

# Variations in physicochemical and functional properties of starches extracted from European soft wheat (*Triticum aestivum* L.): The importance to preserve the varietal identity

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## Abstract

The quality valuation of wheat is based on characteristics like yield, specific weight, protein content and protein quality. Until now, the starch properties are rarely considered as a quality criterion. In this study, we showed the influence of wheat cultivars (*Triticum aestivum* L.) and culture year on the intrinsic properties of starch, extracted from European wheat grown in the same conditions. For example, starch damage varied from 13.2 to 19.9 CDU in function of the cultivars and contribution of the B-type starch granules (<10 µm) to the total volume ranged from 11.6% to 29.9%. Starch viscosity at 95 °C, characterized with α-amylase inactivation by 2 mM AgNO<sub>3</sub> addition, varied from 276.5 to 351.5 BU with the wheat cultivars. It is apparent from this study that starch properties were principally influenced by the wheat cultivar and slightly by the culture year. A good relationship between the pasting properties of whole flour and starch were finally established, showing the important role of starch in the whole flour viscosity. A thorough working knowledge of starch properties could lead to an appropriate selection of wheat cultivar, well-adapted to industrial end uses, without encountering processing or end-products quality problems and with most cost-competitive production.

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**Abbreviations:** BU, Brabender units; CDU, Chopin-Dubois units; cP, Centipoise units; LSD, least significant differences; NSP, non-starch polysaccharide; RVA, rapid visco analyser; UDMSO, urea-dimethyl sulfoxide

**Keywords:** Wheat starch; Extraction; Functional properties; Whole flour

## 1. Introduction

Different aspects are taken into account to evaluate the wheat quality: yield, disease resistance, climatic tolerance, specific weight, protein content, protein quality and baking performance. As an important ingredient in most wheat-based products, flour and proteins exert a major effect on

the wheat quality. But not one of these quality criteria concerns the starch. Starch represents nevertheless 67–68% of wheat whole grain and constitutes the major component of wheat flour (78–82%) (Feillet, 2000). To be able to effectively use flour to make high-quality products without encountering processing or end-product quality problems requires a thorough working knowledge of all aspects of wheat and its flour.

Starch consists of glucose polymers, amylose and amylopectin. Amylose is essentially linear, consisting of (1 → 4)-α-linked D-glucopyranosyl units with molecular

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weights in the range  $10^5$ – $10^6$  g/mol. In contrast, amylopectin is highly branched and consists of chains (1 → 4)- $\alpha$ -linked D-glucopyranosyl units joined through (1 → 6)- $\alpha$ -linkages. Amylopectin is one of the largest biopolymers known with typical molecular weights being in the region of  $10^8$  g/mol (Parker & Ring, 2001). The relative amounts of amylose and amylopectin are known to influence both nutritional and technological properties such as susceptibility to enzymatic hydrolysis, gelling and pasting behavior, which could be of biotechnological importance (Gérard, Barron, Colonna, & Planchot, 2001). For example, high amylose starches are widely used as thickeners and strong gelling agents but present the retrogradation problem. The introduction of amylopectin allows improving the uniformity, stability and texture of gelatinized starches. It also imparts better freeze-thaw stability in frozen foods (Slattery, Kavakli, & Okit-a, 2000).

At maturity, wheat endosperm contains two types of starch granules: large A- and small B-type (Parker & Ring, 2001; Peng, Gao, Abdel-Aal, Hucl, & Chibbar, 1999). A-type starch granules are disk-like or lenticular in shape with an average diameter of 10–35  $\mu$ m and contribute >70% of the total weight and  $\approx$ 3% of the total granule number of endosperm starch. On the other hand, B-type starch granules are roughly spherical or polygonal in shape, ranging from 1 to 10  $\mu$ m in diameter. These account for >90% of the total granule number but <30% of the total weight of starch in wheat endosperm (Morrison & Gadan, 1987). Wheat A- and B-type starch granules are reported to have significantly different chemical compositions and functional properties. Raeker, Gaines, Finney, and Donelson (1998) and Soulaka and Morrison (1985) described higher amylose content in large granule, and higher lipid content in small granules. Sahlström, Baevre, and Brathen (2003) reported A-granule fraction with the highest peak viscosity, minimum viscosity, breakdown viscosity, final viscosity and setback viscosity. These differences can result in the two starch granule types being utilized differently, both in food and non-food uses. For example, starch with predominantly B-type starch granules can be used as a fat substitute (Lim, Jane, Rajagopalan, & Seib, 1992), while starch with a high percentage of A-type starch granules has appli-

cations in the manufacture of biodegradable plastic film and carbonless copy paper (Nachtergaele & Van Nuffel, 1989).

The objective of the present study was to show the importance to preserve the varietal identity of wheat to insure specific starch properties. The variations in starch characteristics isolated from European wheat were evaluated as a function of the botanical sources and the culture year but with the same growing conditions. We investigated the distribution of granule size, amylose content and viscosity behaviour to understand the functionality of starches. The intrinsic properties of the starches were compared and the relationships among functionalities were analyzed. The quality of the starch is important in its many end use and a better knowledge of its properties could avoid encountering processing or end-products quality problems, with most cost-competitive production. Finally, we compared the pasting properties of whole flour and starch to evaluate the starch impact on the development of the whole flour pasting properties. A good relationship between these two characteristics could confirm the important impact of starch on wheat quality characteristics.

