Available online at www.sciencedirect.com
SCIENCE DIRECT
Carbohydrate Polymers 60 (2005) 1–6
Carbohydrate Polymers
www.elsevier.com/locate/carbpol

Effects of annealing on the physicochemical properties of fermented cassava starch (*polvilho azedo*)

Anida M.M. Gomes[a], Claudio E. Mendes da Silva[a], Nágila M.P.S. Ricardo[b,*]

[a]*Department of Food Technology, Federal University of Ceará, P.O. Box 12168, Fortaleza, Brazil*
[b]*Department of Inorganic Chemistry, Federal University of Ceará, P.O. Box 12200, Fortaleza, Brazil*

Received 13 December 2003; revised 19 October 2004; accepted 9 November 2004

Abstract

The fermented cassava starch (*polvilho azedo*) in 1:5 starch to water ratio (w/v), was subject to annealing treatment at 50 °C for 72, 96, 144 and 240 h. The annealing treatment changed the internal structure of *polvilho azedo* when the time was increased. Peak viscosities decreased significantly, denoting that there was a decreasing in leaching of amylose from the granules. The pasting temperature was increased, while hold, final viscosities, and breakdown were reduced showing an enlargement on the stability of the paste. The swelling power and the solubilities underwent reductions in all temperatures. The DSC data showed that there was an increased on T_o, T_p, T_c and ΔH in all samples annealed. The X-ray diffraction pattern did not change but crystallinity increased (all samples annealed), denoting increase in organization of double helical of amylopectin. The *polvilho azedo* samples submitted to annealing treatment acquired some characteristics of cereal starches (waxy starches).

Keywords: Annealing of starch; Relative crystallinity; Fermented cassava starch

1. Introduction

The fermented cassava starch (*polvilho azedo*) is a product obtained from the natural fermentation of freshly extracted cassava starch (*polvilho doce*), for 30–40 days (Cereda, 1987). After fermentation process the starch is submitted to a drying, which is realized naturally at sun and wind, which turns the process totally dependent to climatic conditions. The different periods of fermentation provoke variations on the acid concentration which turns the characteristics of *polvilho azedo* so different. The main objectives of fermentation is to increase the nutritional value, to decrease toxicity and impart to product properties and flavour characteristics (Nakamura & Park, 1975). The fermentative process change the characteristics of *polvilho azedo*, decreasing the pasting temperature and peak viscosity (Cereda, 1983) in relation to *polvilho doce*.

The annealing is a treatment realized in water excess for an extended period, below the gelatinization temperature (T_o) and above the glass transition temperature of starches. Gough and Pybus (1971), after submitting wheat starch to maceration in distillated water at 50 °C for 72 h found that gelatinization temperatures were increased and gelatinization range was narrowed. From this study, several other works utilizing different variety of starch, temperatures and treatment time, has been realized.

This treatment provokes a reorganization of starch molecules, leading to amylopectin double helice to acquire one more organized configuration (Hoover & Vasanthan, 1994; Krueger, Knutson, Inglett, & Walker, 1987; Krueger, Walker, Knutson, & Inglett, 1987). The increase in organization provokes decrease on swelling power and solubilization of starches (Eerlinger, Jacobs, Block, & Delcour, 1997; Hoover & Vasanthan, 1994; Jacobs et al., 1998; Lorenz & Kulp, 1982; Tester, Debon, & Sommerville, 2000), increase on gelatinization temperatures and enthalpy and narrowing of gelatinization range (Gough & Pybus, 1971; Jacobs et al., 1998; Krueger, Knutson, et al., 1987;

* Corresponding author. Tel.: +55 85 288 9436; fax: +55 85 288 9978.
E-mail address: naricard@ufc.br (N.M.P.S. Ricardo).

