^a College of Engineering, China Agricultural University, 17 Qinghua Donglu, Beijing 100083, China

^b College of Food Science and Nutritional Engineering, China Agricultural University, 17 Qinghua Donglu, Beijing 100083, China

^c Central Laboratory, Middle East Technical University, Ankara 06531, Turkey

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ABSTRACT

The effects of starch concentration on the rheological properties of dilute aqueous solutions of cassava starch were investigated. Cassava starch suspensions at different starch concentrations (0.2, 0.4, 0.6, 0.8, and 1.0 wt. %) were heated at 90 °C for 1 h and then rapidly cooled to 25 °C. The apparent viscosities of starch solutions were measured as a function of starch concentration. The cassava starch solution with a concentration of 0.2% showed Newtonian behavior, and as the concentration was increased beyond 0.4%, the solutions showed shear-thinning behavior. The flow behavior (apparent viscosity against shear rate) of the solutions was well described using a power law model. The consistency indices increased and the flow behavior indices decreased with the increasing of starch concentration. After storing the solutions at 25 °C for 24 h, precipitation of starch took place instead of gelation. The volume of precipitation bed increased linearly with starch concentration.

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1. Introduction

Starch, a major plant source, is extensively used as a thickening and gelling agent in food, as a sizing agent in textiles, and as an adhesive for paper and paper products (Che et al., 2007a; Morikawa & Nishinari, 2000; Roberts & Cameron, 2002). Rheological properties of starch solutions, which are concentration (C) dependent, are important for the use of starch and its mixtures (Kislenko, Oliynyk, & Golachowski, 2006). For a successful product formulation and engineering scale up, the knowledge about rheological properties of starch solutions is necessary (Bhandari, Singhal, & Kale, 2002).

The semi-crystalline starch granules precipitate from cold aqueous suspensions. However, when the starch suspensions are heated above gelatinization temperature, the starch granules lose their crystallinity, absorb large amounts of water, swell to much larger sizes, and leach out amylose, as a result, the viscosity of starch solutions increase considerably (Che, Li, Wang, Chen, & Mao, 2007b; Evans & Haisman, 1979; Kim, Lee, & Yoo, 2006). Gelatinized starch dispersions are non-Newtonian fluids which may also exhibit a yield stress at low shear rates (Evans & Haisman, 1979; Lagarrigue & Alvarez, 2001; Nurul, Azemi, & Manan, 1999; Rao, Okechukwu, Da Silva, & Oliveira, 1997).

Overlap concentration (C^*) is defined as the C at which the polymer molecules in solutions just begin to entangle each other. The

solutions whose C are under C^* are considered as dilute solutions, while the others are called concentrated solutions. C^* is a function of molecule weight (M). And to be detailed, C^* is proportional to $M^{-0.8}$ (He, Chen, & Dong, 1990). For amylose ($M = 5 \times 10^5$ Da), C^* is about 1.5%, while for amylopectin ($M = 10^7$ – 10^9 Da), C^* decreases to 0.9% (Oates, 1997; Ring et al., 1987). Therefore, it is reasonable to assume that C^* of commercial starches composed of amylose and amylopectin is in the range of 0.9–1.5%.

The rheological properties of dilute solutions are different from those of concentrated ones. For starch solutions whose C are above C^* , gelation occurs on cooling whereas, for those whose C are less than C^* , precipitation of starch tend to occur on cooling (Miles, Morris, & Ring, 1985; Ring et al., 1987). The amount of precipitate is proportional to the initial starch concentration of dilute starch solutions at a constant pH (Kislenko et al., 2006). For amylopectin, Ring et al. (1987) found that there was a linear relationship between specific viscosity (η_{sp}) and C at C below 0.9%, while the dependence changed to $C^{2.7}$ in the concentration range 0.9–25%. Thus, they took 0.9% as C^* . However, the earlier study of Miles et al. (1985) indicated that η_{sp} of amylose solutions above C^* was proportional to C^2 .

