

## PHYSICAL CHARACTERISTICS OF EXTRUDED CASSAVA STARCH

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**ABSTRACT:** Considering the importance of cassava starch for Brazilian industries, the current work aimed at evaluating the effects of extrusion parameters on the physical characteristics, mainly viscosity properties of extruded cassava starch. A factorial central composite design ( $2^3$ ) with three independent variables and the response surface methodology were used to evaluate the results of expansion index, specific volume, water absorption index, water solubility index, color and paste properties, according to the variations in the moisture content, barrel temperature and screw speed. Results indicated that barrel temperature influenced the expansion index, specific volume, water absorption index, all the color parameters, the initial viscosity, peak and final viscosity. Feed moisture influenced the specific volume, color parameters, final viscosity and retrogradation. The screw speed had effects on water absorption index, color components as well as on the final viscosity and retrogradation of extruded starch. High moisture, low screw speed and intermediate temperature provided lower starch degradation, which is desirable for pre-cooked starch.

**Key words:** *Manihot esculenta*, modified starch, extrusion, viscosity

## CARACTERÍSTICAS FÍSICAS DE AMIDO DE MANDIOCA EXTRUSADO

**RESUMO:** Considerando a importância do amido de mandioca para as indústrias brasileiras, este trabalho teve por objetivo avaliar os efeitos de parâmetros de extrusão sobre as características físicas e propriedades de pasta do amido de mandioca extrusado. Foi utilizado o delineamento fatorial do tipo central composto rotacional com três variáveis independentes ( $2^3$ ) e a metodologia de superfície de resposta para avaliar os resultados de índice de expansão, volume específico, índice de absorção de água, índice de solubilidade em água, cor e propriedades de pasta, de acordo com as variações de umidade, temperatura de extrusão e rotação da rosca. A temperatura de extrusão influenciou o índice de expansão, volume específico, índice de absorção de água, todos os parâmetros de cor e a viscosidade inicial, pico e viscosidade final. Já a umidade teve influência sobre o volume específico, parâmetros de cor, viscosidade final e tendência a retrogradação. A rotação da rosca teve efeito no índice de absorção de água, nos componentes de cor bem como viscosidade final e na tendência à retrogradação do amido extrusado. Nas condições operacionais de elevada umidade inicial da matéria-prima, baixa rotação da rosca e temperatura de extrusão no nível intermediário ocorreu menor degradação do amido, o que é desejável em amidos pré-gelatinizados.

**Palavras-chave:** *Manihot esculenta*, amido modificado, extrusão, viscosidade

### INTRODUCTION

Cassava is the second most important source of starch in Brazil, constituting an industrial input into strategic sectors of the economy. In 2005, the country produced 26.8 million tons of cassava roots within an area of 1.9 million ha, and the main producers were the states of Pará, Bahia, Paraná, Maranhão and Rio Grande do Sul. In the same year, the production of cassava starch yielded 546.5 thousand tons, and the Paraná State was the greatest producer (IBGE, 2007).

Modified starch is mainly used in Brazil by the paper industry, and smaller amounts are destined to the food and textile sectors. Generally, native starch is not the most appropriate form for specific processing. Therefore it is modified in order to obtain starchy products that have the properties needed for specific uses (Franco et al., 2001).

Starch modifications can be divided into physical, chemical, enzymatic and biological. Gelatinization and the subsequent removal of the starch moisture yield pregelatinized starch, which is soluble in water and

constitutes the basis for previously prepared food (Franco et al., 2001).

Extrusion has gained emphasis and expansion in the food industry since it is an important technique which, besides increasing the variety of processed food, presents more advantages when compared with other traditional systems of food processing. The rapid acceptance of food extrusion as an important food processing operation has resulted because of the many advantages of the use of extrusion. First, extruders achieve high productivity in a single processing step by cooling and forming the product, thus, enhancing the cost effectiveness of the process. The ability of an extruder to handle a wide variety of raw ingredients and processing conditions assures its versatility and consequently broadens its applicability to a myriad of food products (Harper, 1981).

Extrusion alters several of the starch functional properties which mainly depend on the relation amylose-amylopectin as well as on the operational parameters of the process such as the raw-material moisture content, the barrel temperature, the screw speed and geometry (Harper, 1981). Extrusion of starchy food results in gelatinization, partial or complete destruction of the crystalline structure and molecular fragmentation of starch polymers, as well as protein denaturation, and formation of complexes between starch and lipids, and between protein and lipids (Colonna & Mercier, 1983).