## 2. Materials and methods

### 2.1. Materials

Reagents were purchased from Sigma–Aldrich (Bornem, Belgium) and were of at least analytical grade.

The 10 European soft wheat cultivars evaluated were: Corvus, Folio, Ordéal, Agami, Apache, Buccaneer, Dream, Mercury, Meunier and Koch. Wheat seeds Corvus were obtained from Aveve (Landen, Belgium), Folio, Buccaneer, Mercury, Meunier and Koch were from Clovis Matton (Avelgem-Kerkhove, Belgium), Ordéal, Agami, Apache and Dream were from Jorion (Frasnes-lez-Buissenal, Belgium). These samples were screened to characterize a large panel of different wheat cultivars (Table 1). Because of the short market availability of some seeds, 6 of the 10 cultivars were planted for three successive growing seasons and the other 4 cultivars were planted for only two seasons.

Samples were obtained from wheat grown in the experimental field at Lonzée, Belgium. Sowing dates were 12

Table 1  
Specific characteristics of wheat cultivars

Cultivars	Origin country	Bread-making quality	Specific characteristics
CORVUS	Germany	+	Low sensitivity for the Hagberg falling number
FOLIO	England	+	High sensitivity for the Hagberg falling number
ORDÉAL	France	–	Late developer with low sensitivity for diseases, very low specific viscosity
AGAMI	England	---	High yield, high specific viscosity
APACHE	France	++	Early maturing plants
BUCCANEER	England	---	Low specific weight, medium specific viscosity
DREAM	Germany	+++	Late developer, high baking quality
MERCURY	Germany	++	Hybrid variety
MEUNIER	France	+++	Low sensitivity for the Hagberg falling number, high sensitivity for diseases
KOCH	Netherlands	---	High yield, low sensitivity for diseases

+++, the highest bread-making quality; ---, the lowest bread-making quality.

October 2001, 11 October 2002 and 17 October 2003, and harvesting dates were 14 August 2002, 4–5 August 2003 and 23 August 2004. The same growing conditions were applied for each cultivar: standard nitrogen fertilization applied under solid form as  $\text{NH}_4\text{NO}_3$  (40–65–75 kg/ha, respectively, for the tillering, the beginning of stem elongation and the flag leaf fractions) and two fungicide protections (first epoxyconazol 125 g/ha and secondly azoxystrobin 250 g/ha + metconazole 60 g/ha). Each sample was grown in four different plots of 16 m<sup>2</sup> in a fully randomized block design. Wheat grains from four different plots were mixed to reduce location effect and to increase the homogeneity of the samples.

## 2.2. Starch isolation

Wheat grains were milled using a Quadrumat Senior mill (Brabender, Duisberg, Germany) after adjusting moisture content to 15.5% on a dry basis. A same grains sample was regularly milled to control the milling conditions.

Starch was isolated from white flours using a modified version of the batter procedure (Roels, Sindic, Deroanne, & Delcour, 1998; Sindic, Chevalier, Duculot, Foucart, & Deroanne, 1993). White flour (2.0 kg) was mixed with 60% (flour hydration capacity basis) water for 2 min in a mixer A200 (Hobart, Troy, Ohio) equipped with a dough hook. The resulting dough was allowed to rest for 8 min. Water (2.0 L) was then added, and the dough was stirred with a flat beater (Hobart, Troy, Ohio) for 25 min. The mixture was transferred into a vessel with 10.0 L of water. The diluted suspension was then stirred continuously for 35 min to agglomerate the gluten. The mixture and about 10.0 L of water were then pumped over vibrating sieves with decreasing pore size (400, 250, 125, 90 and 50  $\mu\text{m}$ ). The sieves retained gluten and fibres and starch was contained in the filtrate. The starch suspension was allowed to rest for 24 h at 4 °C, and then the supernatant was decanted. After the centrifugation for 10 min at 3000g in a Beckman J2-21 centrifuge (Beckman, Fullerton, CA), the supernatant was again decanted. The sediment consisted of a yellow-brown layer of sludge fraction with a starch layer underneath. The top layer was scraped off and the white bottom layer was re-suspended in water. The suspension was then centrifuged a second time, the top layer was scraped off and the starch layer was re-suspended in water and centrifuged a third time. The starch (about 1.5 kg) was then collected, frozen and freeze-dried. The freeze-drying is preferred to air-drying with a subsequent grinding because higher starch damage was obtained for air-dried starch isolates (Grant, 1998).

## 2.3. Analytical methods

Yields of starch extraction were expressed on a dry matter basis as percentage of the total starch content of flour.

*Moisture contents* of the samples (flours and starches) were determined as weight loss of 5.0 g accurately weighed after 165 min at 130 °C (ISO 712:1998).

*Water absorption* (%) was evaluated using a farinograph (Brabender, Duisberg, Germany) according to the approved method 54-21 (AACC, 1983).

