Original Article

A novel method for measuring dissolution kinetics of pulverized konjac flour

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The aim of the current study was to explore a novel method for measuring hydration and dissolution kinetics of the pulverized konjac flour (PK flour) from *Amorphophallus albus* using RVA-3D$^+$ Rapid Visco Analyzer (RVA; Newport Scientific Pty Ltd., Australia). The results showed that RVA was a reliable fast technique for determining the hydration curve of PK flour. The test conditions determining the hydration curves were optimized at the concentration of PK flour with 1.0%, test temperature at 30±1˚C, stirring speed at 160 RPM (Revolution Per Minute), and test time of 16 min. An empirical exponential model has also been established to describe the dissolution kinetics of PK flour at the concentration of 1.0%: $\eta = 161.9343 \cdot EXP(-2.1522/\tau)$ (R^2=0.9762) Where τ is the test time (min); η is the viscosity of the hydration process (RVU) of PK flour. The results also showed that a significant difference among the hydration curves of 1.0% PK flour when dispersed in distilled water and in different concentrations of sucrose aqueous solution.

Key Words: dissolution kinetics, hydration, konjac, Rapid Visco Analyzer

Introduction

Soluble fiber is known to have beneficial effects on gut function and metabolism in humans and experimental animals.[1-2] Study has shown that high intake of konjac glucomannan or the other type soluble dietary fibers improves glycemic control, decreases hyperinsulinemia, and lowers plasma lipid concentrations in patients with type 2 diabetes.[3] The metabolic effects for these hydrocolloids are considered to be of clinical value in the dietary management of diabetes mellitus.[3-6] The beneficial biological activity of soluble fibers were known to strongly dependent on the capacity of these hydrocolloids to hydrate and increase the viscosity of digesta in the stomach and small intestine, whether or not the polymer hydrates to form a molecular dispersion.[7-8]

For instance, the blood glucose-lowering effect of guar gum is most likely a result of viscosity development in the stomach and small intestine in the early post-prandial period.[9-10]

Pulverized konjac flour (PK flour) mainly composed of glucomannan, which could be taken orally as dietary fiber. New immunomodulatory and physiological functions were reported by oral administration of PK flour in BALB/c mice.[11-13] But the particle size distribution, structural properties of PK flour and hydration conditions in the gut environment inevitably affect the hydration properties and the biological activity of PK flour.

The studies on in vitro investigation could facilitate our understanding for the behavior of PK flour in the gut lumen and also provide useful information about the types, doses and mode of administration of PK flour that should be used to optimize its therapeutic effects.[14-15] Studies of the factors that affect the dissolution properties of PK flour are obviously essential to understand the physiological mechanisms and optimize the clinical efficacy of oral intake of PK flour.

Methods and empirical logarithmic models have been developed for describing the hydration kinetics of guar gum powders and other hydrocolloid powders.[16-21] But the research on hydration process of PK flour is still limited partly because of the availability of analytical method. Thus, a precise and rapid rheological method for measuring hydration and dissolution kinetics of PK flour should be much broader interested especially for clinic evaluation of functionalities of the well-known soluble dietary fiber.

The Rapid Visco Analyser (RVA) is widely used to determine the viscous properties of starch slurries.[22] Rotation of the pitched paddle keeps the starch particles suspended duration of the testing time. In addition, It can be able to continuously record the data of viscosities during the hydration process.

The objective of this study was to explore RVA for determining the hydration kinetics of PK flour and set the appropriate test parameters for the PK flour sample. The dissolution kinetics were further quantified by an empirical model through the experimental data.