Gelatinized starches have numerous industrial, nonfood uses such as in drilling oil wells, sizing textiles, paper making, charcoal briqueting, and in obtaining water-base paints. In food, gelatinized starches can be used almost any time thickening is desired. The gelatinization of starch also significantly affects the characteristics and quality of food such as loaf volume and crumb bread, the elasticity and softness of paste products, digestibility and palatability, the tolerance of batter properties in cakes, frostings, and doughnut mixes, the sugar crystal growth in foods, and the texture, volume, shelf-life, and freeze-thaw stability of bread and cakes (Chiang & Johnson, 1977).

The current study aimed at evaluating the effects of the operational conditions of the extrusion pro-

cess on the physical properties of extruded cassava starch, including viscosity due the importance of this starch in Brazil.

## MATERIAL AND METHODS

A commercial cassava starch was used and moisture, ash, protein, lipids, starch, total sugar and fiber content were characterized according to the methodology of AACC (1995). Extrusion was carried out using a complete line of IMBRA RX (Inbramaq), which has a motor coupled with a speed reducer (extrusion by mechanical friction); a single extrusion screw; a hydraulic cooling system for temperature control; variable speed; and capacity of 45 kg h<sup>-1</sup>.

The extrusion process parameters were:

**Constant parameters:** extrusion temperature in the 1<sup>st</sup> (20°C) and 2<sup>nd</sup> zones (60°C); screw compression ratio (3:1); die diameter (5 mm); feed rate (200 g min<sup>-1</sup>); and cutting speed (90 rpm).

**Variable parameters:** feed moisture; barrel temperature in the 3<sup>rd</sup> zone; and screw speed (Table 1). The ranges of variation between the upper and lower limit of variables were established from preliminary tests.

The 'central composite rotational' design for three factors was adopted, according to Cochran & Cox (1957), including a total of 15 treatments, as follows: eight treatments corresponded to a 2<sup>3</sup> factorial, where the three factors were S = screw speed (rpm); T = barrel temperature (°C); M = feed moisture (%); each one at two levels coded as -1 and +1; six treatments included the minimum and maximum levels of each factor coded as - $\alpha$  and + $\alpha$ , respectively, where  $\alpha = 2^{3/4} = 1.682$ ; one central treatment was repeated six times, in which all factors were at an average level coded as zero (Table 1).

The expansion index (EI) was evaluated after extrusion and before drying. It was calculated using the relation between sample and die diameters (Faubion & Hosney, 1982). The considered value was obtained by the arithmetic mean of the measurements of 20 expanded products in each treatment. Specific volume was determined according to the mass displacement method (millet seed) using a graded pipette (Faubion & Hosney, 1982).

Table 1- Levels of variation and variable parameters of the extrusion process.

Independent variable	Levels of variation				
	-<math>\alpha</math>	-1	0	+1	+<math>\alpha</math>
Screw speed (rpm)	140	190	218	246	266
Extrusion temperature (°C)	40	50	65	80	90
Moisture content (%)	12.5	14	16	18	19.5

<math>\alpha</math> = 1.682

Water absorption index (WAI) and water solubility index (WSI) were assessed before and after treatments (Anderson et al., 1969).

Color of the products was evaluated using a colorimeter *Minolta CR-400*. Results were expressed as  $L^*$ ,  $a^*$  and  $b^*$  values, where  $L^*$  (luminosity or clarity) values varied from black (0) to white (100), chroma  $a^*$  values varied from green (-60) to red (+60), and chroma  $b^*$  values varied from blue (-60) to yellow (+60).

To evaluate the properties of cassava starch paste, non-extruded and after treatments, a Rapid Visco Analyser (RVA), series 4, *Newport Scientific*, was used for starch suspensions (3.5 g starch in 25 mL  $H_2O$ ) corrected to 14% humidity base. One g of ethanol was added to the sample as recommended to *Extrusion 2* method of NEWPORT SCIENTIFIC (1998). The apparent viscosity was expressed in RVU. The following characteristics were evaluated from the obtained graph: pasting temperature, maximum peak viscosity, breakdown viscosity (difference between the maximum and post-peak minimum viscosity), final viscosity, and setback (difference between the final viscosity and the minimum post-peak viscosity).

