



## Influence of drying temperature on functional properties of wet-milled starch granules

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### ABSTRACT

Relationships between swelling capacities, pasting properties, rotational flow behaviour and textural properties of hydro-thermally heated wet-milled starch granules from corn dried between 60 and 130 °C were investigated. High-drying temperatures applied during the corn drying process conferred to the wet-milled starch granules (WSG) such a rigidity which reduced their swelling capacities, their water binding capacities and their water solubility index after gelatinization. These granules changes affected their pasting characteristics, their flow behaviour and several textural parameters of gel formed from the wet-milled starch granule after gelatinization. The rigidity of granules was a major factor determining the formation of either starch pastes or gels.

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### 1. Introduction

Corn is a near-perfect starch crop, which is easily dried and yields over 85% of the starch produced all over the world (Eckhoff, 2004).

After the harvesting, corn kernels are usually dried to achieve a safe moisture level and to inhibit the growth of microorganisms. An important consideration when dealing with the heated air process is the drying temperature, which depends on the end use of the grain and the residence time of the grain in the dryer (Jayas & White, 2003). Nowadays it is common to find industrial dryers and pilot plant-size bed dryers operating with air heated above 120 °C (Pallai, Németh, & Mujumdar, 1987) in spite of the consequences of high-drying temperatures on properties of corn and its derivatives (Malumba, Vanderghem, Deroanne, & Béra, 2008).

In wet-milling high-drying temperatures are recognized as detrimental to starch recovery (Eckhoff, 2004; Lasseran, 1973; Mistry, Wu, Eckhoff, & Litchfield, 1993; Peplinski, Paulis, Bietz, & Pratt, 1994; Weller, Paulsen, & Steinberg, 1988; Wight, 1981). Drying

temperature can also have an impact on thermal (Altay & Gunasekaran, 2006; Haros, Tolaba, & Suarez, 2003), rheological (Hardacre & Clark, 2006) and structural properties, reducing the swelling capacity of wet-milled starch granules (WSG).

The ability to form a viscous paste or a gel after appropriate hydrothermal treatments is the most important property which makes starches suitable for numerous uses (Nguyen, Jensen, & Kristensen, 1998). Such a property is closely dependent on structural characteristics of the starch granule and the pasting procedure applied during starch-based food formulation (Doublier, Llamas, & Le Meur, 1987).

Many investigations have dealt with the rheology and pasting properties of gels from starch granules. Rao and Tattiyakul (1999) studied the effect of granules size after hydrothermal treatment of starch in the range of 61–74 °C. They concluded that the swollen granule size distribution plays an important role in the flow properties of starch-aqueous system. Christianson and Bagley (1983) demonstrated that the apparent viscosity of aqueous corn starch dispersions heated to temperatures from 65 to 80 °C of temperature depends strongly on the volume of the granules. Sandhu and Singh (2007) observed that swelling power was positively correlated with the pasting properties of corn starches from different corn varieties. Nayouf, Loisel, and Doublier (2003) described the rheological properties of crosslinked waxy maize pastes related

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to granules properties under conditions close to those used in industry. They showed that these properties can be interpreted on the basis of the swelling capacity of starches.

These previous studies suggest that the changes induced on starch granules structures during the corn drying process may modify rheological properties and pasting behaviour of starch granules, with consequences on process development and properties of starch-based food.

There is little reported work that demonstrate the modifications in pasting behaviours, apparent viscosity and textural properties of wet-milled starch gel induced after gelatinization when corn kernels are subjected to various drying temperature.

The present study investigates relationships between the swelling capacity, pasting properties, rotational flow behaviour, textural properties of hydro-thermally heated wet-milled starch granules and the corn kernel drying temperatures.

## 2. Materials and methods

### 2.1. Fluidized-bed drying and sample preparation

A flint corn of Baltimore variety was field grown at CRA-Wallon Experimental Station (Gembloux, Belgium), and was harvested at approximately 0.45 g of water/g of dry matter. Shelled corns were received in laboratory immediately after harvesting and were stored at  $-18^{\circ}\text{C}$  in sealed plastic bag until the drying process was performed. Before the drying process, samples were equilibrated at ambient temperature for one night.

The drying stage was done in a laboratory fluidized-bed dryer. Approximately 700 g of wet corn grains were dried in a cylindrical duct inside of which air flow, previously heated at experimental temperature, was pulsed. The air flow velocity was chosen to induce fluidization and ensure a maximal mass transfer between the air and the kernels to maintain all kernels at the same temperature.

Experiments were carried out at temperatures between 54 and  $130^{\circ}\text{C}$  in triplicate and undried corns were used as control. For each air-drying temperature, a specific processing time was chosen in order to obtain a final moisture content of grains between 0.12 and 0.15 g of water/g of dry matter. Constant drying temperatures were ensured by a PID regulator and were recorded by 14 thermocouples (Cu/Cn). After drying, corn kernels were equilibrated at ambient temperature and conserved in a sealed bag at  $10^{\circ}\text{C}$ .