*Damaged starch* values were determined amperometrically by the Chopin SD4 method, based on the absorption kinetics of iodine.

*Starch contents* were measured polarimetrically by the Ewers method (ISO 10520:1997).

## 2.4. Granule size distribution of starch

Particle size characteristics of starches were determined by using a Malvern mastersizer 2000, equipped with Malvern application software version 3.20 (Malvern Instruments Inc., Worcestershire, UK). Accuracy of the instrument was checked with Malvern standard glass particles. The instrument is based on the principle of laser light scattering and capable of measuring sizes between 0.1 and 2000  $\mu\text{m}$ . The size distribution is expressed in terms of the volumes of equivalent spheres. The criteria selected are the percentage volume (% vol.) of granules with a diameter lower than 10  $\mu\text{m}$  and the parameters  $d(0,1)$ ,  $d(0,5)$  and  $d(0,9)$  expressed in micrometers. The criterion  $d(0,1)$  ( $d(0,5)$  or  $d(0,9)$ ) means that 10% (50% or 90%) of the particles have a diameter lower than this criterion.

## 2.5. Amylose determination

The apparent amylose content was determined by a modified iodometric method (Morrison & Laignelet, 1983). Prime starch (100 mg) was dispersed in 10 mL of urea-dimethyl sulfoxide solution (UDMSO) and heated for 60–90 min in a boiling water bath. A 100  $\mu\text{L}$  aliquot of starch-UDMSO solution was diluted 100-fold and mixed with 200  $\mu\text{L}$  of  $\text{I}_2$ -KI solution. Apparent amylose content was evaluated from absorbance at 635 nm. The recorded values were converted to percent of amylose by reference to a standard curve prepared with amylose from potato and amylopectin from corn (ICN Biochemicals Inc., Aurora, OH).

## 2.6. Viscosity analysis

Starch viscosity behaviour was evaluated with a Micro Visco-amylograph (Brabender, Duisberg, Germany). Starch suspensions (10% dry matter, total weight 100.0 g) were subjected to the following time–temperature profile: equilibration at 30 °C, a linear temperature increase to 95 °C in 10 min, holding at 95 °C for 10 min, a cooling step (10 min) with a linear temperature decrease to 50 °C and a final isothermal step at 50 °C (5 min). 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), setback 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) were determined and expressed in BU (Brabender units).

Whole flour pasting properties were determined by the Rapid Visco Analyser (RVA) '4-Series viscosimeters' (Newport Scientific Pty. Ltd., Narrabeen, Australia) following the ICC method No. 162. Wheat grains were milled using a Cyclotec model 1093 sample mill (0.5 mm) (Foss Tecator, Amersfoort, Netherlands). 4.0 g (14% moisture basis) was added to a disposable aluminium canister containing 25.0 g accurately weighed distilled water at 20 °C, mixed and placed in an RVA heating block. A disposable plastic stirrer was used to mix the suspension continuously at 160 rpm during the measurement. A Thermocline software program controlled the heating and cooling cycles. The programmed cycle was held at 50 °C for 1 min, ramped to 95 °C at the rate of 12 °C/min, held at 95 °C for 5 min, ramped to 50 °C at the rate of 12 °C/min, and held at 50 °C for 1 min. The peak pasting, final, breakdown, and setback viscosities were recorded and expressed in cP (Centipoise units).

Viscosity measurements (starch and whole flour) were performed in water and with addition of 2 mM AgNO<sub>3</sub> to nullify  $\alpha$ -amylase effects and facilitate comparisons between cultivars (Abdel-Aal, Hucl, Chibbar, Han, & Demeke, 2002; Bason, Ronalds, Wrigley, & Hubbard, 1993; Batey, Curtin, & Moore, 1997; Bhattacharya & Corke, 1996; Crosbie & Lambe, 1993).

## 2.7. Statistical analysis

All analyses were carried out in triplicate. Statistical analyses were performed using Minitab software (version 13.20, Minitab Inc., State College, PA) for correlation analysis and ANOVA. Student test ( $P = 0.05$ ) was performed to determine least significant differences (LSD).

## 3. Results and discussion

### 3.1. Wheat flours and starch extraction

Wheat flour properties and starch extraction characteristics are given in Table 2. With the same milling conditions, water absorption of flours varied from 47.5% (Agami 02) to 63.0% (Folio 04). For the three growing seasons, Dream and Folio showed the highest starch damages and Agami had the lowest values. For all cultivars, the starch damages were lower for 2002 and higher for 2004, indicating an influence of the cultivar but also of the harvest year on this characteristic. Starch damages were correlated with water absorption of flours ( $R = 0.85$ ,  $P < 0.001$ ,  $n = 26$ ), and varied from 13.2 (Agami 02) to 22.1 CDU (Folio 04). The level of the damage varies with the severity of grinding and the hardness of the wheat (Hoseney, 1994). Damaged starch granules hydrate rapidly and are susceptible

to enzymatic hydrolysis (Ranhotra, Gelroth, & Eisenbraun, 1993).