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Table 1. The physicochemical properties of the pulverized and the ethanol refined konjac flour

Samples	Average Particle Size*(μm)	Moisture (%)	Ash (%)	Protein (%)	SO_2 Content (g/kg)	KGM Content (%)	Peak Viscosity** (Pa.s)	Time to Reach Peak Viscosity (hour)
Ethanol refined konjac flour	109.4	11.6	3.28	2.02	0.09	85.5	16.4	0.9±0.2
Pulverized konjac flour	95.4	10.2	2.98	1.78	0.10	89.5	11.5	0.5±0.1

*The average particle size and size distribution of these PK flours were measured by a Laser particle size analyzer JL-1166 (Jinxin, Chengdu, P R China). **peak viscosity in1.0%(m/m) solution was measured by a rotational type viscometer (NDJ-8S,Shanghai,PR China) at 30±1℃, No.4 spindle, rotational speed of 12r/min. ***All the other data were obtained in according to the industrial standard for Konjac Flour (NY/T494-2002), Ministry of Agriculture, P R China.[24] Data were presented in Table1 as means of duplicate.

Materials and Methods

Materials

The pulverized konjac flour (PK flour) was obtained by pulverization of ethanol refined konjac flour through an oscillatory-type ball-mill (Qiangwei, Kunshan, China).[23-24] Ethanol refined konjac flour was obtained by milling raw konjac flour in 30-35% (v/v) ethanol, washed and dehydrated stepwise through 35-80% (v/v) ethanol thereafter. The raw konjac flour was separated from *Amorphophallus albus* cultivated in Liangshan, Sichuan Province.

The physicochemical properties of the pulverized konjac flour and ethanol refined konjac flour are shown in Table 1.

Measurements

0.25g PK flour was weighed. It was then put into the RVA sample canister. And 25.00ml distilled water was added into the canister. The viscosity development during the hydration process was immediately monitored by The RVA instrument. As a result, the hydration profiles (i.e. hydration curves) can be generated.

To find the optimum duration of test time, the test temperature, and stirring speed were set at 30℃±1℃ and 160 RPM, respectively; two duration of test times (16min or 60min) were compared.

For optimum test temperature, stirring speed and duration of test time was set at 160 RPM and 16 minutes respectively. Three test temperatures 30℃±1℃, 40℃±1℃, and 50℃±1℃, were compared.

For the optimum stirring speed, test temperature and duration of test time were fixed at 30℃±1℃, and 16 min. respectively; there stirring speeds, 75, 160 and 320 RPM were tested.

For the optimum concentration of PK flour, test temperature, stirring speed, and duration of test time were fixed with 30℃±1℃, 160 RPM and 16min. Four concentration of PK flour at 0.5%, 1.0%, 1.5%, 2.0% (on a w/v% and on a dry basis) were tested.

Effect of sucrose

The test temperature, the stirring speed, and duration of test time were set at 30℃±1℃, 160 RPM, 16min. PK flour (0.25g dry basis) was weighed into the RVA sample canister, then 25mL of distilled water or aqueous solution containing different concentrations (w/v) of sucrose (A.R.) was added for each test. The samples were then analyzed as described above.