### 2.2. Laboratory wet-milling procedure

The laboratory wet-milling procedure developed by Neryng and Reilly (1984) was performed, with some modifications suggested by Haros and Suarez (1997), Pérez, Haros, Suarez, and Rosell (2003), Singh and Eckhoff (1996) and Steinke and Johnson (1991). Artificially dried corn batches of 500 g were steeped at  $50^{\circ}\text{C}$  for 48 h in 1.2 L of solution containing 0.6% (w/v) of sodium bisulfite.

Steeped corns were finely ground at 3600 rpm, using a mikrocult grinder (Model MCV12, Stephan Machinery corp., Germany) and then sieved through 0.40 and 0.05 mm on an operating sieve shaker (Model Sweco). The fractions retained by each sieve were washed successively with 1 L of distilled water to isolate starch completely. The sieved slurry was decanted during one night at  $2^{\circ}\text{C}$  and the supernatant was separated. Approximately 2 L of settling residues was dispersed in water and centrifuged twice at 7333g (Sorval RC centrifuge 12BP, USA) for 10 min. After each centrifugation, the supernatant was eliminated and the protein layer was scrapped off. More water (1.5 L) was added to the partially cleaned starch, which was sieved again on a small nylon sieve

(0.400 mm openings) in order to eliminate agglomerated residual proteins. The starch slurry was frozen at  $-20^{\circ}\text{C}$  for 24 h and then freeze-dried for 48 h at a pressure  $<0.1$  mbar.

### 2.3. Chemical analysis

The corn kernels moisture contents were measured by oven drying at  $105 \pm 2^{\circ}\text{C}$  for 72 h. The moisture content of freeze-dried starches recovered from the extraction procedures was determined by measuring the weight loss of 5 g of sample after 165 min at  $130^{\circ}\text{C}$  (ISO 712:1998).

Starch yields were calculated on a dry basis as the ratio of the weight of starch recovered to the total weight of the corn sample used on a wet-milling procedure. The starch recovery was calculated as a ratio of the starch yield to the total weight of starch present in the corn.

Starch contents of dried kernels and wet-milled starch recovered were measured using the Ewers method (ISO 10520:1997). The residual protein on a wet-milled starch was determined by the Kjeldahl method, with a 2020 Tecator Digester (Tecator, Sweden) and a 2100 Kjeltac distiller (Tecator, Sweden). Proteins were calculated using the general factor 6.25.

The apparent amylose content of starch granules was determined by a modified iodometric method of Morrison and Laignelet (1983) as reported by Massaux et al. (2008).

### 2.4. Water binding capacity and swelling capacity

In the present study the swelling capacities of WGS were determined by granule size analysis with a laser granulometer (Malvern Instruments Inc., Worcestershire, UK). This gave the distribution of granule volumes (Ziegler, Thomson, & Casasnovas, 1993). A so called “swelling capacity” obtained by the modified method of Leach, McCowen, and Schoch (1959) as described by Tang, Watanabe, and Mitsunaga (2002) was termed in this study as “water binding capacity of gelatinized starch”.

#### 2.4.1. Water binding capacity of unheated WSG

Approximately 5 g of starch (on dry basis) was added to 100 ml of distilled water in 250 ml Beckman centrifuge bottle. The starch suspension was stirred for 1 h and allowed to settle overnight at  $4^{\circ}\text{C}$ . After removing carefully the free water from the settled wet starches, bottles were weighed and the amounts of water held by the starch were determined.

#### 2.4.2. Water binding capacity of gelatinized WSG and solubility Index

Starch (0.1 g) was weighted in triplicate into a centrifuge tube with screw cap and 5 ml of a 2 mM  $\text{AgNO}_3$  solution was added to nullify  $\alpha$ -amylase effects and facilitate comparisons between starches (Abdel-Aal, Hucl, Chibbar, Han, & Demeke, 2002; Massaux et al., 2008). These tubes were placed in a shaking water bath at  $70^{\circ}\text{C}$  for 10 min and then transferred into a boiling water bath for another 10 min. The tubes were then cooled by immersing them in cold water during 10 min and centrifuged at 9000g for 10 min according to Tang et al. (2002).

The supernatant was poured out from tubes and the residue was dried to constant weight in an air oven at  $105^{\circ}\text{C}$  for one night. The sediment was then weighted and water binding capacity after gelatinisation was calculated by the method proposed by Li and Yeh (2001). Measurements were performed in triplicate and the results were presented as a mean with standard deviation.

#### 2.4.3. Swelling capacity

Particle size distribution was determined at room temperature using laser scattering analyser (Malvern Instruments, Ltd., UK) equipped with a Mastersizer 2000 (Ver. 5.22) analysis station data.

To compare the WSG swelling capacities, a swelling ratio defined as  $(D_T/D_0)^3$ , according to Ziegler et al. (1993) was calculated for each sample.  $D_0$  designed the median diameter of WGS extracted from undried corn and  $D_T$  the median diameter of WSG extracted from corn dried at temperatures ( $T$ ) before heating (for the calculation of the swelling capacity of unheated WSG) or after gelatinization (for the swelling capacity of gelatinized WSG).

The swelling ratio of gelatinized WSG was calculated using median diameter reached by the starch granules gelatinized following the similar procedure as described for determining the binding capacity of gelatinized WSG. After the second heating, approximately 40 ml of cold water was rapidly injected into the tube containing gelatinized starch dispersion in order to prevent anymore swelling before measuring the size of particles using the laser granulometer.

