

## Gelling properties of extruded yam (*Dioscorea alata*) starch

R.M.L. Alves\*, M.V.E. Grossmann, R.S.S.F. Silva

Department of Food Science, Londrina State University, Caixa Postal 6001, 86051-970 Londrina, PR, Brazil

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### Abstract

Yam (*Dioscorea alata*) starch was pregelatinized in a single screw extruder. Response Surface Methodology was used to study the effect of extrusion variables: feed moisture (18, 21, 24%), extruder temperature (120, 150, 180°C) and die diameter (3, 4, 5 mm) on cold viscosity, gel-forming capacity and retrogradation of extruded starches. Cold viscosity was higher from starch samples processed at higher moisture and die diameter, at lower temperatures. Slurries of the extruded starch (8%, w/v, d.b.) formed opaque and firm gels. Higher values for gel strength were found in samples extruded at highest and lowest moistures at intermediate temperature and increasing die diameter. Gels of extruded starch had lower retrogradation values than non-extruded gelatinized starch after one and two weeks storage at refrigeration temperature. Prediction equations for the studied functional properties can be used in selecting processing conditions for specific food applications. © 1999 Published by Elsevier Science Ltd. All rights reserved.

### 1. Introduction

Starch is an important ingredient for the food industry. As new food products are developed, starches with specific properties are necessary to impart functionally desirable attributes. Pregelatinized starches play an important role in instant foods, and they have more recently been prepared mainly by extrusion cooking because of various advantages of this process over other traditional methods. By changing raw materials and process parameters it is possible to obtain pregelatinized starches with different functional characteristics.

In Brazil, corn (*Zea mays*) and cassava (*Manihot utilissima*) are the principal sources of starch for the food industry, yam (*Dioscorea alata*) is being studied as an alternative source because of several desirable properties of its starch, such as, stability to high temperature and low pH. However, the starch gels of *D. alata* show very high retrogradations which create a disadvantage that would make them unsuitable for food systems, (Rosenthal, Pelegriño & Correa, 1972).

Preliminary tests showed that, when yam starch was extruded, the gel retrogradation tendency was decreased. The objective of the current research was to utilize Surface Response Methodology to study the effects of extrusion variables (moisture, temperature and

die diameter) on yam starch as related to viscosity, gel-forming capacity and retrogradation.

### 2. Materials and methods

The experiments were realized using yam (*D. alata*) tubers, of a commercially grown land cultivar from Londrina, Brazil.

#### 2.1. Starch isolation and purification

Starch was isolated and purified using modified procedures of Willinger (1964) and Rosenthal and Spindola (1969). Tubers were rinsed, peeled, diced and then, disintegrated in a domestic blender in a sodium bisulfite–water solution (750 ml/l). The mixture was sieved (0.250 mm screen) and the solids retained were exhaustively rinsed on the sieve with the sodium bisulfite solution. The filtrate was allowed to stand at room temperature and the decant was discarded. The starch sediment was treated with 0.15 and 0.10% sodium hydroxide solution, and then, exhaustively rinsed with distilled water until pH 7 on a sieve (0.105 mm). The decants were discarded. The starch sediment was rinsed with ethanol (70%) and the alcohol evaporated. After drying in a convection oven at 40°C until 12% moisture, the starch was ground with a mortar and pestle to pass a 0.105 mm sieve and then stored in polyethylene bags.

\* Corresponding author.

## 2.2. Chemical composition of starch

Protein (N×6.25), ash, moisture and lipid content of yam starch were determined according to American Association of Cereal Chemists (1983) approved methods 46-13, 08-01, 44-15 A and 30-26, respectively. Starch was calculated as ICC Standard no. 123/1 (ICC-Standards, 1994). Each determination was made in triplicate.

## 2.3. Experimental design and statistical analysis

The experiment was conducted using an incomplete factorial design (Box & Behken, 1960) with three independent variables at three levels of variation: sample moisture (18, 21 and 24%), extrusion temperature (120, 150 and 180°C) and extruder die diameter (3, 4 and 5 mm). The three levels of each variable were coded as -1, 0, +1, for statistical analysis. The design consisted of 15 treatments, which included triplication of the center point (Table 1). Dependent variables were cold viscosity, gel strength and retrogradation. Experimental data were analyzed using the Statistical Analysis System (SAS, 1989), to fit second order polynomial equations to response variables. Surface plots were drawn using the

Statistica (StatSoft, OK) computer software to show the effect of two independent variables while the other was held constant.