The rheological properties of concentrated starch solutions have been investigated by many researchers (Karim, Norziah, & Seow, 2000; Lagarrigue & Alvarez, 2001). Chen and Ramaswamy (1999) investigated the effects of C , temperature, pH value, and cooking time on the rheological property parameters (consistency index (K) and flow behavior index (n)) of tapioca starch using a second order composite design. They found that the power law model, Herschel–Bulkley model, and Casson model well described the

* Corresponding authors. Tel./fax: +86 10 62737351.

E-mail addresses: dongli@cau.edu.cn (D. Li), dong.chen@eng.monash.edu.au (X.D. Chen).

Nomenclature		SEP	standard error of prediction, %
C	concentration, wt. %	SPV	specific precipitation volume, %
C^*	overlap concentration, wt. %	$\dot{\gamma}$	shear rate, s^{-1}
c	constant	η_{app}	apparent viscosity, mPa s
M	molecule weight, Da	η_{sp}	specific viscosity
K	consistency index, mPa s ^{<i>n</i>}	τ	shear stress, mPa
N	flow behavior index (dimensionless)	τ_0	yield stress, mPa
R^2	coefficient of determination		

flow behavior of tapioca solutions. Nurul et al. (1999) studied the flow behavior of gelatinized sago starch solutions at various concentration and temperature ranges. The power law model was also used to fit the flow behavior and the effect of temperature was evaluated by the Arrhenius equation. Their results suggested that K increased with the increasing of C and decreased with the increasing of temperature.

Dilute starch solutions are widely used in industry, especially in paper industry, since they can be easily transported through pipes because of their low viscosity. However, only a few studies have been conducted on the rheological properties of dilute starch solutions. Lack of rheological data is one of the problems encountered during numerical simulation and equipment design of starch product processing at low concentrations. The aim of this work was to study the rheological properties of dilute aqueous solutions of cassava starch. The flow behavior and precipitation behavior of dilute cassava starch solutions at different starch concentrations were investigated systematically.

2. Materials and methods

2.1. Materials

Commercial cassava starch was purchased from Beijing Quanfeng Starch Company, China. The cassava was planted in the southwest of China, Guangxi province. The moisture content of the starch was determined by drying sample in an air-oven at 105 °C to constant weight and the value was 9.3 wt. % (wet basis).

2.2. Preparation of cassava starch solutions

Cassava starch suspensions at the concentrations of 0.2, 0.4, 0.6, 0.8, and 1.0 wt. % were prepared by adding required amount of air-oven dried cassava starch into deionized water at room temperature (25 ± 1 °C). Well mixed suspensions in beakers were heated in a water bath at 90 °C for 1 h and then rapidly cooled in another water bath at 25 °C for 0.5 h. The beakers were covered with a preservative film to minimize the loss of moisture during cooking and cooling.

2.3. Rheological studies of cassava starch solutions

Rheological measurements were carried out using a stress-controlled rheometer (AR2000ex, TA Instruments Ltd., Crawley, UK) with concentric cylinder geometry at 25 °C. The concentric cylinder geometry was preferred over other geometries because of the low viscosities of the cassava starch solutions. For each test, 19.6 mL freshly prepared solution was used. Before steady shear experiments, the samples in the geometry were allowed to be equilibrated for 2 min. The shear stress (τ) was then measured as a function of shear rate ($\dot{\gamma}$) which was increased from 1 to 100 s⁻¹ logarithmically. For each point on the flow curves, a constant shear rate was given. Ten points were collected per decade. Four rheological models, including Herschel–Bulkley model ($\tau = \tau_0 + K\dot{\gamma}^n$), power law model ($\tau = K\dot{\gamma}^n$), Bingham model ($\tau = \tau_0 + K\dot{\gamma}$), and modified Bingham model ($\tau = \tau_0 + K\dot{\gamma} + c\dot{\gamma}^2$)

were used to examine the flow behaviors of cassava starch solutions. Where τ_0 is yield stress, c is constant (Al-Malah, Azzam, & Abu-Jdayil, 2000; Sopade & Kiaka, 2001; Yahia & Khayat, 2001).