## 2.5. Pasting behaviour

The Brabender visco-amylograph (Duisberg, Germany) was used to obtain the pasting characteristics of wet-milled starches from corn dried. Ten grams of starch (dry basis) were mixed at room temperature with 100 g of deionised water, and then submitted to a gradual heating ( $6.5\text{ }^\circ\text{C min}^{-1}$ ) from 30 to  $96\text{ }^\circ\text{C}$ . This temperature was maintained during 10 min followed by a gradual cooling step ( $-4.5\text{ }^\circ\text{C min}^{-1}$ ) from 96 to  $50\text{ }^\circ\text{C}$ . This temperature was maintained for 5 min before the end of pasting.

The relevant values obtained from the pasting profile were: the peak viscosity (the maximum viscosity during pasting), breakdown viscosity (the difference between the peak viscosity and the viscosity at the end of the holding phase), set-back viscosity (the difference between the viscosity at the end of cooling and the viscosity at the end of the holding phase) and final viscosity (the viscosity at the end of cooling). All measurements were performed in triplicate and viscosities were expressed in BU (Brabender units).

## 2.6. Rotational flow properties

In order to determine the flow behaviour of gelatinized starch–water system, rotational flow characteristics of 2% starch dispersion were measured at  $20\text{ }^\circ\text{C}$ .

One gram of starch (dry basis) was mixed with 50 g of deionised water containing 2 mM of  $\text{AgNO}_3$ . The slurry was then submitted to a gradual heating ( $5\text{ }^\circ\text{C min}^{-1}$ ) from 20 to  $100\text{ }^\circ\text{C}$ . This temperature was maintained for 30 min and was followed by a cooling to ambient temperature. The heating and the cooling procedures were performed under mixing at 10 rpm in a rotating batch retort A091 (FMC-Europ N.V, Belgium) equipped with a Siemens Simatic TP25 command pilot and a digital interface Ellab TA 9616 for temperature measurements.

Rheological measurements were performed in a Rotovisco Haake RV20 (Germany) rotational viscosimeter fitted with a thermostatic bath for temperature control. A Haake CV20 controller was used to program the tests and the sensor System ME30. The steady shear data on the gelatinized starch dispersion were determined using a stainless steel cone/cylinder ( $4^\circ$  angle, 4 cm diameter) and plate geometry. Each sample (3 ml) was sheared at  $20\text{ }^\circ\text{C}$  from  $0.05$  to  $380\text{ s}^{-1}$  and back to  $0.05\text{ s}^{-1}$  for 10 min. Following Rao, Okechukwu, Da Silva, and Oliveira (1997) and Rao and Tattiyakul (1999) only descending shear data were analyzed for the flow behaviour.

## 2.7. Texture profile analysis (TPA)

Gels used for textural analysis were prepared with a 7% (dry basis) of starch–water dispersion in cylindrical cans having 0.030 m height and 0.073 m diameter hermetically sealed.

The heating procedure was performed at 10 rpm on a rotating batch retort A091. Canned starch suspensions were submitted to a gradual heating from 20 to  $90\text{ }^\circ\text{C}$  for 20 min. This temperature