0144-8617/$ - see front matter © 2004 Elsevier Ltd. All rights reserved.
doi:10.1016/j.carbpol.2004.11.016

Krueger, Walker, et al., 1987; Sekine, Otobe, Sugiyama, & Kawamura, 2000; Stute, 1992; Tester et al., 2000), increase on enzymatic susceptibility (Wang, Powel, & Oates, 1997), reductions on peak viscosity, increase on stability of paste, decrease in retrogradation trend (Jacobs, Eerlingen, Clauwaert, & Delcour, 1995; Jacobs, Eerlingen, & Delcour, 1996; Kuge & Kitamura, 1985; Stute, 1992), increase on crystallinity (Jacobs et al., 1995; Larsson & Eliasson, 1991; Paredes-López & Hernández-López, 1991; Stute, 1992).

The heat-moisture treatment is very similar to annealing, because modify the physicochemical properties of starch, without destroying granule structure, but is realized in totally different conditions (normally 16 h at 100 °C). Some authors (Kawabata et al., 1994; Lorenz & Kulp, 1982; Sair, 1967) stated that potato starch changed your X-ray pattern B to A pattern when submitted to heat-moisture. The annealing treatment change the bond forces between crystals and amorphous matrix, and according to many authors does not modify the X-ray pattern.

Mendes da Silva, Sasaki, Freitas e Silva, Jorge and Romeu (1997), through interpretation of crystallinity patterns (integrated intensity) of fermented cassava starch (*polvilho azedo*) submitted to annealing treatment at 50 °C, for several times (0, 48, 96, 120, 144 and 168 h), showed that there was indication of increase on intensity in fermented cassava starch, which can characterize increase on crystallinity.

The hydrothermal treatments such as heat-moisture and annealing, can impart to starches some important characteristics, such as waxy feature. The waxy starches when submitted to gelatinization do not retrograde during the cooling and hence are more stable during storage. This behavior is very useful to food industry, because formulations that contain these starches do not lose their characteristics and do not harden by the storage time, maintaining their texture always soft and consequently increasing their shelf life.

The objectives of this work was to study the changes on properties of *polvilho azedo* caused by annealing treatment and to verify whether a tuber starch when submitted to annealing treatment acquires some similar properties of the cereal starches (waxy starches).

2. Materials and methods

2.1. Annealing treatment

The commercial fermented cassava starch (*polvilho azedo*) samples were submitted to steeping at 50 °C in 1:5 (w/v) starch to water ratio for 72, 96, 144 and 240 h. After the incubation period the samples were filtered and dried in air circulation oven.

2.2. Swelling power and solubility

These parameters were determined by Schoch's methodology (Schoch, 1964). About 1 g of *polvilho azedo* and 40 ml of water was subject to heating at temperatures of 55, 65, 75, 85 and 95 °C. After cooling the starch slurry was centrifuged at $5000 \times g$ for 30 min. The supernatant was separated and 10 ml was carefully withdrawn to calculate the soluble content. The sediment was weighed to determinate the swelling power as a ratio of weight of swollen starch granules to the weight of dry starch. Results used for calculations were means of triplicate measurements.

2.3. Pasting characteristics

A Rapid Visco Analyzer (RVA), model RVA-4 (Newport Scientific Pty. Ltd, Australia) was used for the study of the pasting properties. About 3.5 g of *polvilho azedo* (moisture content 14%) were mixed with distilled water to make a total weight of 28 g in the RVA canister. A programmed heating and cooling cycle was used at constant shear rate. The samples were run in triplicate. In each analysis the samples were held for 1 min at 50 °C and temperature was progressively increased up to 95 °C (13 °C/min), and held at 95 °C for 3 min. Afterwards the temperature was lowered for 50 °C (13 °C/min), and fixed in this temperature for 3 min. Test was finished after 13 min.

2.4. Differential scanning calorimetry (DSC)

DSC analysis was performed with a Shimadzu DSC-50. Approximately 3 mg of *polvilho azedo* was placed into an aluminum sample pan and 15 μl of distilled water was added. After 1 h of equilibration, pans were hermetically sealed. The samples were heated at 25–100 °C (10 °C/min). An indium standard was used to calibrate the instrument. The enthalpy (ΔH), onset (T_o), peak (T_p), and conclusion temperatures (T_c) were computed automatically.