## RESULTS AND DISCUSSION

The centesimal composition of the cassava starch indicated: 12.2% of moisture, 88.43% of starch, 0.15% of fiber, 0.14% of total sugar, 0.26% of lipids, 0.07% of protein and 0.1% of ash, showing that the product is in agreement with the legislation demands (BRASIL, 1978). Table 2 summarizes the estimated regression

coefficients for the quadratic models fitted to the experimental results and their significance together with their corresponding coefficients of determination.

The EI of cassava starch expanded products ranged from 2.79 to 3.69. There were no effects of extrusion temperature, feed moisture and screw speed. However, the lower expansion index was observed at the maximum level of temperature and central condition of moisture and screw speed. The degree of expansion of extrudate was closely related to the size, number and distribution of air cells surrounded by the cooked material. At very high temperature, however, the vaporization occurs in a violent way and may cause breakage of the structure, hampering expansion (Hashimoto & Grossmann, 2003).

Chang & El-Dash (2003) reported similar results for extruded cassava starch. Moisture (26 - 24%), followed by barrel temperature (120–200°C), affected the expansion at constant speed of 100 rpm. Under low moisture conditions, an increase in barrel temperature led to increased expansion. High feed moisture and high barrel temperature conditions decreased expansion. Expansion is related to the starch gelatinization degree (Chang & El-Dash, 2003). Low moisture content in the material may restrain its flow inside the extruder, increasing shear and residence time, which could increase the gelatinization degree.

The specific volume varied from 3.56 mL  $g^{-1}$  to 7.57 mL  $g^{-1}$ . There was an effect of barrel temperature and feed moisture on the specific volume of expanded products. Under low feed moisture and high barrel temperature conditions, the obtained products presented greater specific volumes (Figure 1).

Table 2- Regression equation coefficients (model  $Y_k = b_0 + \sum_{i=1}^3 b_i x_{ik} + \sum_{i=1}^3 \sum_{j=1}^3 b_{ij} x_{ik} x_{jk} + e_k$ ) (\* =  $p < 0.05$ , \*\* =  $p < 0.01$ , \*\*\* =  $p < 0.001$ ).

Parameters	Response variables											
	EI	SV	WAI	WSI	$L^*$	$a^*$	$b^*$	IV	VP	B	RT	FV
$b_0$	3.54	5.13	5.97	24.44	82.54	1.91	13.88	11.42	11.09	6.39	5.79	10.49
$b_1$	0.101	1.09***	0.28*	2.10	0.24	-0.44*	-0.83	1.15	1.12	0.40	0.17	0.76*
$b_2$	-0.014	-0.54**	0.08	-1.04	1.82**	-0.38*	-1.77**	0.22	0.83	0.53	0.87*	1.16**
$b_3$	0.022	0.20	-0.29*	1.50	-0.54	0.35*	1.14*	0.17	-0.22	-0.65	-1.33**	-1.04**
$b_{11}$	-0.200*	0.08	-0.16	-0.79	-1.88***	0.42*	1.03*	-1.70*	-1.31*	-0.47	0.36	-0.57
$b_{22}$	-0.033	0.26	-0.09	-0.23	-1.64**	0.40*	1.40**	0.10	0.21	0.22	-0.15	-0.09
$b_{33}$	-0.102	0.05	-0.17	-1.86	-0.33	0.09	0.33	0.41	0.43	-0.07	-0.29	0.09
$b_{12}$	0.034	-0.23	-0.27	0.13	-0.45	0.36	0.71	-0.70	-0.53	-1.17	-0.94	-0.29
$b_{13}$	0.034	-0.02	0.12	-0.29	1.25*	-0.03	0.45	0.22	0.61	1.34	0.65	-0.08
$b_{23}$	-0.049	0.03	-0.0	0.92	0.37	-0.26	0.21	0.09	0.24	-0.43	-0.83	-0.17
$R^2$	0.53	0.89	0.71	0.30	0.87	0.81	0.81	0.58	0.61	0.46	0.74	0.80

$b_1$  = extrusion temperature;  $b_2$  = moisture;  $b_3$  = screw speed; EI = expansion index; SV = specific volume; WAI = water absorption index; WSI = water solubility index; IV = viscosity index; VP = Viscosity Peak; B = breakdown; RT = retrogradation tendency; FV = final viscosity;  $L^*$  = luminosity;  $a^*$  = chroma  $a^*$ ;  $b^*$  = chroma  $b^*$ .