2.4. Precipitation observation

Cassava starch solutions sealed in glass cylinders (25 mL) were kept in a water bath at 25 °C for 24 h. The level of the interface was recorded and the precipitation behavior was described as specific precipitation volume (SPV) which was calculated from the following equation:

$$SPV = \frac{\text{Volume of precipitation bed}}{\text{Volume of starch solution}} \times 100\% \quad (1)$$

2.5. Evaluation criteria

To evaluate the fitting and prediction accuracy of the reported models, the coefficient of determination (R^2) and standard error of prediction (SEP) were, respectively, employed. The R^2 was calculated by Excel (Microsoft Office 2003). The SEP was calculated from the following equation:

$$SEP = \sqrt{\frac{\sum (\text{Measured value} - \text{Predicted value})^2}{\text{Number of measured values}}} \times \frac{100\%}{\text{Mean of measured values}} \quad (2)$$

3. Results and discussion

3.1. Flow behavior of cassava starch solutions at different concentrations

The flow curves (apparent viscosity ($\eta_{app} = \tau/\dot{\gamma}$) versus shear rate) of the dilute cassava starch solutions with various C at 25 °C are shown in Fig. 1. And the parameters of different rheological models obtained from regression analysis are summarized in Table 1.

As can be seen from Fig. 1, the η_{app} of cassava starch solution with a C of 0.2% does not change with $\dot{\gamma}$ indicating that this starch solution shows Newtonian behavior. When C is increased beyond 0.4%, the starch solutions show shear-thinning or pseudoplastic behavior and the deviation from Newtonian behavior increases with the increasing of C . The pseudoplastic property of gelatinized starch is important in many products since a pseudoplastic material has suspending properties at low shear rates and its viscosity becomes sufficiently low when it is processed at higher rates of shear (Nurul et al., 1999). The non-Newtonian behavior of starch solution was reported by many other researches (Evans & Haisman, 1979; Nurul et al., 1999; Rao et al., 1997). It is accepted that the viscosity of a liquid is a function of the intermolecular forces that restrict molecular motion (Nurul et al., 1999). With the increasing of $\dot{\gamma}$, disentanglement of long chain molecules occurs, as a result, the intermolecular resistance to flow (i.e. viscosity) is reduced. Furthermore, the highly solvated molecules or particles presented in the dispersion medium might be progressively

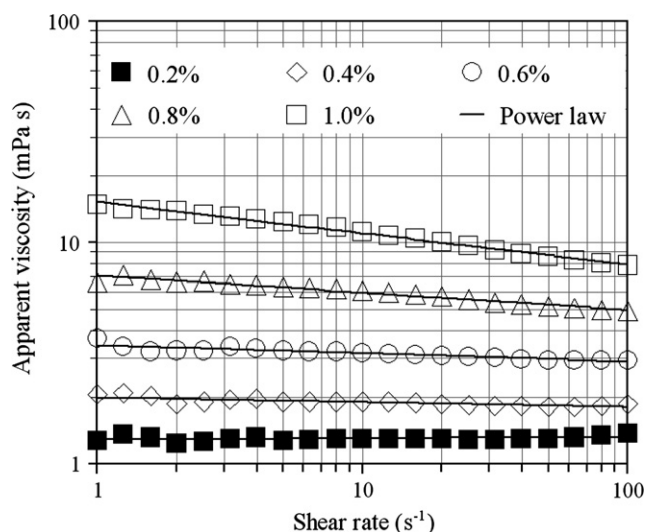


Fig. 1. Flow curves (apparent viscosity against shear rate) for cassava starch solutions with various starch concentrations at 25 °C. Symbols represent the measured values and the predicted results based on power law model are shown with solid lines.