Total starch content in white flours, expressed on dry basis, ranged from 79.9% to 84.6%. Starch was isolated from wheat flour by 'Batter' extraction with purity always higher than 97% (expressed on dry basis). Starch yields, expressed as a percentage of the starch content in the white flour, ranged from 70% to 83%. The lower starch yields were associated with Agami, Buccaneer and Koch, which had a low bread-making quality. For these three cultivars, the gluten agglomeration was more difficult during starch isolation and a part of the starch associated with the protein fraction may have been lost.

### 3.2. Granule size distribution of starch

Granules size distributions of the wheat starches are given in Table 3. Contribution of the B-granules population ( $<10 \mu\text{m}$ ) to the total volume ranged from 11.6% to 29.9% among 26 samples. The comparison of the three harvest seasons showed globally the same observations. Agami had the lowest percentage by volume of small granules and Dream and Corvus had the highest percentage. The criterion  $d(0,1)$  confirmed this observation: 10% of the starch particles had a diameter lower than 3.2–3.4  $\mu\text{m}$  for Dream and Corvus whereas Agami showed a  $d(0,1)$  value of 6.7, 7.1 and 7.9. The criteria  $d(0,5)$  and  $d(0,9)$  varied from 15.9 to 21.1 and 31.8 to 37.2, respectively. As would be expected, the lower values were associated with Dream and Corvus cultivar, and the higher values with Agami cultivar. The B-granules contents were globally higher for 2002, indicating an influence of the harvest year on this parameter. This result could be associated with the lower values of starch damages for 2002. Dodds (1971) suggested that larger granules were more likely to be damaged during milling or grinding.

The relative proportions of the starch A- and B-granules in the wheat cultivars may result in differences in chemical composition that affect the functionality of the starch. For example starch granule size has been related to the pasting properties of starch, rheological properties of dough, baking characteristics and compositional differences (Peng et al., 1999). The starch granule size distribution is potentially an important index of wheat quality. Raeker et al. (1998) found a correlation between high amylose content and a high proportion of A-type granules. The larger granules were reported by Meredith (1981) to be more susceptible to the action of  $\alpha$ -amylases than the smaller granules. It also has been suggested that smaller granules were more resistant to external influences and less inclined to transformation (Fortuna, Januszewska, Juszczak, Kielski, & Palasinski, 2000). Small granules were also reported as containing more fatty acids, phospholipids and lysophospholipids and more surface proteins in comparison to large granules (Soulaka & Morrison, 1985). These differences could result in the two starch granule types being utilized differently, both in food and non-food uses (Ellis et al.,

Table 2  
Flour water absorption and starch characteristics of wheat cultivars

Cultivars	Flour water absorption (%)	Starch content in flour (%)	Starch damage (CDU)	Starch purity (%)	Starch yield (% starch)
Corvus 2002	54.5	84.5	16.4	98.8	79
Corvus 2003	53.0	81.9	17.3	98.6	79
Corvus 2004	54.5	83.7	18.3	98.7	80
Folio 2002	59.0	83.2	18.6	99.6	76
Folio 2003	59.5	81.3	19.9	99.3	79
Folio 2004	63.0	80.7	22.1	97.0	79
Ordéal 2002	54.5	80.9	16.9	97.6	83
Ordéal 2003	55.0	82.9	17.9	98.1	77
Agami 2002	47.5	84.6	13.2	99.0	70
Agami 2003	53.0	82.8	–	97.3	79
Agami 2004	49.5	83.2	15.9	97.3	80
Apache 2002	52.5	83.2	15.2	99.3	76
Apache 2003	56.0	81.4	17.1	97.0	76
Buccaneer 2002	56.5	84.3	17.4	98.1	73
Buccaneer 2003	54.5	82.7	17.2	97.4	80
Dream 2002	56.5	82.7	18.6	99.6	82
Dream 2003	56.0	79.9	19.2	97.8	82
Dream 2004	56.5	82.9	20.7	97.0	79
Mercury 2002	56.0	83.2	17.2	99.5	78
Mercury 2003	55.0	83.3	17.2	99.0	81
Mercury 2004	56.5	83.4	19.1	98.8	81
Meunier 2002	53.5	82.2	15.3	99.6	82
Meunier 2003	56.0	83.1	17.2	98.2	78
Meunier 2004	57.0	81.2	18.8	97.1	76
Koch 2003	50.5	83.4	16.6	98.3	81
Koch 2004	51.8	81.9	17.1	97.4	72
LSD	2.7	0.9	1.4	0.6	–

Table 3  
Starch granule characteristics and apparent amylose content of 26 isolated starches from wheat