## 2.4. Extrusion

Samples of yam starch were processed randomly under the different experimental conditions in a single screw laboratory model extruder (Cerealtec CT-L15, Campinas, Brazil). The extruder has a barrel 19.4 mm in diameter and 420 mm in total length, three zones (each 140 mm in length) and die assembly electric heaters, and a 3:1 compression ratio screw. Processing temperature in zone 1 was kept constant at 80°C, while zones 2, 3 and the die had temperature and diameter as indicated by the experimental design. Feed rate was maintained at 90 g/min, and the screw speed was 150 rpm. The extruder was operated at steady state for each set of conditions. Attainment of steady state was judged by constant amperage. Samples (500 g, w.b.) were then collected, dried at 50°C in a convection oven to 12% moisture, ground in a pin mill (Alpine, model 160 Z, Augsburg, Germany) fitted with a 0.5 mm screen, and sieved with the fraction passing a 0.178 mm screen used for further study.

## 2.5. Properties of pregelatinized starches

Cold viscosity was determined in a Brabender viscoamylograph (Brabender Instruments, Germany) equipped with a sensitivity cartridge of 700 cmg. Extruded starch suspensions (8% w/v, d.b.) were prepared using 450 ml of water and transferred quantitatively to the viscoamylograph container. Viscosity was determined at 30°C and 75 rpm.

Gel strength measurements were made using a Stevens-LFRA Texture Analyzer (Texture Technologies Corp, NY). The starch gels were prepared suspending starch (8% w/v, d.b.) in water and 20 ml of the formed pastes were poured into glass tubs (2 cm in diameter and 6 cm high). These were sealed with plastic film and stored at 5°C for 24 h before texture analysis. Gel strength was defined as the force required for a cylindrical plunger 12.5 mm in diameter to penetrate 4 mm into gel at 1 mm/s. These parameters were chosen in order to obtain accurate and more reproducible results.

Table 1  
Experimental design

Run	Coded variables <sup>a</sup>			Real variables <sup>a</sup>		
	$X_1$	$X_2$	$X_3$	$X_1$	$X_2$	$X_3$
1	-1	-1	0	18	120	4
2	+1	-1	0	24	120	4
3	-1	+1	0	18	180	4
4	+1	+1	0	24	180	4
5	-1	0	-1	18	150	3
6	+1	0	-1	24	150	3
7	-1	0	+1	18	150	5
8	+1	0	+1	24	150	5
9	0	-1	-1	21	120	3
10	0	+1	-1	21	180	3
11	0	-1	+1	21	120	5
12	0	+1	+1	21	180	5
13	0	0	0	21	150	4
14	0	0	0	21	150	4
15	0	0	0	21	150	4

<sup>a</sup>  $X_1$  = moisture (%);  $X_2$  = temperature (°C);  $X_3$  = die diameter (mm).

Table 2  
Regression models for response variables

Response variable	Regression model <sup>a</sup>	$R^2$
Cold viscosity	$Y = 160 + 27.5X_1 - 30X_2 + 12.5X_3 - 20X_1^2 - 25X_2^2 + 50X_3^2 + 15X_1X_2 + 50X_1X_3 - 15X_2X_3$	0.87
Gel strength	$Y = 105.89 + 10.58X_1 - 19.5X_2 + 10.66X_3 + 20.55X_1^2 - 30.44X_2^2 + 6.05X_3^2 - 0.5X_1X_2 - 4X_1X_3 + 0.33X_2X_3$	0.81
Retrogradation	$Y = 1.61 + 0.53X_1 - 0.69X_2 + 0.02X_3 - 0.12X_1^2 - 0.54X_2^2 + 0.001X_3^2 - 0.1X_1X_2 + 0.64X_1X_3 - 0.08X_2X_3$	0.88

<sup>a</sup>  $X_1$  = moisture;  $X_2$  = temperature;  $X_3$  = die diameter.