Table 1
Comparison of different models for the flow behavior of cassava starch solutions

Concentration (wt. %)	Model	K (mPa s ^{n})	n	τ_0 (mPa)	R^2
0.2	Herschel–Bulkley	1.174	1.030	0.17	0.9998
	Power law	1.279	1.007		0.9998
	Bingham	1.347		−0.60	0.9994
	Modified Bingham	1.243		0.23	1.0000
	Bingham				
0.4	Herschel–Bulkley	1.808	1.005	0.31	0.9998
	Power law	2.002	0.977		0.9998
	Bingham	1.840		0.16	0.9997
	Modified Bingham	1.764		0.78	0.9999
	Bingham				
0.6	Herschel–Bulkley	3.219	0.980	0.36	0.9997
	Power law	3.448	0.962		0.9997
	Bingham	2.909		1.84	0.9999
	Modified Bingham	2.968		1.37	0.9999
	Bingham				
0.8	Herschel–Bulkley	7.069	0.922	0.03	0.9998
	Power law	7.089	0.922		0.9998
	Bingham	4.897		8.56	0.9986
	Modified Bingham	5.423		4.35	0.9997
	Bingham				
1.0	Herschel–Bulkley	14.989	0.862	0.32	0.9997
	Power law	15.207	0.858		0.9997
	Bingham	7.832		25.40	0.9960
	Modified Bingham	9.250		14.03	0.9993
	Bingham				

sheared away with increasing $\dot{\gamma}$ causing a reduction in the effective size of the particles and hence a reduction in η_{app} (Bhandari et al., 2002; Holdsworth, 1971; Nurul et al., 1999).

It also shows that higher C resulted in higher η_{app} in the tested shear rate range, consistent with what was found earlier by Nurul et al. (1999) and Bhandari et al. (2002). As discussed previously, the viscosity of starch solutions derived from the swelled starch granules and amylose leached out from the granules. Starch molecules entangled by overlapping and interpenetrating into each other at higher C . This limits the water penetration and restricts the fluidity of starch solutions. Consequently, higher η_{app} was always observed at higher C . As $\dot{\gamma}$ increases, the starch molecules often get oriented in the direction of the shear resulting in the decrease of η_{app} . The correlation between η_{app} and C is valuable

for setting guidelines for estimating the rheological behavior of formulated products containing starch (Bhandari et al., 2002).

As can be seen from Table 1, the rheological data for the cassava starch solutions can be adequately described using one of the models since R^2 values for all the models were very close to unity indicating an almost perfect fit. The η_{app} of cassava starch solutions increased as $\dot{\gamma}$ approached zero, which suggested the existence of τ_0 (Rao et al., 1997). Therefore, the Herschel–Bulkley model could be used to describe the flow behavior of the tested cassava starch solutions at different C . The parameters of Herschel–Bulkley and power law models are very close to each other due to the extremely low values of τ_0 . The Herschel–Bulkley and power law models generally gave high R^2 and showed better fit for data points under most other circumstances (Abu-Jdayil, Az-zam, & Al-Malah, 2001; Al-Malah et al., 2000; Chen & Ramaswamy, 1999; Nayouf, Loisel, & Doublier, 2003; Nurul et al., 1999; Rao et al., 1997; Rao & Tattiyakul, 1999; Sopade & Kiaka, 2001). The Bingham model is a special case of Herschel–Bulkley model when n is equal to unity (Abu-Jdayil et al., 2001; Al-Malah et al., 2000). For the values of n in Herschel–Bulkley ranged from 0.862 to 1.030, which were near unity, the studied cassava starch solutions could also be well represented by the Bingham model. The τ_0 values of the cassava starch solutions determined using Herschel–Bulkley model do not show any trend with C suggesting that Herschel–Bulkley model is not suitable for determining τ_0 . Therefore, the τ_0 determined using Herschel–Bulkley model can only be considered as a model fitting parameter not a true τ_0 of the samples. The τ_0 values of the cassava starch solutions determined using Bingham model showed a trend with C excluding the τ_0 value of the starch solution with a C of 0.2%, which was negative (−0.6 mPa). Since a negative τ_0 is not possible, in order to obtain more realistic τ_0 values, the modified Bingham model was used. The relationship between τ_0 determined using modified Bingham model and C is shown in Table 2. It was found that the τ_0 values of cassava starch solutions increased exponentially with the increasing of C . The τ_0 represents a finite stress required to initiate flow. As C increased from 0.2% to 1.0%, the interaction between different starch molecules which restricted molecular motion increased, hence τ_0 increased.