Statistics and modelling

The real-time data of viscosities was recorded in the RVA

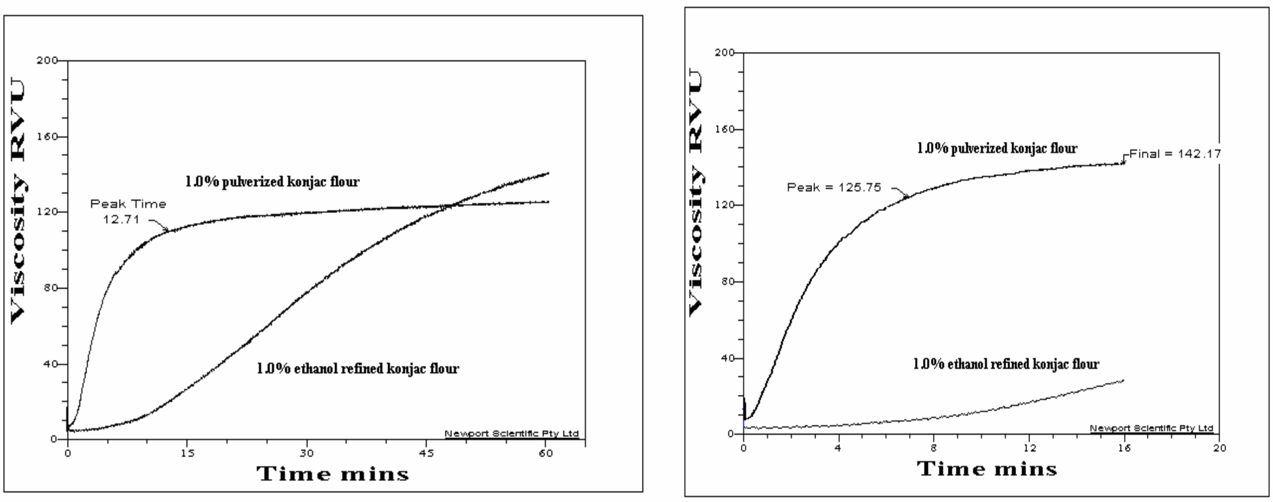

Figure 1. Hydration curves of 1.0% pulverized konjac flour and ethanol refined konjac flour (Instrumental Setup: Test Temperature 30±1℃; Stirring Speed 160r/min.) RVA parameters for 1.0% pulverized konjac flour determined at the end of 16min, the peak viscosity: 128.38±4.58[a];the final viscosity: 143.62±5.00[a];Peak/Final (%)=88.92%. [a] Mean ± standard deviation of triplicate.

unit versus test time. The RVA parameters including peak time, peak viscosity, final viscosity were determined from the hydration curves (hydration profiles) by Thermocline for Windows, Version 1.2. The evaluated RVA parameters were determined in Mean ± standard deviation of triplicate in order to confirm the reproducibility of data. Data processing and regression analysis were carried out by a software program of DPS (Data Processing System, School of agriculture and biotechnology, Zhejiang University, Hangzhou, China).

Results and discussion

Setting test conditions

For duration of test time, the results were shown in Fig 1. The hydration curve with 1.0% PK flour showed a smooth and steady curve. It took only 12.71min to reach to the peak viscosity. The curve then reached a plateau regime and got to the final viscosity at the end of 60 minutes. With the duration of test time at 16 min, the peak viscosity could be 85% more than the final viscosity measured at the end of 16min. Therefore, the test time of 16min was enough to show the hydration and dissolution properties of 1.0% PK flour. On the other hand, the viscosity was not able to reach the peak value for 1.0% Ethanol refined konjac flour even though the duration of test time was lasted to 60 minutes. The results on this will be shown on another paper submitted by the authors.

Fig 2. shows the results for test temperature effect. The results indicate that increased temperature greatly speeded-up the hydration process and decreased the final viscosity for 1.0% PK Flour as well. Since the PK flour could hydrate to form enough high viscous aqueous solution at 30℃, this temperature was chosen in this experiment.

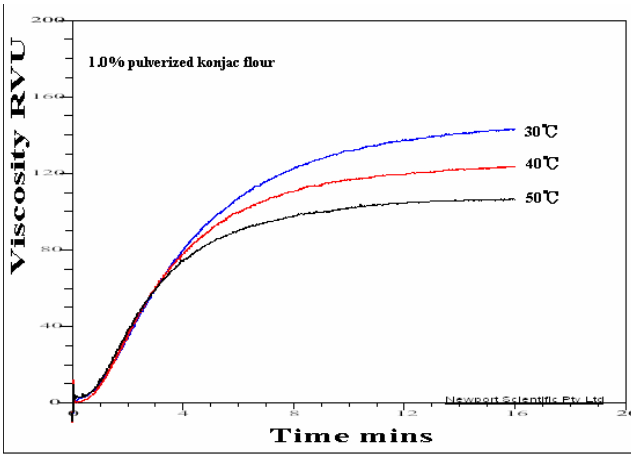

Figure 2. Hydration curves of 1.0% pulverized konjac flour r at different test temperature (RVA Setup: Stirring Speed 160r/min; Test Time 16 min)

The effect of different stirring speeds on the hydration of 1.0%PK Flour was shown in Fig.3.The results showed the smooth hydration curves generated from 160 and 320 RPM. However, the figure indicates that the hydration curve generated from 320 RPM did not reach the plateau regime. With the stirring speed of 75r/min, an unstable and jagged curve was obtained (Fig3). Therefore, the stirring speed was chosen as 160 RPM in the experiment.