Cultivars	Granules <10 $\mu\text{m}$ (% vol.)	$d(0,1)$ ( $\mu\text{m}$ )	$d(0,5)$ ( $\mu\text{m}$ )	$d(0,9)$ ( $\mu\text{m}$ )	Apparent amylose (%)
Corvus 2002	28.6	3.4	16.7	32.9	25.4
Corvus 2003	26.6	3.4	17.5	33.7	26.7
Corvus 2004	26.0	3.4	17.6	33.3	26.9
Folio 2002	24.2	3.9	17.3	32.9	26.0
Folio 2003	23.0	4.1	18.0	34.3	28.4
Folio 2004	26.2	3.7	16.9	33.0	27.1
Ordéal 2002	19.7	4.3	18.8	35.1	25.6
Ordéal 2003	15.5	6.2	19.7	35.7	27.7
Agami 2002	14.3	6.7	20.4	36.8	27.0
Agami 2003	11.6	7.9	21.1	37.2	27.5
Agami 2004	15.3	7.1	20.6	36.5	27.5
Apache 2002	20.5	4.4	18.1	33.6	25.2
Apache 2003	21.1	4.3	18.2	33.9	27.7
Buccaneer 2002	18.1	5.2	19.8	37.0	26.1
Buccaneer 2003	17.3	5.1	20.0	36.7	27.3
Dream 2002	29.9	3.2	15.9	31.8	26.7
Dream 2003	26.5	3.4	16.9	32.4	28.1
Dream 2004	24.0	3.4	17.5	31.9	28.4
Mercury 2002	23.7	3.9	18.1	34.6	25.9
Mercury 2003	22.1	4.0	18.5	35.0	27.2
Mercury 2004	23.0	4.1	18.1	34.6	27.1
Meunier 2002	19.0	4.3	19.4	35.9	27.0
Meunier 2003	17.0	4.5	19.8	35.9	27.9
Meunier 2004	23.2	4.4	18.0	34.9	27.9
Koch 2003	26.0	4.4	17.1	34.0	26.9
Koch 2004	25.1	4.6	17.0	33.4	27.3
LSD	4.6	1.3	1.3	1.6	0.6



1998). In actual fact, there is little separation of A- and B-granule types on a commercial basis. Enriched fractions are sometimes prepared, but this is on a limited scale.

### 3.3. Apparent amylose

Apparent amylose contents of the starches (with lipids presence) are given in Table 3. Amylose content varied from 25.2% to 27.0% for 2002 cultivars, 26.7% to 28.4% for 2003 cultivars and from 26.9% to 28.4% for 2004 cultivars. The measured values were globally lower for 2002, related to the higher proportion of B-granules measured for this harvest year and confirming that amylose content was higher in large granule (Raeker et al., 1998; Soulaka & Morrison, 1985). Among cultivars of a same year, the range was small and no significant difference in amylose content was found.

Colonna and Buléon (1992) reported typical levels of amylose and amylopectin are, respectively, 25–28% and 72–75%. The relative amounts of amylose and amylopectin are known to influence both nutritional and technological properties such as susceptibility to enzymatic hydrolysis, gelling and pasting behaviour, which could be of biotechnological importance (Gérard et al., 2001). For example, high amylose starches are widely used as thickeners and strong gelling agents but present the retrogradation problem. The introduction of amylopectin improves the uniformity, stability and texture of gelatinized starches. It also imparts better freeze-thaw stability in frozen foods (Slatery et al., 2000). Zeng, Morris, Batey, and Wrigley (1997) linked higher peak paste viscosity, greater breakdown, and lower final viscosity of wheat starch to lower amylose contents. Black, Panozzo, Wright, and Lim (2000) reported that the peak viscosity and final viscosity are positively correlated and a negative correlation is found between final viscosity and amylose content for the land-race population. Because of the small range observed, all these relationships could not be established in this study.

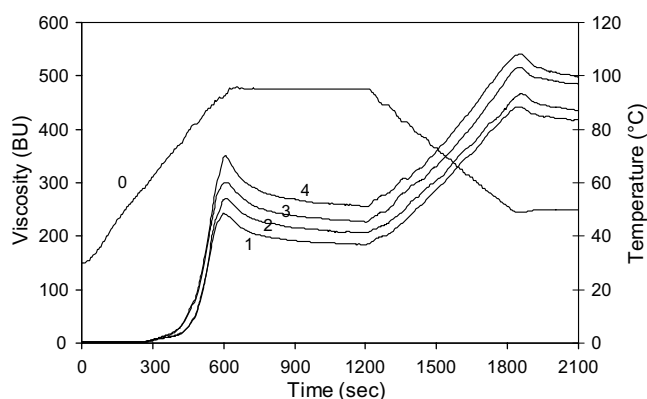


Fig. 1. Brabender viscosity of starches isolated from wheat as response to the applied temperature program (line 0): Dream starches without (line 1) or with (line 2) enzymes inactivation and Agami starches without (line 3) or with (line 4) enzymes inactivation, harvest 2003.