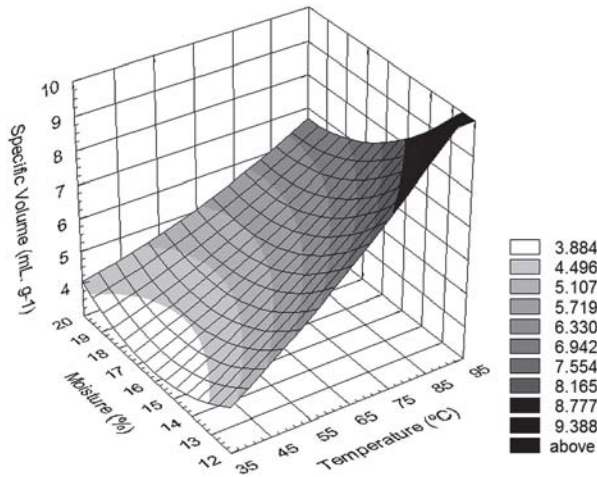


Figure 1- Effect of barrel temperature and feed moisture on the specific volume of extruded cassava starch at 218 rpm screw speed.

The increase of specific volume could be explained by the greater expansion in both axial and radial conditions. The volume expansion phenomena are basically dependent on viscous and elastic properties of melted dough. When temperature increases melt viscosity is reduced and the axial expansion increases (Liang et al., 1994). Alves and Grossmann (2002) also observed the influence of the raw-material moisture and the extrusion temperature on the production of snacks obtained from *Dioscorea alata* flour. For 16% moisture, 170°C and die diameter of 3mm, a greater specific volume (6.32 mL g<sup>-1</sup>), inferior to the mean value for commercial corn snacks (8.72 mL g<sup>-1</sup>), was obtained.

The water absorption index (WAI) measures the volume occupied by the starch after swelling in excess water, and indicates the integrity of starch in aqueous dispersion. Water solubility index (WSI), often used as an indicator of degradation of molecular components, measures the degree of starch conversion during extrusion which is the amount of soluble polysaccharide released from the starch component after extrusion (Yang et al., 2008). The WSI obtained in the different extrusion treatments ranged from 12.3 to 30.74%. Considering the WSI of cassava starch before extrusion (0.69%), there was a pronounced increase in solubility with the process, independent of the tested conditions. Effects of barrel temperature, feed moisture and screw speed were not observed on this parameter.

The WAI of extruded cassava starch varied from 4.19 to 6.41g g<sup>-1</sup>; such values were higher than those observed for non-extruded starch (1.64 g g<sup>-1</sup>). WAI was affected by the extrusion temperature and screw speed. The higher water absorption index was observed at high extrusion temperature and low screw speed conditions (Figure 2). Only gelatinized starch

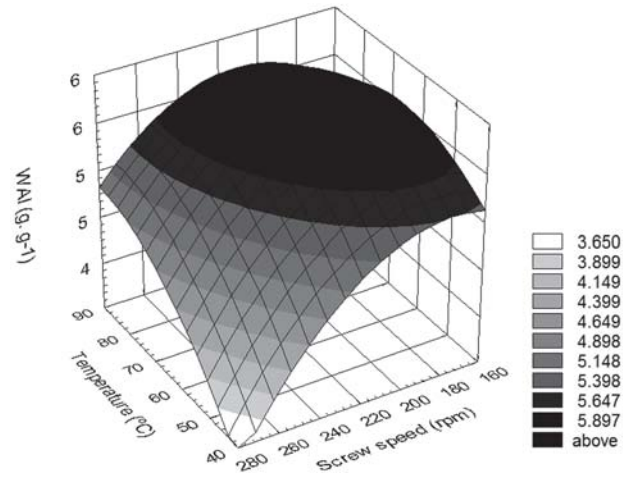


Figure 2 - Effect of barrel temperature and screw speed on water absorption index of extruded cassava starch at 16% of moisture.

granules absorb water at room temperature and swell; however, starch fragmentation increases when the gelatinization degree increases, decreasing thus water absorption. WAI and WSI are important parameters to define the applications of extrudates as ingredients. High WSI is related to the stickiness of extruded products (Hashimoto & Grossmann, 2003).

Color is an important characteristic of extruded foods. Color changes can give information about the extent of browning reactions such as caramelization, Maillard reaction, degree of cooking and pigment degradation that take place during the extrusion process (Altan et al., 2008). Analysis of the starch color components before and after extrusion evidenced that the values of the L\* component from extruded starch varied from 72.08 to 82.98, indicating a decrease in luminosity after extrusion when compared with the initial luminosity of 92.21. Out of the parameters that constitute the regression model, barrel temperature and feed moisture affected luminosity. The interaction between barrel temperature and screw speed also had a significant effect.