Gel retrogradation was studied using Biliaderis' (1982) method with some modifications. Starch pastes (10% w/v, d.b.) were prepared by suspending extruded starch in water and pouring into glass recipients (2 cm in diameter and 6 cm high). The recipients were sealed with plastic film and stored at 10°C. After 7 and 15 days, the water released was removed and the percent of weight decrease expressed as retrogradation. The values of gel strength and retrogradation for each sample were the average of three determinations.

### 3. Results and discussion

The analysis of the chemical composition of yam starch used in the experiment showed the following values expressed as dry base percentage; ash 0.15, lipids 0.15, protein 0.20, starch 98.30, and other carbohydrates 1.20. These values are near to those expected in tuber starches and similar to those reported by Kay (1973).

Regression models fitted to experimental results of starch properties (Table 2) showed good correlation coefficients ( $\geq 80\%$ ) for all functional properties. However, variation coefficients were considered high (21.1, 20.9 and 38.2% for viscosity, gel strength and retrogradation, respectively). The models should be used with caution, but they can be considered adequate to study response tendencies (Joglekar & May, 1987).

Cold viscosity was affected mainly by the quadratic effects of die diameter ( $p=0.038$ ) and diameter  $\times$  moisture interaction ( $p=0.033$ ). The linear effects of moisture and temperature were also significant ( $p=0.073$  and  $p=0.057$ , respectively). Response surfaces (Fig. 1) show that cold viscosity decreased when moisture and die diameter decreased and temperature increased. This was expected as harsher extrusion conditions promote starch degradation inducing lower viscosity (Owusu-Ansah, van de Voort & Stanley, 1983).

Die diameter inversely affected viscosity, when moisture levels were low or high. Lower moisture extrusion reduced viscosity with increase of die diameter while high moisture increased viscosity with increasing die. The same pattern was observed by Grossmann, El-Dash and Tavares (1989) when extruding cassava starch.

Cold viscosity is an important property if the extruded starch will be used as an ingredient in foods that require cold thickening capacity, like instant soups, creams or sauces. For these applications, it would be interesting to process yam starch at higher moisture (24%) and die diameter (5 mm) at lower temperatures (120°C), to obtain the highest cold viscosity (285 Brabender Units).

Gel strength was significantly affected only by temperature, linear ( $p=0.052$ ) and quadratic ( $p=0.043$ )

terms. Starches extruded at intermediate temperatures formed stronger gels (Fig. 2). This may indicate that some degree of starch degradation is desirable to promote gel formation. However, with greater degradation,