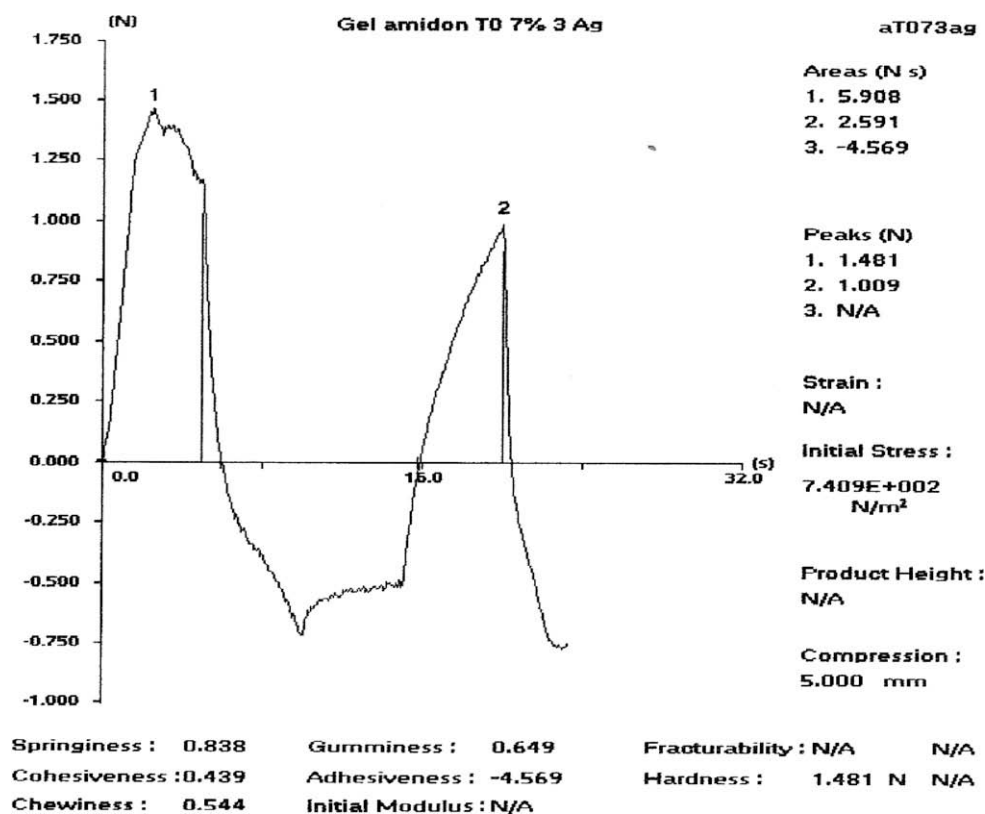


Fig. 1. Trace of force–time curve obtained for a starch gel 7% as measured by a stable micro-system texture analyser.

was maintained for 10 min and followed by a rapid cooling step without agitation, in order to obtain gels with a horizontal surface on which the texture measurements could be applied.

The gels formed in the cans were characterized by a texture profile analysis (TPA). Opened cans containing starch gel were placed on the sample holder of the texturometer TA/XT2 (Stable MicroSystem, Surrey, England) and the gels were compressed with a cylindrical probe (TA/XT2 3.6R). The compression distance was 5 mm which represents approximately 5% of the total gel thickness. Measurements were repeated twice in the same gel to generate the force–time curve. The pre-test and test speeds were fixed at 1 mm/s and the plot speed was 2 mm/s. Measurements were performed in triplicate using three different gels.

Fig. 1 shows a typical force–time curve of a starch gel and texture profile parameters as obtained by using the TA/XT2 texturometer.

Parameters calculated from TPA were springiness (ratio between recovered height after the first peak and the original gel height), cohesiveness (ratio between the area under the second peak and the first), adhesiveness (negative area of the curve during retraction of the probe), chewiness (result of multiplying the gumminess by the springiness), gumminess (result of multiplying the hardness by the cohesiveness), and hardness (height of first peak).

### 3. Results and discussion

#### 3.1. Wet-milling starch yield and starch chemical characterization

The temperatures used for corn drying, the moisture content of kernels (on dry basis) after the fluidized-bed drying, the performance of the wet-milling procedure and the chemical characteristics of WSG from corn dried at different temperatures are presented in Table 1.

The results showed that wet-milling starch recovery decreased significantly when the drying temperatures increased. However, starches recoveries here are lower than those obtained by Haros et al. (2003). This situation may be caused by a more drastic purification procedure in the present study. The starch yields obtained by Haros and Suarez (1997) were higher than those reported in the literature, probably due to an important amount of residual protein content on recovered WSG (0.9 to 5.72 g/100 g).

The purity of all samples was higher than 95%. Above 80 °C, the residual protein contents of WSG increased slightly with the drying temperature. However, the protein residues were almost less than 1.5% in all samples analyzed in this study.

Debet and Gidley (2006) associated an increase of the residual protein and the lipid on starch granule to the restriction of their swelling capacity during gelatinization. According to Altay and Gunasekaran (2006) and Haros et al. (2003) the increase of this

residual protein in WSG could participate on changes of the hydro-thermal's properties of WSG. Unfortunately there is little published data on the importance of residual protein on properties developed by WSG.

When drying temperatures increase from 60 to 130 °C, the apparent amylose contents of WSG range between 20.7 and 21.7 g/100 g of starch. A similar range of amylose contents was observed by Sandhu, Singh, and Kaur (2004) for a dent corn. However the apparent amylose content of WSG from the undried corn (20.7) was lower than that reported by Tester and Morrison (1990) (29.4) for normal corn. The variation in amylose contents among the starches from different corn variety may be attributed to the different starch isolation procedures and analytical methods used to determine amylose content (Singh, Singh, Kaur, Sodhi, & Gill, 2003).

Lii, Tsai, and Tseng (1996) stated that the rigidity of starch granular structure might be in proportion to its amylose content and inversely proportional to the degree of granular swelling. Lu, Duh, Lin, and Chang (2007) showed that granule's properties and interactions occurred between the amylose and the starch granules play an important role during the gelation of starch. Mua and Jackson (1997) demonstrated that the pasting and gel textural properties hydrothermal treatment related to the corn amylose content. Tester and Morrison (1990) also postulated that inclusion complexes may be formed between the natural starch lipids and the residual amylose in the granules during heating, and this can prevent the leaching of amylose from granules during gelatinization. Jane et al. (1999) concluded that the amylose content and the amylopectin branch chain-length distributions predominantly affect the pasting properties of starches.

Samples used in the present study were submitted to the same fractionation and analytical procedures and they did not reflect significant changes on the apparent amylose content. Differences observed between functional properties could not be attributing to the change in this parameter.

#### 3.2. Water binding capacity of WSG and water solubility index

Using the Leach, McCowen, and Schoch (1959) method, Bagley and Christianson (1982) defined the swelling capacity of starch as the weight of swollen granules divided by their dry weight. Therefore, the swelling capacity determines water entrapped in the gel formed by the heating granules starch in excess of water until complete gelatinization. This definition assumes that the sediment obtained after cooling and centrifugation of the heated starch suspension is entirely filled with swollen granules without interstitial water between particles (Nayouf et al., 2003).