2.5. X-ray diffractometry

The X-ray diffractograms were obtained with a Rigaku Dmax-B X-ray diffractometer, using a Cu Kα radiation detector ($\lambda = 0.1542$ nm) with a scintillation counter operating at 40 kV and 25 mA. The starch powder was scanned through the 2θ angle range of 3–40 °C and scanning rate of 1/2 °C/min.

2.5.1. Relative crystallinity calculations

The relative crystallinity (RC) was determinate according to Herman's methods, as described in Fujita, Yamamoto, Sugimoto, Morita, and Yamamori (1998), using a peak-fitting software (Origin-version 6.0, Microcal, Inc., Northampton, MA, USA). The amorphous and

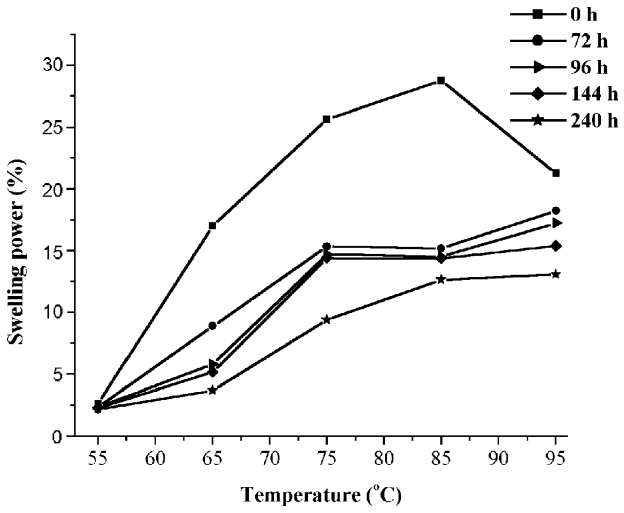

Fig. 1. Effects of annealing time on the swelling power of *polvilho azedo* samples annealed for 0, 72, 96, 144 and 240 h.

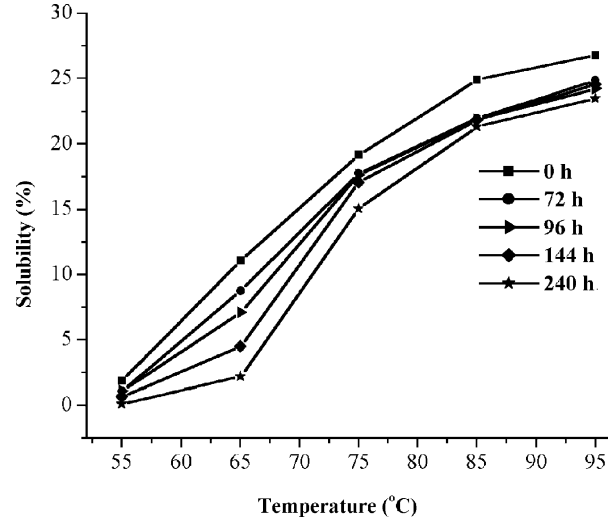

Fig. 2. Effects of annealing time on the solubility of *polvilho azedo* samples annealed for 0, 72, 96, 144 and 240 h.

crystalline regions on diffractograms, were separated by a baseline with 27 points drawn from 3 to 40° (2θ) and the areas were integrated to obtain the amorphous (A_a) and crystalline (A_c) areas by equation $RC = A_c/(A_a + A_c)$.

3. Results and discussion

3.1. Swelling power and solubility

The swelling power of the samples annealed for 240 h at 55, 85 and 95 °C were restricted in 17.1, 56.1 and 38.5% (w/w), respectively, when compared to the control sample (Fig. 1).