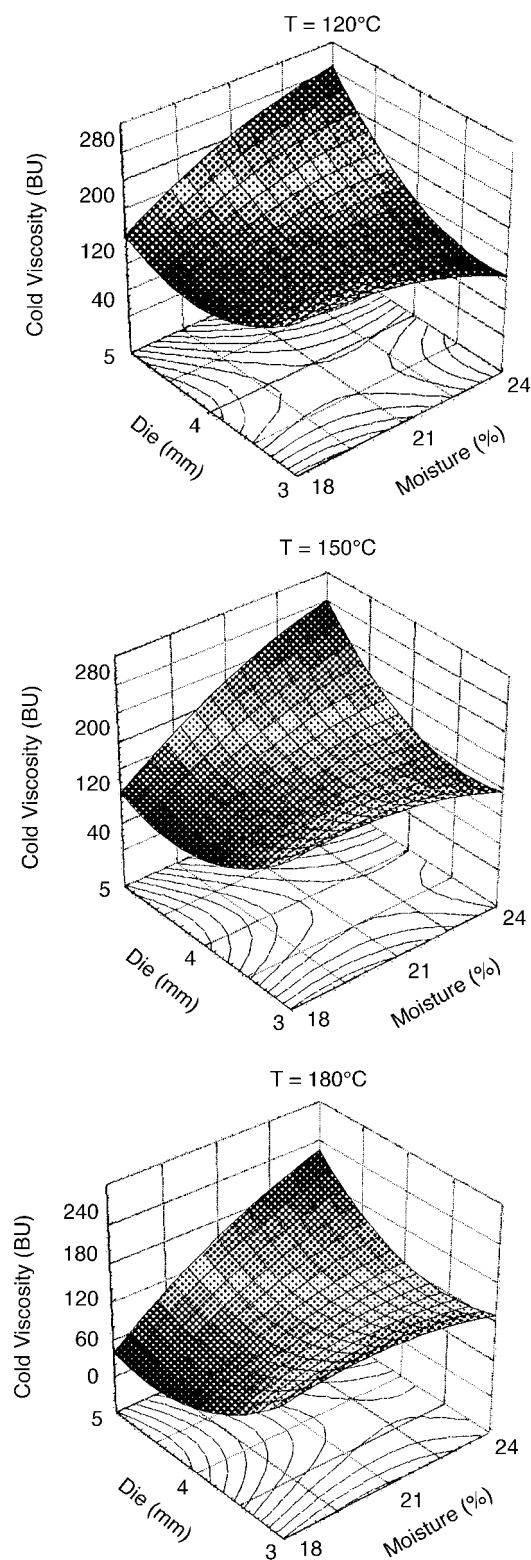


Fig. 1. Effect of feed moisture, extrusion temperature and die diameter on starch cold viscosity, (BU = Brabender Units).

as probably occurs at high temperatures, the gels will present a weaker structure.

Although gel formation has been considered as desirable in specific foods, the tendency of the starch to retrograde is undesirable.

Pastes of non-extruded gelatinized yam starches (8% w/v, d.b.), stored at 10°C for 24 h, presented high retrogradation, with extensive syneresis, higher than that observed in gels of other sources (Fig. 3). Rosenthal (1972) also observed this pattern and explained it by the high linear fraction content of yam starch.

The gels of extruded starches, however, presented low retrogradation, even when stored for two weeks at 10°C. Extrusion probably caused starch degradation, forming smaller chains with lower retrogradation tendency. Regression analysis demonstrates that changes, after 1 week's storage, in the retrogradation response variable, were promoted by moisture (linear term,  $p=0.026$ ),

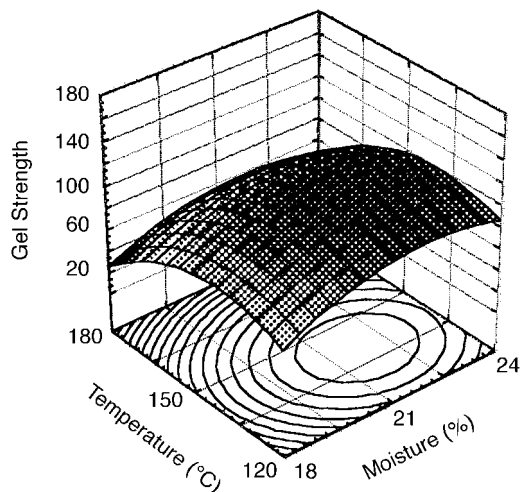


Fig. 2. Gel strength as a function of feed moisture and extrusion temperature.

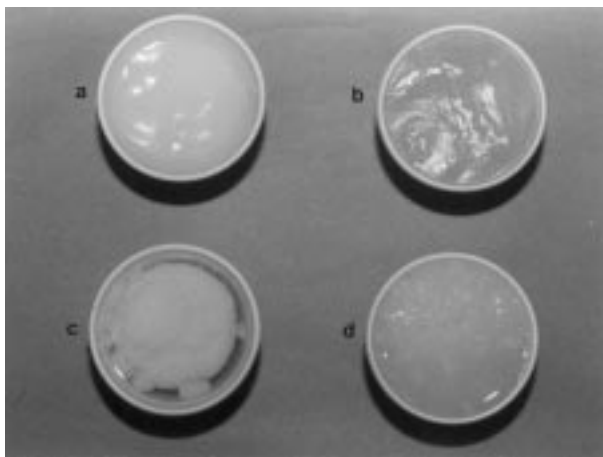


Fig. 3. Appearance of starch gels of different sources stored at 10°C for 24 h: (a) corn, (b) cassava, (c) yam, (d) pregelatinized yam.