In reality, gels obtained after complete gelatinization, cooling and centrifugation are the three dimensional crosslinked hydrophilic networks able to absorb and hold water under ordinary

**Table 1**  
Drying parameters, wet-milling performance and chemical characteristics of starch yield after fractionation of corn dried at different temperatures

| Air drying temperature (°C) | Final moisture content (g/100 g of grains) | Starch yield (g/100 g) | Starch recovery (g/100 g of starch) | Apparent amylose content (g/100 g starch) | RPS <sup>a</sup> (g/100 g of starch) | WSG purity (g/100 g of starch) |
|-----------------------------|--|------------------------|-------------------------------------|---|--------------------------------------|--------------------------------|
| Untreated                   | 47.4 ± 0.6                                 | 64.4 ± 0.4             | 90.7 ± 0.5                          | 20.7 ± 0.4                                | 0.69 ± 0.01                          | 99.2 ± 0.1                     |
| 54                          | 14.8 ± 0.0                                 | 61.5 ± 0.4             | 86.4 ± 0.5                          |   | 0.69 ± 0.01                          | 98.3 ± 0.1                     |
| 60                          | 14.3 ± 0.2                                 | 60.5 ± 0.4             | 85.2 ± 0.5                          | 20.8 ± 0.8                                | 0.69 ± 0.02                          | 98.7 ± 1.0                     |
| 70                          | 14.3 ± 0.0                                 | 57.8 ± 0.6             | 81.4 ± 0.8                          |   |                                      |                                |
| 80                          | 13.9 ± 0.0                                 | 55.8 ± 0.5             | 78.2 ± 0.7                          | 21.7 ± 0.3                                | 0.69 ± 0.00                          | 99.27 ± 1.1                    |
| 100                         | 11.4 ± 1.1                                 | 47.3 ± 0.8             | 66.6 ± 1.1                          | 21.0 ± 0.3                                | 0.79 ± 0.04                          | 99.4 ± 1.4                     |
| 110                         | 14.2 ± 0.2                                 | 44.7 ± 0.5             | 63.0 ± 0.7                          |   |                                      |                                |
| 120                         | 12.0 ± 0.7                                 | 44.5 ± 0.4             | 62.7 ± 0.6                          |   | 1.38 ± 0.03                          | 97.6 ± 0.8                     |
| 130                         | 14.2 ± 0.4                                 | 43.1 ± 0.1             | 61.8 ± 0.1                          | 21.0 ± 0.2                                | 1.27 ± 0.09                          | 95.4 ± 1.3                     |

<sup>a</sup> RPS = residual protein in wet-milled starch.



pressure (Pal, Banthia, & Majumdar, 2008). These gels are composed by swollen granules and granule constituents which leach inside the suspending medium as stated by Lu et al. (2007).

Changes in the water binding capacity of an unheated and gelatinized WSG together with their solubility indexes after hydrothermal treatment are summarized in the Table 2. A significant increase in the water binding capacity was observed by the unheated WSG from corn dried at 130 °C. Therefore, this suggests a partial gelatinization of starches imbedded in corn kernel during the drying process at this temperature. Little change occurred below 100 °C.

The water binding capacity of a gelatinized WSG from undried corn was in the same range as values reported by Tsai, Li, and Lii (1997) for a normal corn starch. Leach et al. (1959) found a value of 22 g water/g MS. Sandhu and Singh (2007) reported values in the range of 13–20.7 g H<sub>2</sub>O/g dry starch for nine corn landraces.

In contrast with the water binding capacity of unheated WSG, the water binding capacity of gelatinized starches decreases when the corn drying temperature increases. Haros and Suarez (1997) observed the same phenomenon. Probably, this is in related to the restriction on the swelling capacity conferred to granules by the high-drying temperature.

The water solubility index of WSG decreases significantly when corn kernels are dried above 100 °C. High-drying temperature increase the immobilization of starch constituents within the granule. Leach et al. (1959) postulated that the bonding force within the starch granules would influence its manner of swelling. Thus, a highly associated starch should be relatively resistant to swelling.

### 3.3. Swelling capacity of WSG

Similarly to the water binding capacity, when aqueous suspension of WSG where heated above the temperature onset of normal corn (>62 °C reported by Jenkins & Donald (1998)), granules starches from corn dried at higher temperature presented lower swelling ratio than the ones from corn kernels dried at lower temperature (Table 3). This swelling trend is in contrast to what was observed for unheated WSG.

Therefore, differences among swelling behaviours of various starches can be attributed to changes conferred to the structure of starch granules during the corn drying procedures.

### 3.4. Pasting behaviour of WSG

Pasting is the phenomenon following gelatinization in the dissolution of starches. It involves granular swelling, exudation of the granular molecular components, and eventually, total disruption of the granules (Atwell, Hood, Lineback, Varriano-Marston, & Zobel, 1988). Viscosity changes of starch dispersions during gelatinization are the most frequently measured with the Brabender

**Table 3**

The swelling ratio ( $D_T/D_0$ )<sup>3</sup> of unheated and gelatinized WSG from corn dried at different temperature

| Drying temperature (°C) | Unheated WSG | Gelatinized WSG |
|-------------------------|--------------|-----------------|
| To                      | 1.0 ± 0.0    | 20.7 ± 0.6      |
| 60                      | 1.0 ± 0.0    | 19.2 ± 0.9      |
| 80                      | 1.1 ± 0.0    | 16.6 ± 0.7      |
| 100                     | 1.2 ± 0.0    | 13.8 ± 1.0      |
| 130                     | 2.4 ± 0.1    | 13.6 ± 0.3      |

$D_0$  is the median diameter of WSG extracted from undried corn (without anymore heating).