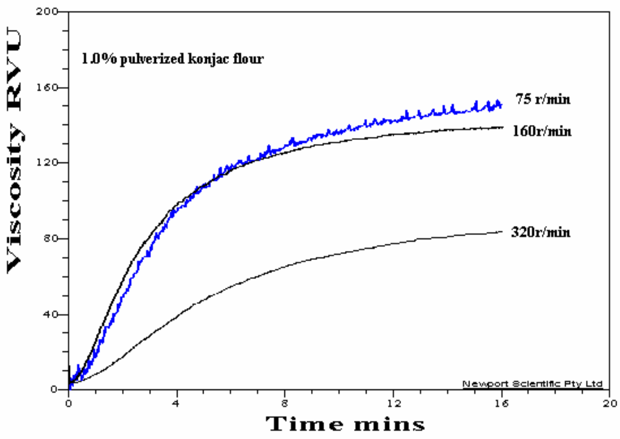

Figure3. Hydration curves of 1.0% pulverized konjac flour with different stirring speeds (RVA Setup: Test Temperature 30±1℃; Test Time 16 min)

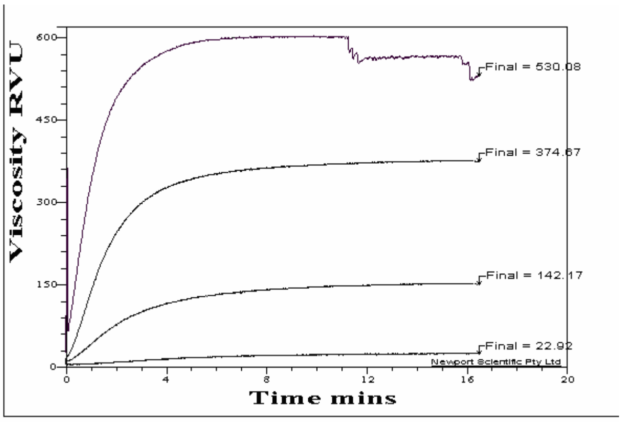

Figure 4. Hydration curves of pulverized konjac flour in different concentrations (RVA Setup: Test Temperature 30±1℃; Stirring Speed 160r/min; Test Time 16 min)

Effects of PK flour concentration

As might be expected, PK Flour concentration had a significant effect on the hydration rate. There was a positive relationship between the flour concentration and the hydration rate. The hydration rate increased as the increase of concentration of PK Flour (Fig 4).

The results indicated that the hydration curve at the concentration of 2.0% showed very unstably. This was because the paste became too viscous to keep in homogeneous during the test process. In addition, the peak viscosity of hydration curve with 0.5% PK flour was too low to be used for comparison purposes. However, the hydration curves of 1.0% and 1.5% PK flour were smooth and steady. Therefore, the test concentration of PK flour with 1.0% was a good choice for an intermediate concentration in this kind of study.[17]

Effect of sucrose

The results in Fig 5 showed that a significant difference among the hydration curves of 1.0% PK flour when dispersed in distilled water and in different concentrations of sucrose aqueous solution. The hydration curves showed clearly that a high concentration of sucrose significantly suppressed the dissolution process of PK flour. The hydration rate decreased with increasing sucrose concentra-