As can be observed from Table 1, K increases with the increasing of C while n decreases. Similar observations were reported by Nurul et al. (1999) for sago starch pastes and Bhandari et al. (2002) for corn starch. The dependence of K on C is frequently represented by a power law type correlation or an exponential function (Lagarigue & Alvarez, 2001). The values of K in Table 1 obtained using power law model were regressively analyzed to investigate the effect of C on K . It was found that an exponential function and a polynomial function were suitable for describing the relationship between K and C in this study (see Table 2). Similarly, the relationship between n and C was described using a polynomial function as shown in Table 2.

Values of n less than unity indicate a shear-thinning behavior while values greater than unity suggest a shear-thickening behavior (Al-Malah et al., 2000). Excluding the solution with a C of 0.2%, all the cassava starch solutions show shear-thinning behavior, for

Table 2

Relationships between the parameters of rheological models and starch concentration for the cassava starch solutions with starch concentrations ranging from 0.2% to 1.0%

Parameter ^a	Equation	R^2
K	$K = 0.61e^{3.1C}$	0.9879
	$K = 39.1C^3 - 40.1C^2 + 17.0C - 0.8$	0.9999
n	$n = -0.41C^3 + 0.57C^2 - 0.36C + 1.06$	0.9982
τ_0	$\tau_0 = 0.0871e^{4.9702C}$	0.9890

^a K and n are from the power law model, τ_0 is from the modified Bingham model.

their values of n in power law model are less than unity. The value of n in power law model for the cassava starch solution with a C of 0.2% was almost unity, so this solution can be considered as a Newtonian fluid.

All the models offered an excellent fit to the rheological data for the cassava starch solutions, but Bingham and Herschel–Bulkley models can not provide true yield stress information. Thus, it is possible to say that power law model is the most appropriate model for describing the rheological behavior of cassava starch solutions with C ranging from 0.2% to 1.0% (see Fig. 1). After the empirical equations describing the relationships between K and C and n and C have been inserted into the power law model, we obtain the following equation:

$$\eta_{\text{app}} = (39.1C^3 - 40.1C^2 + 17.0C - 0.8)\dot{\gamma}^{(-0.41C^3 + 0.57C^2 - 0.36C + 0.06)} \quad (3)$$

Eq. (3) was used to predict the η_{app} of cassava starch suspensions as a function of C and $\dot{\gamma}$. Fig. 2 shows the relationship between the measured and predicted η_{app} of the cassava starch solutions. As can be seen from Fig. 2, the η_{app} of cassava starch solutions with various C ranging from 0.2% to 1.0% can be adequately described by Eq. (3).

3.2. Precipitation observation of cassava starch solutions at different concentrations

The photograph of cassava starch solutions at different C after 24 h storage at 25 °C is illustrated as Fig. 3. The starch precipitated from the cooled solutions forming opaque flocculent structure, and the volume of precipitation bed increased with the increasing of C . The precipitation might be attributed to the recrystallization of amylose. The dilute aqueous solutions of amylose are inherently unstable at room temperature. Amylose precipitates from solutions after a limited period of time (a few hours). The precipitated amylose yields an X-ray powder diffraction pattern of B-type (Miles et al., 1985).

The SPV was regressively analyzed (intercept was taken as 0) as a function of C , and the result is shown in Fig. 4. The regression equation is adequate to fit the experimental data with high R^2 (0.97). It was found that SPV increased linearly with C . In dilute solutions, each starch molecule occupies certain space and the interaction between two molecules is negligible (He et al., 1990).