Under high feed moisture and high barrel temperature conditions, there was less darkening of the extruded starch, i.e. greater L\* (Figure 3). These results were different from those observed by Gutkoski & El-Dash (1999), who studied extruded oat products with moisture content varying from 17 to 24%, barrel temperature from 90°C to 150°C, and constant speed of 100rpm, and verified that the luminosity decreased linearly when barrel temperature increased.

Chroma a\* (redness) values ranged from 1.52 to 5.26, which indicate that there was little variability in this parameter during the extrusion process, considering it can vary from -60 to +60. Analysis of the regression coefficients showed effect of all three fac-

tors (barrel temperature, feed moisture and screw speed) on the chromaticity component  $a^*$ . According to the model, under low feed moisture and low barrel temperature conditions and screw speed of 218rpm, higher chroma  $a^*$  values were observed (Figure 4). Its highest values occurred at constant temperature (65°C), high screw speed and low feed moisture (Figure 5).

Responses of the  $b^*$  color parameter, which represents variation from blue to yellow, varied according to the treatment (from 11.35 to 20.71). All samples, however, had yellow color. Regression analysis indicated effects of moisture, screw speed and temperature on this chromaticity component: under high feed moisture, low screw speed and intermediate barrel temperature conditions, chroma  $b^*$  values were lower (Figures 6 and 7). The increase in barrel temperature increases color intensity, and high moisture conditions

result in brighter products since increase in moisture decreases the residence time, providing less non-enzymatic darkening of extruded products (Badrie & Mellows, 1991).

The initial viscosity at 25°C was too close to zero (as expected) due to the absence of gelatinized starch. The curve showed an acute peak (characteristic of granules presenting structural homogeneity) followed by a pronounced fall, even before reaching 95°C, which revealed little stability of the hot paste during agitation. The viscosity peak (VP) of cassava starch was 439.67 RVU, breakdown (B) was 319.75 RVU, final viscosity (FV) was 217.83 RVU, retrogradation tendency (R) was 97.92 RVU, and paste temperature was 67.20°C (Figure 8). The initial viscosity or cold viscosity indicates the capacity of the starch to absorb water at room temperature and to form paste, gel or viscous liquid. Cold viscosity values obtained for ex-

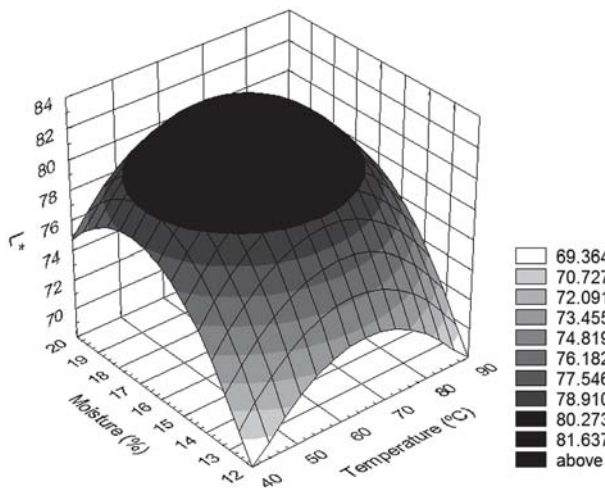


Figure 3 - Effect of barrel temperature and feed moisture on the luminosity ( $L^*$ ) of extruded cassava starch at 218 rpm screw speed.

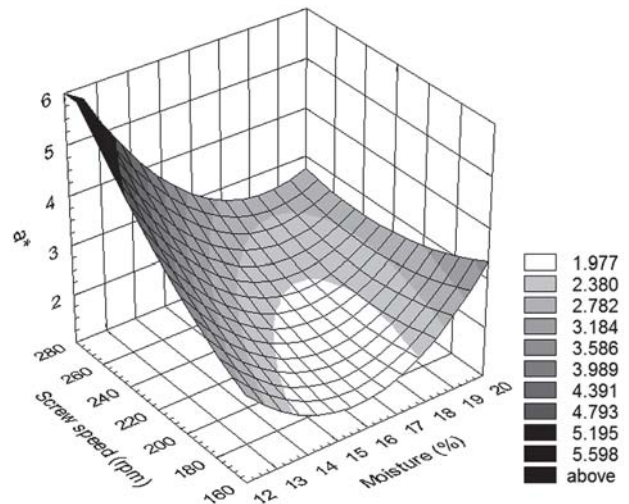


Figure 5 - Effect of feed moisture and screw speed on chroma  $a^*$  of extruded cassava starch at 65°C barrel temperature.