The *polvilho azedo* already is a hydrolyzed starch by the fermentation process, whose bonds became weaker, which facilitated the water penetration in the amorphous zones and leaching of amylose from the granules during the heating process, mainly close to its gelatinization temperature.

The solubility of the sample annealed for 240 h at 55 °C suffered restriction of 94.1% (w/w), indicating that there was a strengthening of the bonds (Fig. 2). The solubility is a consequence of the leaching of amylose. The decrease in solubility as the time of annealing was increased, indicate an increase in the interactions between amylose and amylopectin molecules or between amylopectin molecules (forming a more stable structure), impeding them to leaching out from the granules.

3.2. Pasting characteristics

The annealing treatment reduced the peak viscosities of the *polvilho azedo* samples when the time of annealing was increased These reductions were more significant on samples annealed for 240 h (29.3%) and 144 h (29.6%) in relation to control sample (Table 1). The annealing treatment reinforced the connections among the molecules of starch taking them to assume a more stable conformation, leading to amylose having a smaller tendency to leaching out from the granules.

All the *polvilho azedo* samples suffered increase in the paste temperatures, when the time of annealing was increased, indicating that there was a strengthening of the bonds, being necessary, larger temperatures for the gelatinization of the granules.

In the breakdown there was a decrease of 26.4 and 26.7% in the samples annealed for 240 and 144 h when compared to control sample (Fig. 3). These samples still had a reduction in the hold of 13.7 RVU (55.5%) and 14.0 RVU

Table 1
Pasting characteristics of *polvilho azedo* samples submitted to annealing treatment[a]

Time (h)	Peak viscosity (RVU[b])	Breakdown[c] (RVU)	Hold (RVU)	Setback[c] (RVU)	Final viscosity (RVU)	Pasting temperature (°C)
0	251.2±0.2	226.5±1.7	24.7±1.9	13.7±0.3	38.2±2.0	69.2±0.5
72	241.8±4.0	225.1±1.6	16.7±2.5	14.7±0.1	31.4±2.4	74.3±0.1
96	236.0±2.0	220.2±0.5	15. 8±2.4	15.3±0.6	31.1±3.0	74.9±0.5
144	176.9±1.1	166.2±0.6	10.7±0.5	11.4±0.1	22.1±0.5	75.0±0.1
240	177.7±0.7	166.7±0.1	11.0±0.8	12.4±0.3	23.4±0.5	75.9±0.1

[a] Mean of three determinations ± standard deviation.
[b] RVU = rapid visco-analyzer units.
[c] Breakdown = peak visc. − hold; setback = final visc. − hold.

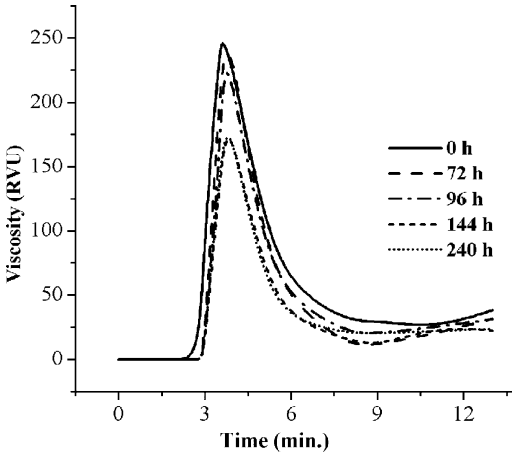

Fig. 3. RVA-curves of *polvilho azedo* samples submitted to annealing treatment for 0, 72, 96, 144 and 240 h.

(56.7%), respectively. This behavior indicates that the *polvilho azedo* possesses a low stability on shear and heating. The setback of the samples practically was not changed, because the native *polvilho azedo* already possesses a low tendency to the retrogradation when compared to other starches. The low amylose content (18.8%) of high degree of polymerization (DP = 1000–6000; which can provoke a repulsive effect among the molecules, impeding the association among them) and the low content of lipids (which decreases the formation of amylose complexes) are factors that, probably, contribute to the low tendency to the retrogradation of the cassava starches.