### 3.4. Viscosity behaviour

Visco-amylograms of wheat starches with and without prior enzymes inactivation are presented in Fig. 1. The pasting temperature was approximately 75 °C. The starch granule swells to several times its initial size, ruptures and simultaneously amylose leaches out from inside the granules; a rapid rise in viscosity occurs. For a same sample, the peak viscosity of the starch was higher when the enzymes were inactivated with AgNO<sub>3</sub>. When starch was heated in excess water during viscosity characterization, aqueous solution and high temperature were very good conditions for raw starch degradation by amylase activity. Kim, Nanmori, and Shninke (1989) reported high degradation of wheat and potato starch granules at 60 °C. Slaught-ter, Ellis, and Butterworth (2001) found the rate of amylolysis of native and heat-treated starch suspensions is essentially linear for up to 2 h but the rate over the first 60 s or so is often higher than over the remaining time course. Various chemical treatments have been reported to inhibit amylase activity and consequently improve the pasting profile of sprout-damaged wheat, e.g., HCl followed by neutralization with NaOH, AgNO<sub>3</sub>, HgCl<sub>2</sub>, cycloheptaamylose, and EDTA (Abdel-Aal et al., 2002; Bason et al., 1993; Batey et al., 1997; Bhattacharya & Corke, 1996; Crosbie & Lambe, 1993). Silver nitrate was used in this study as  $\alpha$ -amylase inhibitor to verify the absence of endogenous amylases in extracted starches and characterize the pasting properties of starch without enzyme interference. The high differences observed for the peak viscosity with and without enzymes inactivation, confirmed the presence of enzymes in the extracted starches. Therefore, these results showed the many washing steps during the starch isolation procedure were not sufficient to completely eliminate amylase activity.

The whole viscosity behaviours of the wheat starches are summarized in Table 4. Comparison of the three growing seasons showed the same observations. The peak viscosity (the maximum viscosity during pasting) varied from 160.3 to 321.0 BU and from 276.5 to 351.5 BU, respectively, without and with amylases inactivation. Agami and Meunier had the highest peak viscosity whereas Dream and Mercury had the lowest values in presence of silver nitrate. As expected, the higher differences for the peak viscosity with and without enzyme inactivation were measured for 2002, a harvest year with serious sprouting problems, especially for Mercury ( $\Delta$  peak of 130.8 BU), a cultivar with high sensitivity of sprouting.

The breakdown viscosity (the difference between the peak viscosity and the viscosity at the end of the holding phase) was used to examine the response of starches to shear thinning. With enzyme inhibitor, breakdown was greatest for Agami and Meunier and least for Dream. The height of the maximal peak 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

Table 4  
Pasting and gelatinization properties of isolated starches from wheat (in Brabender units)

Cultivars	Measurements in water				Measurements in water + 2 mM AgNO <sub>3</sub>				$\Delta$ peak viscosity (2) – (1)
	Peak visc. (1)	Final visc.	Breakd. visc.	Setb. visc.	Peak visc. (2)	Final visc.	Breakd. visc.	Setb. visc.	
Corvus 2002	272.3	465.0	60.7	253.3	307.7	502.0	68.3	262.7	35.4
Corvus 2003	286.0	487.0	58.5	259.5	298.0	478.5	73.5	254.0	12.0
Corvus 2004	294.5	519.5	54.0	279.0	297.0	502.0	57.0	262.0	2.5
Folio 2002	231.3	393.3	85.0	247.0	311.0	492.0	80.7	261.7	79.7
Folio 2003	275.0	472.5	64.0	261.6	298.5	484.5	71.5	257.5	23.5
Folio 2004	250.0	462.0	43.0	255.0	287.5	487.5	57.0	257.0	37.5
Ordéal 2002	235.3	416.7	60.7	242.0	312.7	512.3	65.7	265.3	77.4
Ordéal 2003	275.0	485.0	51.5	261.5	300.0	492.0	68.0	260.0	25.0
Agami 2002	254.7	404.7	89.3	239.3	343.0	535.3	82.3	274.7	88.3
Agami 2003	301.5	495.5	74.5	268.5	351.5	524.0	96.0	268.5	50.0
Agami 2004	300.5	527.0	72.5	279.0	345.7	562.0	79.3	275.7	45.2
Apache 2002	239.0	376.0	87.3	224.3	325.3	476.3	89.7	250.7	86.3
Apache 2003	259.5	463.5	55.5	259.5	311.0	509.0	67.0	265.0	51.5
Buccaneer 2002	265.0	453.7	68.7	257.3	322.7	520.7	70.0	268.0	57.7
Buccaneer 2003	264.0	477.0	50.0	263.0	296.5	489.5	65.0	258.0	32.5
Dream 2002	233.0	428.7	47.0	242.7	283.0	483.3	53.7	254.0	50.0
Dream 2003	242.0	428.5	57.5	244.0	282.5	459.0	57.0	243.5	40.5
Dream 2004	242.0	462.0	34.0	254.0	281.0	477.5	47.0	243.5	39.0
Mercury 2002	160.3	253.0	53.3	166.0	291.1	450.3	60.3	239.7	130.8
Mercury 2003	237.0	424.0	56.5	253.5	276.5	453.0	59.5	246.0	39.5
Mercury 2004	239.0	446.5	46.5	254.0	282.5	490.5	52.0	260.0	43.5
Meunier 2002	321.0	516.3	85.3	280.7	345.7	532.3	89.0	275.7	24.7
Meunier 2003	301.5	494.5	74.5	267.5	324.5	506.5	82.5	264.5	23.0
Meunier 2004	294.0	539.0	70.5	285.5	321.5	539.5	78.5	266.5	27.5
Koch 2003	260.5	437.0	71.5	253.0	284.0	437.5	76.0	234.5	23.5
Koch 2004	288.5	493.5	62.5	262.5	319.5	518.0	73.5	267.0	31.0
LSD	29.9	47.7	19.8	19.7	20.1	25.6	12.0	9.8	–

cooking and hence exhibit a significant viscosity decrease after reaching the maximum value (Singh, Singh, Kaur, Sodhi, & Gill, 2003). As expected among the samples, Agami and Meunier showed breakdown values (with AgNO<sub>3</sub>) higher than Dream and Mercury.