$D_T$  is the median diameter of WSG extracted from corn dried at temperatures ( $T$ ) before heating (for the calculation of the swelling capacity of unheated WSG) or after gelatinization (for the swelling capacity of gelatinized WSG).

Visco-amylograph which is used in the food industry as a quality control instrument (Lagarrigue & Alvarez, 2001).

The visco-amylograph curves of WSG from corn kernels dried at different temperatures are presented in the Fig. 2. It can be observed that the onset temperatures increase, while the peak viscosities decrease when the corn drying temperature increase. These phenomena were also observed by Mistry et al. (1993). Similar observations were made after the heat-moisture treatment of starches from corn (Hoover & Manuel, 1996) and from different botanical origins (Hoover & Vasanathan, 1994; Jacobs, Eerlingen, & Delcour, 1996; Stute, 1992).

According to Barichelo, Yada, Coffin, and Stanley (1990) high gelatinization transition temperatures are indicative of a high degree of crystallinity, which provides structural stability and makes starch gelatinization difficult.

Possible explanation of this effect consists of an increase of thermal stability of granules during the drying process at high temperatures. This stability can be due to some changes within the amorphous and crystalline phase of granules (Jacobs & Delcour, 1998) and it can also induce the reducing of final swelling capacity of granules and that of amylose leaching out.

Quantitative values of onset temperatures, peak viscosities, final viscosities after the cooling of gel and the calculated value of breakdown of the viscosity during the heating period and set-back of viscosity are presented in the Table 4.

The peak and breakdown of viscosity, which are reached by WSG on the heating period, decrease significantly with the increase of drying temperatures. The peak's height of viscosity at a given concentration reflects the ability of the granules to swell freely before their physical breakdown. Starches that are capable of swelling to a high degree are also less resistant to breakdown on cooking and hence exhibit viscosity decreases significantly after reaching the maximum value (Singhet al., 2003). Thus, it can be concluded that high-drying temperature decreased the swelling capacity of WSG and their susceptibility to breakdown during hydrothermal cooking.

A slight tendency to the increasing of final viscosity and set-back value with corn drying temperature was observed till 110 °C. Above 110 °C the starch granule rigidity was associated with an important restriction of the leaching of amylose in aqueous medium which had as consequence an important reducing of the network strength formed.

The decrease of the breakdown viscosities with the increase of drying temperatures is an indication of the rigidity conferred to starches during the corn kernel drying. Singh et al. (2003) postulated that differences in the breakdown values of starches may be attributed to the granule's rigidity, lipid content and peak in the dynamic modulus of viscosity.

**Table 2**

Water binding capacity and water solubility index of WSG from corn dried at different temperatures

| Drying temperature (°C) | Water binding capacity of unheated WSG (g H <sub>2</sub> O/g of starch) | Water binding capacity of gelatinized WSG (g H <sub>2</sub> O/g of starch) | Water solubility index (g solubles/100 g of starch) |
|-------------------------|---|--|---|
| Undried                 | 2.1 ± 0.1   | 16.3 ± 0.8   | 13.9 ± 0.1  |
| 60                      | 2.2 ± 0.1   | 17.2 ± 0.9   | 14.8 ± 1.1  |
| 80                      | 2.3 ± 0.1   | 15.3 ± 1.0   | 14.5 ± 0.4  |
| 100                     | 2.2 ± 0.0   | 15.1 ± 1.2   | 11.5 ± 0.8  |
| 130                     | 3.4 ± 0.2   | 13.3 ± 0.3   | 11.5 ± 0.6  |