3.3. Gelatinization temperature

The results of DSC analyses of the *polvilho azedo* samples are shown in Table 2. The gelatinization temperatures of all samples were increased by the time of annealing treatment. The onset temperature (T_o) was progressively increased, while conclusion temperature (T_c) was almost unchanged for 72 h. The gelatinization temperature range of majority of the annealed samples suffered a very small narrowing, which turns the peaks of the thermograms narrowest in relation to the native *polvilho azedo* (Fig. 4).

The enthalpy (ΔH) of the samples annealed for 72, 144 and 240 h, was increased by 275.8, 127.3 and 154.5%, in relation to the control sample when the time of treatment was increased. The possible causes of this behaviour were either the lack of heating uniformity during the annealing process or the changes are more intensive in the beginning of the time of annealing, decreasing when time is increased. The increase in the enthalpy during the gelatinization of the starch is related to the melting of crystalline zone. The more crystalline the sample, the larger is the amount of energy that will be spent to melt those crystals. While the increase in the gelatinization temperature of the samples annealed, indicates more perfect crystals.

Table 2
Effects of annealing time on gelatinization temperatures and ΔH of *polvilho azedo* samples

Time (h)	T_o (°C)	T_p (°C)	T_c (°C)	ΔT ($T_c - T_o$)	ΔH (J/g)
0	61.7	69.4	71.9	10.2	3.3
72	68.4	73.2	79.8	11.4	12.4
96	68.7	73.3	78.8	10.1	7.2
144	69.1	73.9	79.2	10.1	7.5
240	70.0	74.7	80.1	10.1	8.4

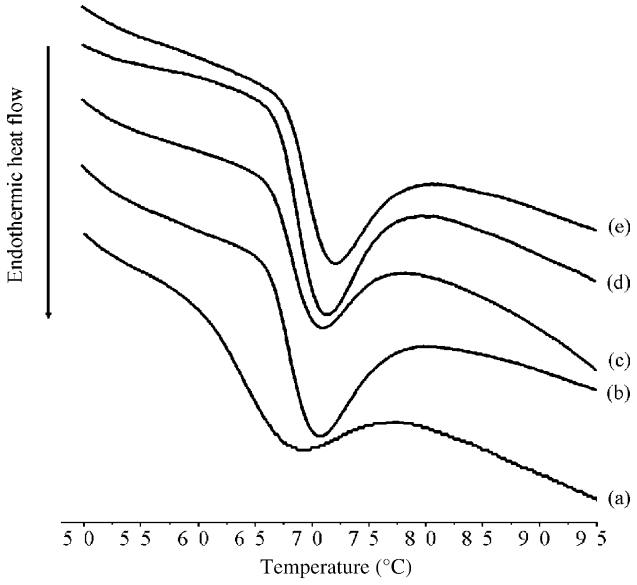

Fig. 4. Effects of annealing time on DSC curves of *polvilho azedo* samples. (a) 0, (b) 72, (c) 96, (d) 144 and (e) 240 h.

The increase in ΔH cannot be caused by the formation of complex amylose-lipids, as defended by Hoover and Vasanthan (1994), because the cassava starch possesses low lipids content and the minimum amount present already meets complexing. It cannot also be caused by the increase in the amount of double helices, because according to Tester, Debon, and Karkalas (1998) that quantified the number of double helices before and after annealing using RMN 13C-CP/MAS, the number of double helices stays constant after the annealing, but with an improved organization. As the number of double helices stays constant after the annealing, it is probable that increases in ΔH is due to an increase in the perfection of the crystals. This indicates that the crystals suffered reorganization and probably, an increase in its dimensions, assuming a condition of 'more perfect'.