The last portion of the Brabender pasting curve measures the increase in viscosity associated with gelation and retrogradation during cooling. The setback viscosity represents the difference between the viscosity at the end of cooling and the viscosity at the end of the holding phase whereas the final viscosity was the viscosity at

the end of cooling and reflected the network formation. In presence of silver nitrate, setback and final viscosity were higher for Agami and Meunier starches, and lower for Dream and Mercury. Sahlström et al. (2003) noted correlations between samples with a high percentage of A-type granules and an increase in high peak viscosity, breakdown viscosity, setback viscosity and final viscosity. In this study, the higher negative correlations between percentage of B-type granules and viscosity parameters were observed in presence of  $\alpha$ -amylase inhibitor (Table 5).

Table 5  
Correlation coefficients between percentage of B-type granules and starch viscosity parameters with and without  $\alpha$ -amylases inhibitor

%B-granules		Viscosity properties in water				Viscosity properties with AgNO <sub>3</sub>			
		PV 1	FV 1	Bkd 1	Setb 1	PV 2	FV 2	Bkd 2	Setb 2
%B-granules	–								
PV 1	–0.30ns	–							
FV 1	–0.16ns	0.91***	–						
Bkd 1	–0.28ns	0.11ns	–0.29ns	–					
Setb 1	–0.17ns	0.91***	0.98***	–0.19ns	–				
PV 2	–0.63***	0.55**	0.25ns	0.64***	0.27ns	–			
FV 2	–0.53**	0.64***	0.58**	0.06ns	0.57**	0.75***	–		
Bkd 2	–0.35ns	0.18ns	–0.21ns	0.94***	–0.15ns	0.67***	0.04ns	–	
Setb 2	–0.48*	0.68***	0.61**	0.14ns	0.62**	0.70***	0.94***	0.10ns	–

ns, not significantly; \*\*\*,\*\*,\* =  $P < 0.05, 0.01, 0.001$ , respectively,  $n = 26$ .

Table 6  
Pasting and gelatinization properties of wheat whole flours (in cP units)

Cultivars	Measurements in water				Measurements in water + 2 mM AgNO <sub>3</sub>				$\Delta$ peak viscosity (2) – (1)
	Peak visc. (1)	Final visc.	Breakd. visc.	Setb. visc.	Peak visc. (2)	Final visc.	Breakd. visc.	Setb. visc.	
Corvus 2002	1972	2020	995	1043	3723	3996	1450	1723	1751
Corvus 2003	2305	2730	905	1330	3391	3862	1281	1752	1087
Corvus 2004	2185	2700	818	1333	3081	3811	1030	1759	897
Folio 2002	505	182	414	91	3363	3566	1301	1505	2858
Folio 2003	1976	2400	758	1181	2904	3559	984	1638	928
Folio 2004	1120	1157	621	657	2481	3169	772	1460	1353
Ordéal 2002	776	447	596	268	3329	3819	1145	1635	2554
Ordéal 2003	1898	2249	829	1080	3163	3795	1073	1705	1265
Agami 2002	456	103	397	44	3989	4235	1528	1779	3534
Agami 2003	2437	2605	1127	1295	3860	4347	1449	1936	1423
Agami 2004	681	371	549	239	3576	4365	1177	1965	2896
Apache 2002	865	419	698	253	3689	3717	1523	1551	2825
Apache 2003	1687	1744	854	912	3476	3978	1265	1767	1789
Buccaneer 2002	980	659	725	405	3538	3960	1238	1660	2559
Buccaneer 2003	2319	2657	933	1271	3267	3861	1096	1690	948
Dream 2002	1439	1475	783	820	3040	3675	1040	1675	1601
Dream 2003	2377	2931	897	1452	2981	3539	1092	1649	605
Dream 2004	1268	1477	612	772	2302	3067	799	1564	1034
Mercury 2002	479	175	420	116	3286	3548	1303	1565	2807
Mercury 2003	1793	1859	887	953	3208	3633	1180	1605	1415
Mercury 2004	809	645	555	391	2699	3377	886	1564	1890
Meunier 2002	2397	2448	1148	1200	4052	4188	1549	1685	1655
Meunier 2003	2728	3085	1049	1406	3613	3993	1344	1724	885
Meunier 2004	2585	3385	716	1516	3064	3998	881	1815	429
Koch 2003	2178	2116	1055	993	3271	3261	1407	1397	1093
Koch 2004	2112	2390	944	1221	2824	3316	1118	1610	712
LSD	484	666	152	307	280	280	135	109	–

Pasting properties of wheat whole flours are summarized in Table 6. Results of the three growing seasons showed differences in the viscosity parameters. High differences between the viscosity with and without enzyme inactivation were measured, especially for 2002. During this year, serious sprouting damages were observed. Without AgNO<sub>3</sub> addition, Folio, Agami and Mercury showed very low parameters of viscosity. As expected, the values increased with enzyme inactivation and were in the viscosity range of the other cultivars.