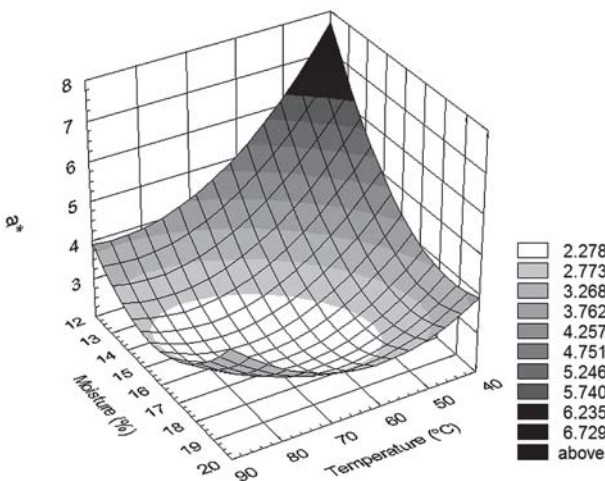


Figure 4 - Effect of feed moisture and barrel temperature on chroma  $a^*$  of extruded cassava starch at 218 rpm screw speed.

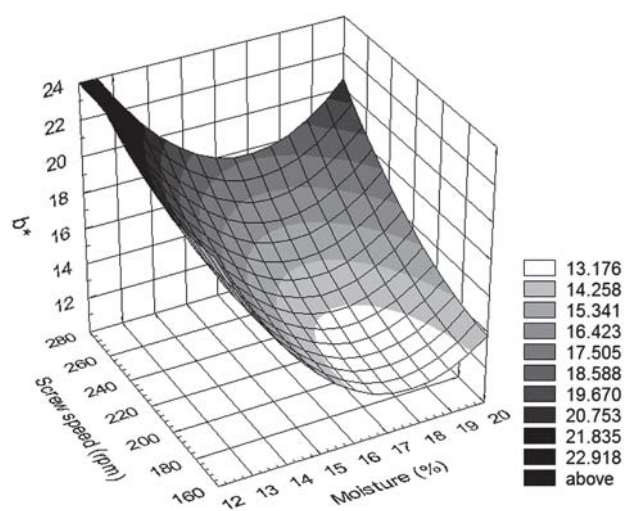


Figure 6 - Effect of feed moisture and screw speed on chroma  $b^*$  of extruded cassava starch at 65°C barrel temperature.

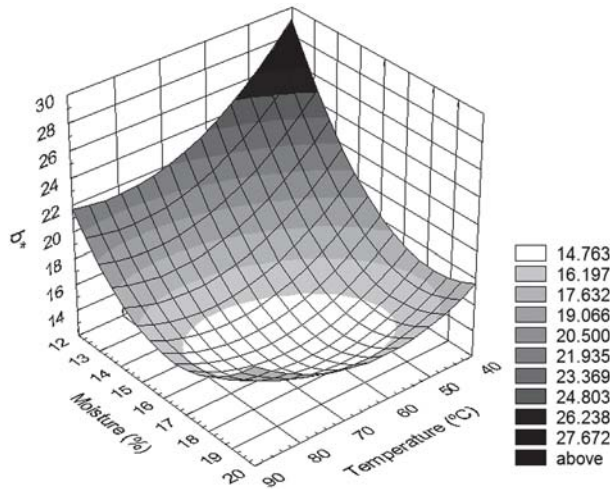


Figure 7 - Effect of feed moisture and barrel temperature on chroma b\* of extruded cassava starch at 218 rpm screw speed.