3.4. X-ray patterns and crystallinity

The native *polvilho azedo* sample presented a X-ray pattern type A, with the main peaks in 15° (3b), 17° (4a), 18° (4b), 20° (5a), 23° (6a), that correspond to the spacing-*d* in

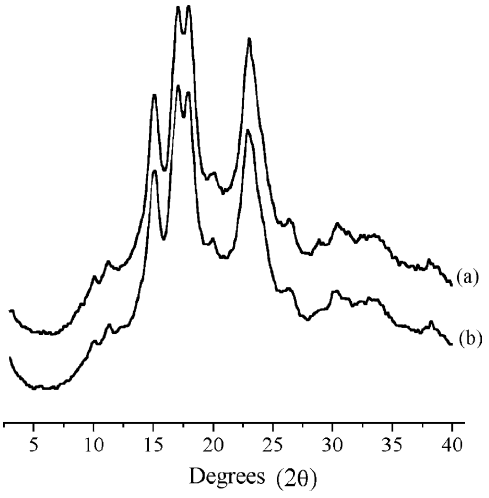

Fig. 5. Diffractograms of native (a) and annealed (b) *polvilho azedo* samples.

5.8, 5.2, 4.8, 4.4 and 3.8 Å, respectively (Fig. 5). However was not observed uniformity in the X-ray patterns among the cultivars of cassava starch, cited by the literature. Moorthy (1985), Franco, Ciacco, and Tavares (1988), Gallant et al. (1982), Rosenthal, Nakamura, Spindola, and Jochimek (1974), and Plata-Oviedo (1991), found patterns which varied from the A, B, C, C_A up to C_B type.

The diffractograms of the *polvilho azedo* samples showed narrow peaks, indicating that the hydrolysis during the fermentation attacked the amorphous areas, leaving intact the crystalline areas. There were no modifications in X-ray patterns of the sample annealed, but the main peaks of the diffractograms of the samples 72 h (4a, 4b and 6a), 96 h (4b and 6a), 144 h (6a) and 240 h (4a, 4b and 6a) presented increases in the diffraction intensities (Table 3). The sample annealed for 72 h presented increase on intensity in almost all the rings, except in 3b and 5a.

The relative crystallinities of the native and annealed samples are presented in Table 4. All samples annealed showed increases in the RC in relation to control sample. The main increases were in the samples annealed for 72 h (7.8%) and 240 h (3.8%). That behavior, showed a good correlation between increase in RC and thermal parameters (T_o and ΔH), indicating that the crystalline structures acquired a more compact packing, which is similar to the presented by some waxy starches (wheat and corn), in relation to the native starches.

Table 3
Effects of annealing time on the intensities of X-ray diffraction and intercrystalline spacings (between brackets) of the *polvilho azedo* samples on principal peaks of diffraction

Time (h)	Intensities[a] with interplanar spacings (d) in Å				
	3b (14°)	4a (15°)	4b (17°)	5a (20°)	6a (23°)
0	2707 (5.8)	3417 (5.2)	3359 (4.9)	2074 (4.5)	3018 (3.9)
72	2693 (5.8)	3429 (5.2)	3449 (4.9)	2072 (4.4)	3241 (3.9)
96	2682 (5.9)	3384 (5.2)	3362 (4.9)	1986 (4.5)	3040 (3.9)
144	2599 (5.9)	3348 (5.2)	3320 (4.9)	1971 (4.4)	3061 (3.8)
240	2666 (5.9)	3448 (5.2)	3402 (4.9)	1985 (4.5)	3032 (3.9)

[a] Counts per seconds (cps).

Table 4
Effects of annealing time on the relative crystallinity (RC) of the *polvilho azedo* samples

Time (h)	RC (%)
0	17.9
72	19.3
96	18.5
144	18.5
240	18.6

In despite of the waxy starches present some similar characteristics to the starches submitted to the annealing and heat-moisture treatment, they do not possess spacing in 4.4 Å, which is characteristic in the formation of amylose–lipids complex. All the samples annealed, presented reductions in diffraction intensity in the ring 5a (4.4 Å), which disagrees to the statements of Hoover and Vasanthan (1994) and confirms that the increase of crystallinity could not be caused by the formation of those complexes.