The peak viscosity ranged from 456 to 2728 cP and from 2302 to 4052 cP, respectively, without and with amylases inactivation. Similarly to the starch measurements, Agami and Meunier had the highest peak viscosity whereas Dream and Mercury had lower values in presence of AgNO<sub>3</sub>. Agami and Meunier showed also the highest final viscosity

whereas Dream and Mercury were associated with lower values (with AgNO<sub>3</sub>). In 2002, the Agami cultivar showed the lowest peak viscosity without enzyme inactivation but also one of the highest peak viscosities with enzyme inactivation. This result underlined the important impact of amylase activity on the pasting profile of sprout-damaged wheat. With enzyme inhibitor, a little decrease of peak viscosity was also observed between the harvest years (2002 > 2003 > 2004), indicating a slight impact of the growing season on this characteristic.

In this study, the relationships between the pasting properties of whole flour and starch were studied (Table 7). For the three growing seasons, good correlations were observed between these two matrixes for the peak viscosity and the final viscosity with enzyme inactivation (respectively,  $R = 0.74$  and  $0.70$ ,  $P < 0.001$ ,  $n = 26$ ). To integrate

Table 7  
Correlation coefficients between viscosity parameters of starch and whole flour, with and without  $\alpha$ -amylases inhibitor

	Viscosity properties in water				Viscosity properties with AgNO <sub>3</sub>			
	PV	FV	Bkd	Setb	PV	FV	Bkd	Setb
2002, 2003, 2004 ( $n = 26$ )	0.64***	0.63***	0.07ns	0.57**	0.74***	0.70***	0.87***	0.65***
2002 ( $n = 9$ )	0.73*	0.72*	−0.24ns	0.58**	0.91***	0.88**	0.84**	0.61*
2003 ( $n = 10$ )	0.57ns	0.41ns	0.69*	0.11ns	0.82**	0.91***	0.79**	0.87***
2004 ( $n = 7$ )	0.52ns	0.54ns	0.54ns	0.46ns	0.86**	0.89**	0.71*	0.72*

ns, not significantly; \*\*\*,\*\*\* =  $P < 0.05$ ,  $0.01$ ,  $0.001$ , respectively.



the impact of the harvest year, the correlation coefficients were also calculated for each growing season and better relationships were then noted. From this last observation, we concluded that a viscosity measurement on whole flour could be used to rapidly estimate the starch viscosity behaviour without the laborious isolation step. These results confirmed also the important influence of starch in the viscosity development of whole flour. The correlation coefficients were relatively moderate; this could be explained by the multiple interactions of starch with other wheat components in whole flour (proteins, lipids, non-starch polysaccharides (NSP), ...). For example, the formation of starch–lipid complexes has been shown to affect gelatinization and pasting properties of wheat starch (Morrison, 1988). NSP is assumed to interact with other components and influence the physical properties and end-product quality of wheat flour (Sasaki, Kohyama, & Yasui, 2004).

In addition, pasting properties of starch were traditionally studied using the Brabender Amylograph whereas the RVA was used for measuring whole flour pasting properties. Deffenbaugh and Walker (1989) suggested indeed that the results from the two instruments are similar but not interchangeable.

#### 4. Conclusions

Actually, in many countries, the wheat arriving at a mill is not of one variety or from a single production area; instead it is a blend of varieties grown on many farms and in many environments. As new varieties with specific end-use characteristics are developed, released, and grown, preserving the varietal identity of wheat (i.e., keeping it segregated by variety) will become more important.

In most wheat-based products, flour exerts a major effect on their quality. Until now, wheat quality is associated with characteristics like yield and protein content but starch is never considered as a quality criterion. However, starch is added as a functional ingredient to many food products and the European wheat starch industry has been rapidly growing for the past 20 years. For the production of flour corresponds to the best starch application, it is important that the end users evaluate also the starch quality.

In this study, starch was extracted with a high purity and the results showed isolated starch properties were principally influenced by the wheat cultivar and slightly by the culture year. Between the different studied cultivars, variations of 20% in starch viscosity and 60% in B-granules population were observed. To make high-quality products without encountering processing or end-product quality problems, a thorough working knowledge of all aspects of wheat and its flour, but also of its starch, is required. The selection of an adapted wheat cultivar could lead to different starch properties, which are interesting to valorize in well-selected end uses, with most cost-competitive production. For example, Agami, a cultivar with very low

bread-making quality, showed a starch with the highest viscosity properties among the ten studied cultivars. Agami could then not use in the production of breads and related dough-based products but its starch could be valorized in suitable applications as a native ingredient for the food or non-food industry. Finally, a good correlations is found between the pasting properties of whole flour and starch, with  $\alpha$ -amylase inactivation. Therefore, it is apparent the viscosity parameters of whole flour were strongly influenced by the intrinsic properties of starch. Recently considered like inert filler, the starch can be a substantial, active and thus quality-determining part of the final product.

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