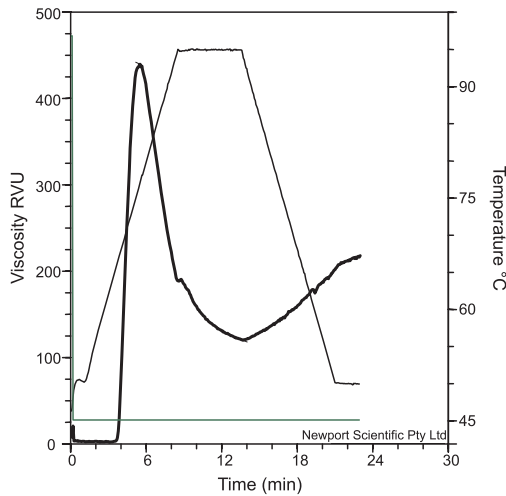


Figure 8 - Viscoamulography profile of cassava starch.

truded starch varied from 5.3 to 14.7 RVU. Considering that the initial viscosity before extrusion was practically zero, the extrusion treatments led to a considerable increase in cold viscosity. Out of the factors constituting the model, barrel temperature affected viscosity, and no interference of feed moisture and screw speed were observed. The increase in extrusion temperature led to an increase of the initial viscosity and, above 70°C, there was a tendency towards fall, indicating degradation of the crystalline structure (Figure 9).

The viscosity peak increased as barrel temperature increased and decreased at the highest temperatures (Figure 10). However, the values obtained (6.33 to 14.58 RVU) were notably below those observed before extrusion, indicating that the processing conditions provided high degradation of the starchy structure. Chiang & Johnson (1977) evaluated the effect of gelatinization on wheat flour as a function of screw speed, operational temperature and water content, and

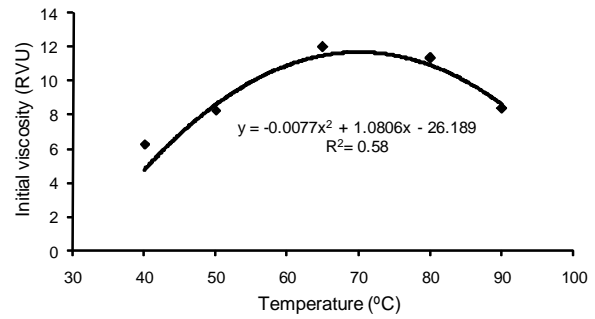


Figure 9 - Effect of barrel temperature on the initial viscosity of extruded cassava starch at 16% feed moisture and 218 rpm screw speed.

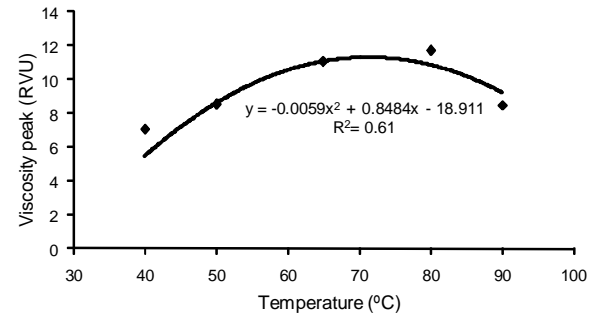


Figure 10 - Effect of barrel temperature on viscosity peak of extruded cassava starch at 16% feed moisture and 218 rpm screw speed.

noticed that as temperature increased, gelatinization increased. They considered extrusion temperature and initial moisture content of the raw material as the variables that had more pronounced effects on starch gelatinization; they also verified that the maximum gelatinization degree occurred when these variables acted with extreme opposite values.

The breakdown of the extruded products varied from 2.83 to 12.83 RVU. Analysis of the regression coefficients showed there was not effect of the factors that constitute the model. However, the highest breakdown value was observed under low barrel temperature, low speed and high feed moisture conditions. Low breakdown values occurred because degradation of the starchy fraction did not lead to significant viscosity peaks. The high moisture content in the feed might act as a lubricant and reduce the melt viscosity during extrusion leading to high breakdown viscosity values.

The final viscosity (FV) of extruded starch varied from 7.25 to 13.1 RVU. Analysis of the regression coefficients showed effect of barrel temperature, feed moisture and screw speed. Under low moisture conditions and high screw speed, FV were lower (Figure 11). Low moisture in the feed possibly increased frictional damage, particularly when the residence time was lower due to high screw speed. The highest val-

ues occurred under the highest barrel temperature and feed moisture conditions (Figure 12).

Bhattacharya et al. (1999) evaluated the effect of the initial moisture content of the samples of mixtures of wheat flour and potato on the behavior of paste extruded products using Rapid Visco Analyser (RVA) and did not observe an accentuated increase in apparent viscosity during cooling, which indicated degradation of the starchy fraction during extrusion, resulting thus in an increase in FV at 50°C. Similarly to the present research, the highest values of FV found by those authors occurred under high moisture conditions.