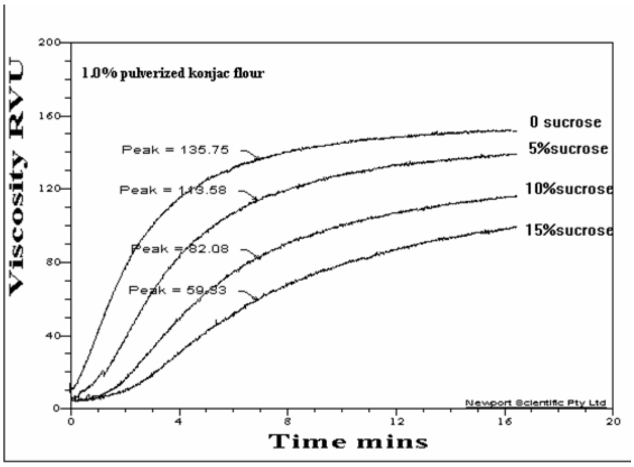

Figure 5. Hydration curves of 1.0% pulverized konjac flour dispersed in water and in different level of sucrose solutions (RVA Setup: Test Temperature 30±1℃; Stirring Speed 160r/min; Test Time 16 min)

Table 2. Analysis of variance (ANOVA) for the regression model

Variance Source	Square Sum	df	Mean Square	F	Significance level
Regression	18221.28	2	18221.28	3662.01	p<0.001
Surplus	69.6606	14	4.97576		
Sum	18290.94	15	1219.396		

tion and the final viscosity of the process was showed inversely proportional to the concentration of the sucrose solution, probably due to the possible interaction between sucrose and konjac glucomannan.[25] The absence of a precipitate suggests that sucrose molecules adsorbed on the surface of pulverized konjac particles, creating a coating that competes with the water molecules. This would prevent swelling of the polysaccharide chains.

Modeling

Data from hydration process of 1.0% PK Flour was used to fit an exponential model. The established regression model was:

$$\eta = 161.9343 \cdot EXP(-2.1522/\tau) \quad (R^2=0.9762) \quad (1)$$

Where τ is the test time (independent variable, min); η is the viscosity of the hydration process (RVU). Analysis of variance (ANOVA) for the regression model was summarized in Table 2.

The result of the ANOVA in Table 2 showed that the model's coefficient of determination (R^2) is 0.9762, which is close to 1.00; the model's significance level (p) was less than 0.01, which indicated that the data of viscosity generated from 1.0% PK flour in the hydration process could be well fitted by the regression model. In other words, the following mathematical model (negative exponential growth function) could be used for describing the dissolution kinetics of 1.0% PK flour:

$$\eta=\eta_{max} \cdot EXP(-b/\tau) \quad (2)$$

Its linear transformation equation is:

$$\ln \eta = \ln \eta_{max} - b/\tau \quad (3)$$

In equation (3) η_{max} is the attainable maximum viscosity. When $\tau \to \infty$, $\eta=\eta_{max}$; b is the rate constant for the linear transformation of exponential equation. When $\eta=\eta_{max}/2$, $\tau_{0.5}$=1.4427 b is the time to reach the half of maximum viscosity. Therefore, the rate constant b of the equation or $\tau_{0.5}$ could be used as indicating parameters for the hydration rate of PK flour. The longer is $\tau_{0.5}$, the slower is the hydration process.

Conclusions

A method for determination of hydration and dissolution kinetics for well-dispersed PK flour was developed using RVA. The optimum test concentration for determination of hydration curves is 1.0%. The optimum test temperature, stirring speed, and test time were 30±1℃, 160 RPM, and 16 min, respectively. The RVA method described here not only showed very good reproducibility, but also a solution to the two most difficult issues that often face how to measure hydrocolloid dissolution kinetic, i.e. random formation of lumps and their stickiness to the stirrer. It also could accurately quantify the hydration rate of PK flour at the concentration of 1.0% by an empirical exponential model established through the hydration data determined by RVA. The application of the model to an in vitro digestion system for studying the viscosity development of foods/diets containing PK flour is still needed. Moreover, the model has much broader relevance to the hydration of other high-molecular weight polymer powders, such as guar gum powders.

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