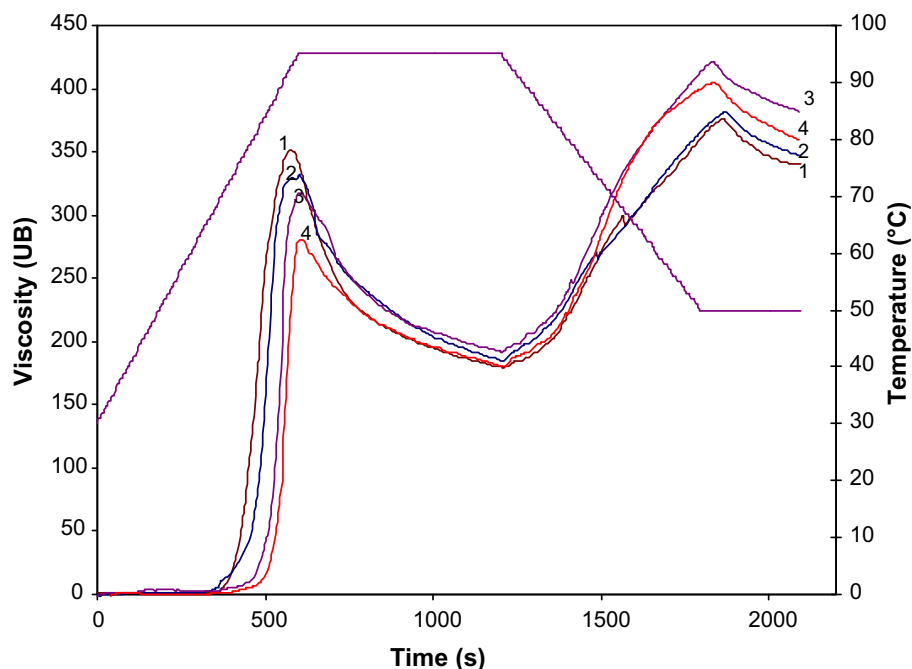


Fig. 2. Brabender amylograph curves of WSG from undried corn (1) and corn dried at 60 °C (2); 100 °C (3); 120 °C (4).

### 3.5. Flow properties

Fig. 3 shows that the apparent viscosity of the aqueous starch dispersion heated at 100 °C for 30 min decreases when the shear rate increases. For comparative purpose the flow behaviour is only presented for values of shear rate between 0.7 and 8 s<sup>-1</sup>. Above the shear rate of 10 s<sup>-1</sup>, the apparent viscosity did not show significant changes.

The results presented in the Fig. 3 show that the aqueous starch dispersion heated at 100 °C for 30 min were shear rate dependent in the range of 0.7 to 8 s<sup>-1</sup>. This behaviour was defined as shear-thinning (or pseudo-plastic). The shear rate dependent behaviour of gelatinized starch dispersion was also observed by Christianson and Bagley (1983), Christianson and Bagley (1984), and Doublier et al. (1987). Rao et al. (1997) observed a shear-thinning behaviour of corn starch when heated at 85 °C.

The apparent viscosity of heated WSG dispersions rose as the shear rate approached zero. This behaviour was suggestive of the existence of yield stress, which requires a minimum value of stress necessary to initiate the flow. Similar flow behaviour of heated starch dispersions was observed by Rao et al. (1997).

### 3.6. Textural properties

Texture is the sum of different properties of foods that are sensed by several different organs of the human body (Bourne,

1968). By modifying physicochemical properties of wet-milled starch granule, it was assumed that textures of starch gel may be influenced.

In the present study, it was observed that textural parameters of WSG varied with the temperature applied during the corn drying (Table 5). The increasing of drying temperature induced a diminution of hardness and adhesiveness of gel formed all over the range of drying temperature; whereas their gumminess, springiness and chewiness increased slightly below 100 °C before decreasing abruptly above 110 °C.

Cohesiveness did not decrease as the other textural parameters of starch gels. In contrast, this value increased with the increasing of corn drying temperature. According to Bourne (1968) and Sandhu and Singh (2007), cohesiveness was calculated as the ratio between the area under the second peak and that of the first peak of compression. The decreasing of the area generated by the first compression of gel was more important than that of the second compression when drying temperature increased and thus induced an increasing of cohesiveness with drying temperature.

Gels produced by WSG from corn dried at higher temperature were weak and seems to be completely fluid for corn dried at 120 and 130 °C indicating a lack of a gel network building. For these fluid suspensions obtained, the area of first compression tends to decrease and furthermore the second area was maintained.

Table 4

Pasting properties (Brabender units) of wet-milled starch granule isolated from corn dried at different temperature

| Drying temperature (°C) | Onset Temperature (°C) | Peak viscosity | Final viscosity | Breakdown viscosity | Set-back viscosity |
|-------------------------|------------------------|----------------|-----------------|---------------------|--------------------|
| Untreated               | 73.1 ± 0.8             | 346.7 ± 4.9    | 340.3 ± 1.5     | 160.7 ± 8.0         | 175.7 ± 6.4        |
| 60                      | 72.6 ± 1.4             | 351.7 ± 8.0    | 357.7 ± 15.0    | 173.7 ± 9.7         | 178.7 ± 2.5        |
| 70                      | 74.5 ± 0.7             | 346.0 ± 15.5   | 370.0 ± 16.9    | 175.5 ± 10.6        | 190.5 ± 0.7        |
| 80                      | 75.4 ± 1.2             | 330.0 ± 17.7   | 373.0 ± 3.9     | 160.7 ± 18.1        | 204.0 ± 5.2        |
| 100                     | 78.0 ± 0.1             | 313.5 ± 6.3    | 379.0 ± 4.2     | 125.0 ± 0.1         | 222.5 ± 4.9        |
| 110                     | 80.2 ± 0.1             | 311.3 ± 7.5    | 409.0 ± 3.6     | 130.3 ± 1.5         | 232.3 ± 2.9        |
| 120                     | 80.4 ± 0.0             | 284.3 ± 6.8    | 355.3 ± 4.9     | 110.7 ± 3.3         | 217.3 ± 0.9        |
| 130                     | 80.8 ± 0.8             | 269.7 ± 1.5    | 353.7 ± 6.6     | 95.0 ± 6.9          | 215.5 ± 0.7        |