The retrogradation tendency values of the extruded products varied from 2.50 to 12.50 RVU. These low values indicated the severity of the treatments, causing degradation of polymers, rupture of their molecular structures, and reduction of their recrystallization

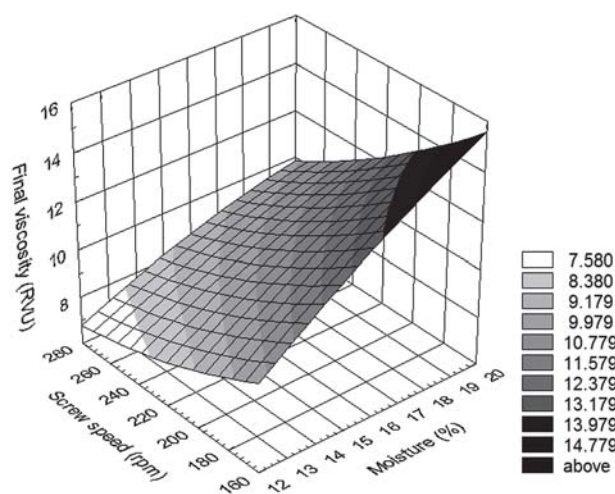


Figure 11 - Effect of feed moisture and screw speed on the final viscosity of extruded cassava starch at 65°C barrel temperature.

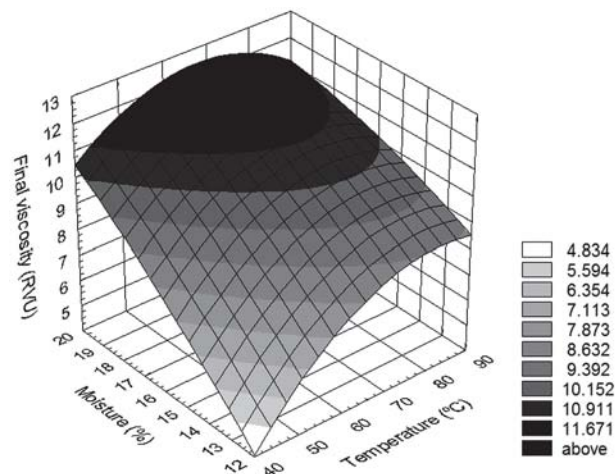


Figure 12 - Effect of barrel temperature and feed moisture on the final viscosity of extruded cassava starch at 218 rpm screw speed.

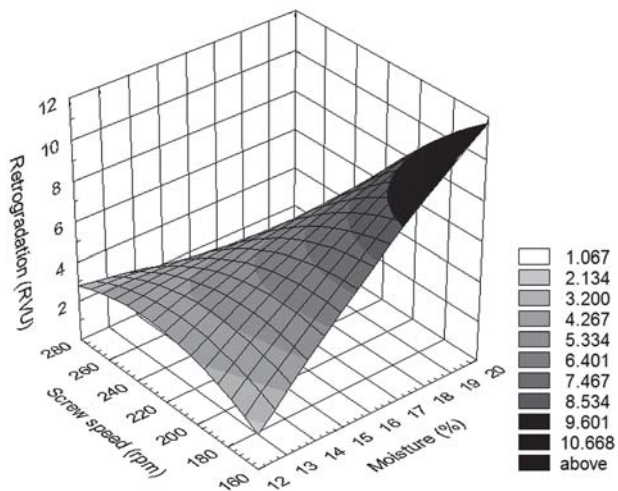


Figure 13 - Effect of feed moisture and screw speed on the retrogradation tendency of extruded cassava starch at 65°C barrel temperature.

capacity. The adopted regression model was significant, and there was effect of feed moisture and screw speed on retrogradation. The highest retrogradation values were observed under high moisture and low screw speed conditions (Figure 13).

## CONCLUSIONS

The operational conditions of the extrusion process affect paste properties. Cassava starch show small quantity of non-starchy components and its solubility and water absorption indexes were characteristic of non-gelatinized natural starch.

Extruded starch had less luminosity, higher chroma  $a^*$  level, and greater intensity of chroma  $b^*$  than native starch.

There is a pronounced increase of the water solubility and water absorption indexes with the extrusion process. High moisture, low screw speed and intermediate temperature provide lower starch degradation what is desirable in pre-cooked starch.

Out of the extrusion parameters, barrel temperature has the most pronounced effect on expansion, paste properties and color of extruded starch, followed by feed moisture and screw speed.

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