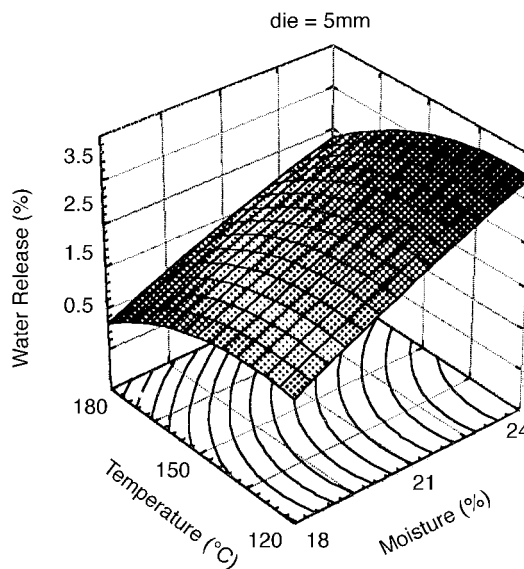
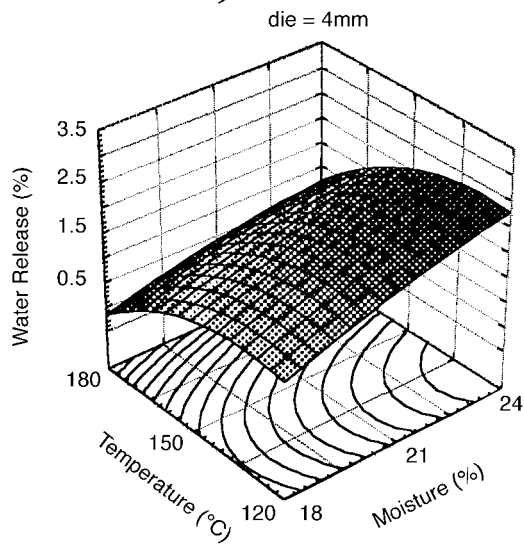
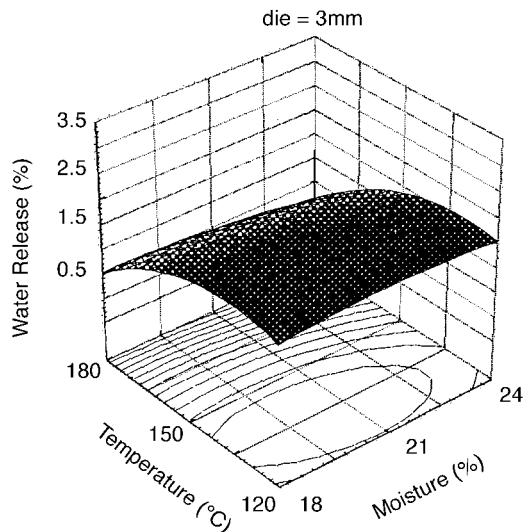


Fig. 4. Effect of feed moisture, extrusion temperature and die diameter on water release of starch gels stored for 7 days at 10°C.

temperature (linear and quadratic effects,  $p=0.010$  and  $p=0.081$ , respectively) and by moisture $\times$ die diameter interaction ( $p=0.044$ ). The model accounts for 88% of the response variation (Table 2).

With a die diameter of 3 mm (Fig. 4), water release was lower in gels prepared with starches extruded with higher moisture. With dies of 4 or 5 mm the effect of moisture was inverted; when the moisture increased the retrogradation also increased. For every die diameter, retrogradation increased with temperature until 150°C, then decreased. Retrogradation was lower in samples processed under harsher conditions: high temperature, low moisture, small die. Under these conditions, starch degradation is more intense (Owusu-Ansah et al., 1983). After 2 weeks of storage the mean water release was approximately 2.3 times higher than after 1 week's storage (data not shown) and the response surface was not the same as that observed in the first week. However, the samples that presented the lowest retrogradation were also those processed under harsher conditions.

#### 4. Conclusions

The decrease of retrogradation tendency of yam starch was observed even when the starch was extruded under brand conditions (low temperature, high moisture and die diameter). Under these conditions yam starch gels also presented high cold thickening capacity and high gel strength, and can be recommended for application in instant creams and puddings.

Prediction equations for the studied functional properties of extruded yam starches can help in selecting processing conditions for specific food applications.

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