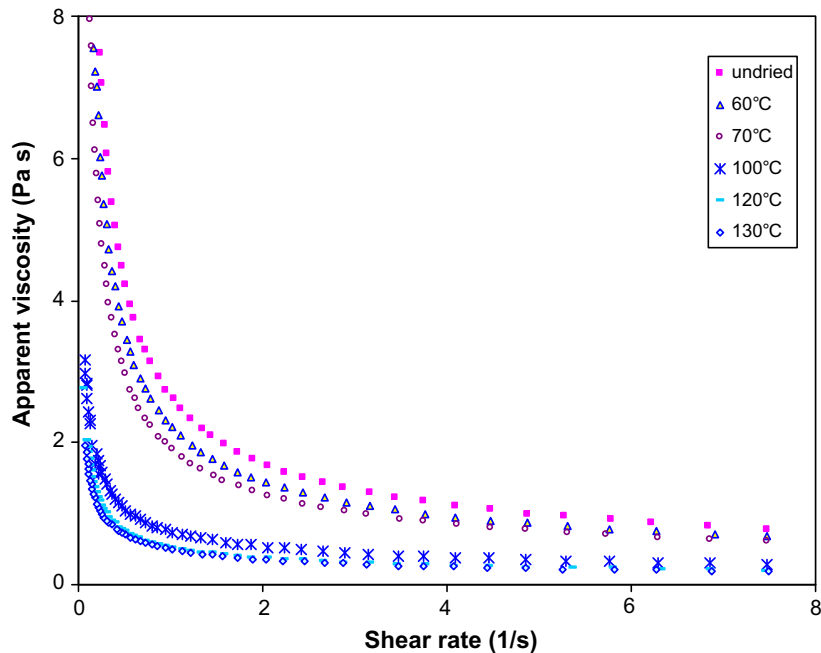


Fig. 3. Flow behaviour at ambient temperature of 3% wet-milled starch granule's dispersions heated at 100 °C for 30 min.

Table 5

Textural properties of gels formed with 7% of WSG heated at 90 °C

|                    | Hardness (N)     | Cohesiveness  | Gumminess     | Springness       | Chewiness       | Adhesiveness   |
|--------------------|------------------|---------------|---------------|------------------|-----------------|----------------|
| <b>Témoïn (T0)</b> | 1,47 ± 0,07 a    | 0,44 ± 0,01 a | 0,63 ± 0,03 a | 0,84 ± 0,00 a    | 0,53 ± 0,02 a   | −4,34 ± 0,33 a |
| <b>60</b>          | 1,48 ± 0,05 a    | 0,44 ± 0,01 a | 0,64 ± 0,01 a | 0,85 ± 0,00 a    | 0,55 ± 0,01 a   | −4,28 ± 0,35 a |
| <b>70</b>          | 1,49 ± 0,07 a    | 0,43 ± 0,01 a | 0,64 ± 0,06 a | 0,86 ± 0,00 a, e | 0,53 ± 0,01 a   | −4,16 ± 0,30 a |
| <b>100</b>         | 1,26 ± 0,17 a, b | 0,57 ± 0,07 b | 0,66 ± 0,10 a | 0,91 ± 0,01 b    | 0,60 ± 0,08 a,b | −1,64 ± 0,15 b |
| <b>110</b>         | 1,14 ± 0,11 b    | 0,59 ± 0,01 b | 0,68 ± 0,06 a | 0,95 ± 0,02 c    | 0,64 ± 0,07 b   | −1,10 ± 0,06 b |
| <b>120</b>         | 0,24 ± 0,01 c    | 0,77 ± 0,01 c | 0,18 ± 0,00 b | 0,89 ± 0,00 d, b | 0,16 ± 0,00 c   | −0,30 ± 0,04 c |
| <b>130</b>         | 0,25 ± 0,02 c    | 0,70 ± 0,05 c | 0,17 ± 0,00 b | 0,87 ± 0,01 e    | 0,15 ± 0,00 c   | −0,45 ± 0,06 c |

Means in the same column followed by different letters are significantly different according to Tukey's test ( $p < 0,05$ ).

The lack of a hydrogel network building during the starch gel preparation of wet-milled starch granule from corn dried at 120 and 130 °C may be attributed to the rigidity conferred to starch granule by high-drying temperature, which avoided the gel formation after hydrothermal treatment of starch. Tsai et al. (1997) concluded that rigidity of swollen starch granules was a major factor determining the formation of either pastes or gels.

#### 4. Conclusion

High-drying temperature appears to confer to starch granules such a rigidity which reduces their swelling capacities and their water solubility indexes during the gelatinization. Such structural changes conferred to starch granules affect the pasting characteristics of the corn starch by reducing their peak and breakdown of viscosity during the heating period of the pasting procedure and increase the onset temperatures of WSG gelatinization. The flow properties of starch dispersion heated at 100 °C displayed a specific shear-thinning character with an apparently decrease of yield stress when drying corn temperatures rise. The textural parameters of gel from wet-milled granules starches were also significantly affected by the air-temperature applied during the corn drying as measured instrumentally by the texture profile analyser. High temperatures applied during the corn drying have conferred to wet-milled starch granules (WSG) such a rigidity which was a major factor determining the formation of either pastes or gels.

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