4. Conclusions

Annealing time affected the physicochemical properties of the *polvilho azedo* samples, because the bonds are already weaken by fermentation process and therefore, are more prone to the reorganization.

The increase in pasting temperatures and decrease in peak viscosities, swelling power and solubilities of the annealed *polvilho azedo* samples as the time of treatment was increased, were caused by the rearrangement in the structure (increase on order) of the amylopectin and amylose molecules.

There were no modifications in the X-ray patterns of the *polvilho azedo* samples, but the same annealed samples which presented increase in the gelatinization temperatures and ΔH showed increase in diffraction intensities and relative crystallinity, which indicates that was a increase in the order of the helical packing.

The modifications provoked by annealing on the physicochemicals properties of the *polvilho azedo* samples made them to acquire some characteristics common to the waxy starches, such as smaller peak viscosity and larger ΔH, crystallinity and gelatinization temperatures.

Acknowledgements

Many thanks to CAPES and CNPQ for financial support that allowed the development of this research, Cereals Laboratory of DEEA-UFC, X-ray Laboratory of DEFIS-UFC for X-ray analysis, Thermoanalysis Laboratory of DEQUI-UFC for DSC analysis and CNPAT/EMBRAPA-RJ for RVA analysis.

References

Cereda, M. P. (1983). Determinação da viscosidade em Fécula fermentada de mandioca (polvilho azedo). *Boletim da SBCTA*, *17*(1), 15–24.

Cereda, M. P. (1987). Tecnologia e qualidade do polvilho azedo. *Informe Agropecuário*, *13*(145), 63–68.

Eerlinger, R. C., Jacobs, H., Block, K., & Delcour, J. A. (1997). Effects of hydrothermal treatments on the rheological properties of potato starch. *Carbohydrate Research*, *297*, 347–356.

Franco, C. M. L., Ciacco, C. F., & Tavares, D. Q. (1988). Studies on the susceptibility of granular cassava and corn starches to enzymatic attack. Part II. Study of the granular structure of starch. *Starch/Stärke*, *40*(1), 29–32.

Fujita, S., Yamamoto, H., Sugimoto, Y., Morita, N., & Yamamori, M. (1998). Thermal and crystalline properties of waxy wheat (*Triticum aestivum* L.) starch. *Journal of Cereal Science*, *27*, 1–5.

Gallant, D. J., Bewa, H., Buy, Q. M., Bouchet, B., Szylit, O., & Sealy, L. (1982). On the ultrastructural and nutritional aspects of some tropical tuber starches. *Starch/Stärke*, *30*(8), 255–262.

Gough, B. M., & Pybus, J. N. (1971). Effect on the gelatinization temperature of wheat starch granules of prolonged treatment with water at 50 °C. *Die Stärke*, *23*, 210–212.

Hoover, R., & Vasanthan, T. J. (1994). The effect of annealing on the physicochemical properties of wheat, oat, potato and lentil starches. *Food Biochemistry*, *17*, 303–325.

Jacobs, H., Eerlingen, R. C., Clauwaert, W., & Delcour, J. A. (1995). Influence of annealing on the pasting properties of starches from varying botanical sources. *Cereal Chemistry*, *32*, 480–487.

Jacobs, H., Eerlingen, R. C., & Delcour, J. A. (1996). Factors affecting the visco-amylograph and rapid visco-analyzer: Evaluation of the impact of annealing on starch pasting properties. *Starch/Stärke*, *48*(7/8), 266–270.

Jacobs, H., Mischenko, N., Koch, M. H. J., Eerlingen, R. C., Delcour, J. A., & Reynaers, H. (1998). Evaluation of the impact of annealing on gelatinisation at intermediate water content of wheat and potato starches: A differential scanning calorimetry and small-angle X-ray scattering study. *Carbohydrate Research*, *306*, 1–10.