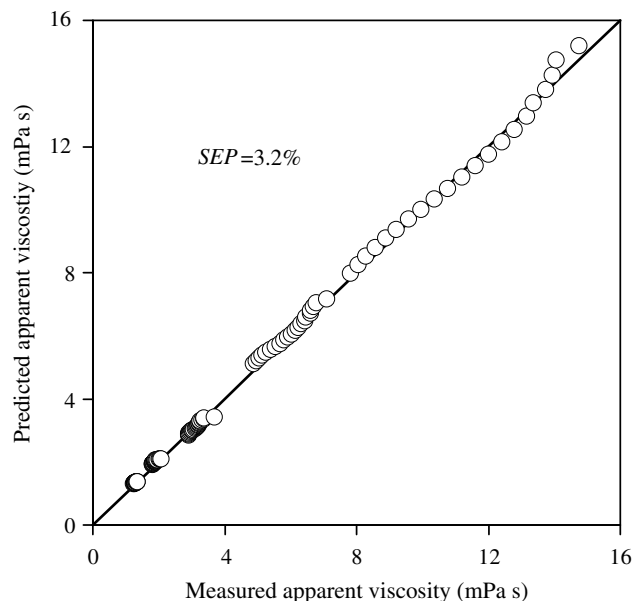


Fig. 2. Comparison between the measured and predicted apparent viscosities of the cassava starch solutions with starch concentrations ranging from 0.2% to 1.0%.



Fig. 3. Photograph of the cassava starch solutions at different concentrations after 24 h storage at 25 °C.

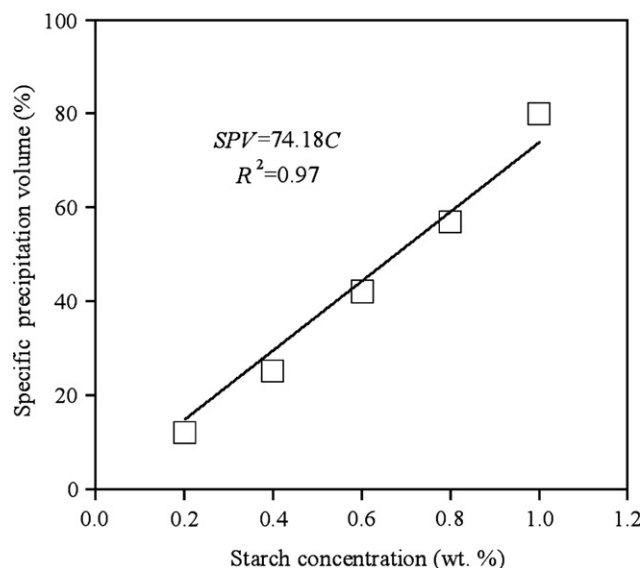


Fig. 4. Relationship between specific precipitation volume and cassava starch concentration after 24 h storage at 25 °C.

So the volume of precipitation bed is only the function of starch content. The volume of the cassava solutions is fixed in this study, so the volume of precipitation bed is in direct proportion to C . The viscosity of continuous phase would affect starch precipitation. However, the settling time in this study (24 h) was sufficient for complete precipitation. So the influence of continuous phase could be neglected. Calculated from the corresponding regression equation, the C at which SPV increases to 100%, which means no precipitation, is about 1.35%. This C can be regarded as C^* , since the

precipitation phenomena distinguishes dilute solutions from concentrated ones. This finding indicates the potential of determining C^* of starches by precipitation observation.

4. Conclusions

The influence of starch concentration on the rheological properties of dilute aqueous solutions of cassava starch was investigated. The dilute cassava starch solutions exhibit some special properties differ from those of concentrated solutions. The apparent viscosity of cassava starch solutions increased with the increasing of starch concentration. The cassava starch solutions changed from Newtonian fluid to shear-thinning one when the starch concentration was increased beyond 0.4%. Herschel–Bulkley, power law, and Bingham models were suitable for representing the flow behavior of the tested samples. Herschel–Bulkley and Bingham models were not suitable for predicting the yield stress of the solutions used in this study. The consistency indices increased with the increasing of starch concentration while flow behavior indices decreased. The apparent viscosity of cassava starch solutions as a function of starch concentration and shear rate was successfully predicted using an equation based on power law model. After storing the cassava starch solutions at 25 °C for 24 h, precipitation of starch took place instead of gelation. The volume of precipitation bed increased linearly with starch concentration. The result suggests a novel method for determination of overlap concentration of starches by precipitation observation.

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