Kawabata, A., Takase, N., Miyoshi, E., Sawayama, S., Kimura, T., & Kudo, K. (1994). Microscopic observation and X-ray diffraction of heat/moisture treated starch granules. *Starch/Stärke*, *46*, 463–469.

Krueger, B. R., Knutson, C. A., Inglett, G. E., & Walker, C. E. (1987). A differential scanning calorimetry study on the effects of annealing on gelatinisation behavior of corn starch. *Journal of Food Science*, *52*, 715–718.

Krueger, B. R., Walker, C. E., Knutson, C. A., & Inglett, G. E. (1987). Differential scanning calorimetry of raw annealed starch isolated from normal and mutant maize genotypes. *Cereal Chemistry*, *64*, 187–190.

Kuge, T., & Kitamura, S. (1985). Annealing of starch granules warm water treatment and heat-moisture treatment. *Journal of Japanese Society of Starch Science*, *32*, 65–83.

Larsson, I., & Eliasson, A.-C. (1991). Annealing of starch at an intermediate water content. *Starch/ Stärke*, *43*, 227–231.

Lorenz, K., & Kulp, K. (1982). Cereal and roots starch modification by heat-moisture treatment. I. Physico-chemical Properties. *Starch/Stärke*, *34*, 50–54.

Mendes da Silva, C. E., Sasaki, J. M., Freitas e Silva, J. A., Jorge, F. A. da S. . , & Romeu, M. A. P. (1997). *Efeito do Annealing e do Tipo de Radiação no Crescimento do Biscoito de Amido de Mandioca Obtido por Fermentação Natural. X*. Buenos Aires: Seminário Latino-americano y del Caribe de Ciência y Tecnologia de Alimentos.

Moorthy, S. N. (1985). Effect of different types of surfactants on cassava starch properties. *Journal of Agricultural and Food Chemistry*, *33*, 1227–1232.

Nakamura, I. M., & Park, Y. K. (1975). Some physicochemical properties of fermented cassava starch (polvilho azedo). *Starch/Stärke*, *27*(9), 295–297.

Paredes-López, O., & Hernández-López, D. (1991). Application of differential scanning calorimetry to amaranth starch gelatinization: Influence of water, solutes and annealing. *Starch/Stärke*, *43*, 57–61.

Plata-Oviedo, M. S. V. (1991). Efeito do tratamento Ácido nas propriedades físico-químicas e funcionais do Amido de Mandioca. Dissertação de Mestrado. Faculdade de Engenharia de Alimentos, Campinas (135p).

Rosenthal, F. R. T., Nakamura, T., Espindola, A. M. C., & Jochimek, M. R. (1974). Structure of starch granules. *Die Stärke*, *26*(2), 50–55.

Sair, L. (1967). Heat-moisture treatment of starch. *Cereal Chemistry*, *44*, 8–26.

Schoch, J. T. (1964). Swelling power and solubility of starch. In R. L. Whistler, R. J. Smith, & I. N. BeMiller, *Methods in carbohydrate chemistry* (Vol. 4) (pp. 106–108). London: Academic Press, 106–108.

Sekine, M., Otobe, K., Sugiyama, J., & Kawamura, Y. (2000). Effects of heating, vacuum drying and steeping on gelatinization properties and dynamic viscoelasticity of various starches. *Starch/Stärke*, *52*, 398–405.

Stute, R. (1992). Hydrothermal modification of starches: The difference between annealing and heat-moisture treatment. *Starch/Stärke*, *44*, 205–214.

Tester, R. F., Debon, S. J. J., & Karkalas, J. (1998). Annealing of wheat starch. *Journal of Cereal Science*, *28*, 259–272.

Tester, R. F., Debon, S. J. J., & Sommerville, M. D. (2000). Annealing of maize starch. *Carbohydrate Polymers*, *42*, 287–299.

Wang, W. I., Powel, A. D., & Oates, C. G. (1997). Effects of annealing on the hydrolysis of sago starch. *Carbohydrate Polymers*, *33*, 195–202.