

# IN-LINE MEASUREMENT OF THE VISCOSITY BEHAVIOUR OF WHEAT STARCH DURING EXTRUSION. APPLICATION TO STARCH CATIONISATION

F. BERZIN\*, A. TARA, L. TIGHZERT

CERME – Ecole Supérieure d'Ingénieurs en Emballage et Conditionnement,  
Esplanade Roland Garros, BP 1029, 51686 Reims Cedex 2, France

\*E-mail: francoise.berzin@univ-reims.fr  
Fax: x33 3.26913803

Received: 24.7.2006, Final version: 6.10.2006

## ABSTRACT:

A specific twin channel slit die was used to measure in-line the viscous behaviour of an extruded wheat starch. This allows to put in evidence the influences of temperature, water content and specific mechanical energy (SME). The proposed rheological law permits to satisfactorily predict the viscosity of a wheat starch for any processing condition. Original results are presented for the behaviour of cationic starches obtained by reactive extrusion.

## ZUSAMMENFASSUNG:

Ein spezieller Doppelwellenextruder wurde verwendet, um das Extrusionsverhalten von Weizenstärke on-line zu erfassen. Der Einfluss der Temperatur, des Wassergehalt und des Energieeintrages auf das Extrudat kann somit ermittelt werden. Die vorgeschlagenen rheologischen Gesetze erlauben eine ausreichende Viskositätsbestimmung für Weizenstärke bei allen Prozessbedingungen. Ergebnisse für das Fließverhalten von modifizierter Stärke unter Reaktionsextrusion werden desweiteren vorgestellt.

## RÉSUMÉ:

Une filière plate à double canaux est utilisée pour mesurer en ligne le comportement rhéologique d'un amidon de blé extrudé. Ce système permet de mettre en évidence les influences respectives de la température, de la teneur en eau et de l'énergie spécifique. La loi de comportement proposée permet de prédire de manière satisfaisante le comportement d'un amidon de blé, pour des conditions de transformation données. Pour la première fois, des mesures sont présentées pour des amidons cationiques, obtenus par extrusion réactive.

**KEY WORDS:** wheat starch, slit die, twin screw extrusion, specific mechanical energy, cationisation

## 1 INTRODUCTION

Starch is the main storage substance synthesized by plants from solar energy. It can be found in numerous species: cereals, seeds, tubercles, etc. In non-food applications, starch is generally used after hydrothermal or chemical transformations. New applications are rapidly developing, mainly as replacing synthetic polymers, for example in the field of packaging materials. Starch is very often modified by chemical and enzymatic treatments, by depolymerization or cross-linking, or by esterification or etherification reactions based on the free hydroxyl groups. A wide range of modified starches can then be prepared, according to the desired properties [1].

In preceding papers [2 - 4], we have studied the cationization of wheat starch by reactive extrusion, using a co-rotating twin screw extruder. Cationic starches are mainly used in paper industry, as coating agents, ingredients in paper pulp or surfacing agents [5]. One of the purposes of this work was to build a numerical model of the reactive extrusion process, able to predict the progress of the reaction along the screws and the final degree of substitution of the cationic starches. In order to achieve this objective, it is necessary to know the viscous behaviour of the molten starch inside the extruder, with and without reaction. This is the subject of the present paper.

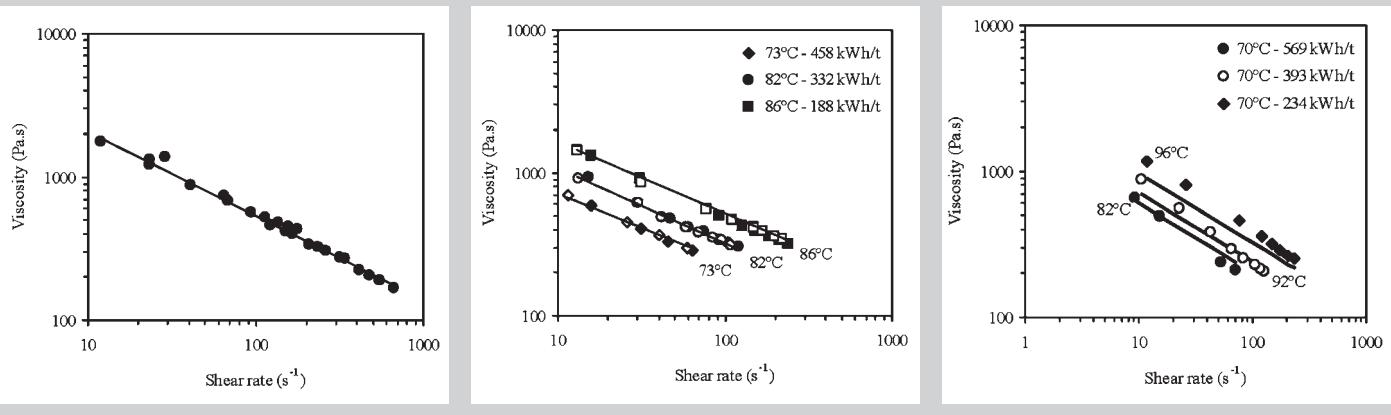
© Appl. Rheol. 17 (2007) 21222-1 – 21222-7

This is an extract of the complete reprint-pdf, available at the Applied Rheology website  
<http://www.appliedrheology.org>

This is an extract of the complete reprint-pdf, available at the Applied Rheology website  
<http://www.appliedrheology.org>

Applied Rheology  
Volume 17 · Issue 2

21222-1



**Figure 9 (left):**  
Mastercurve of viscosity, for three different water contents, after rescaling at 90°C and 200 kWh/t.

**Figure 10 (middle):**  
Comparison of viscosities for products extruded in the same conditions, without (full symbols) and with (open symbols) cationization reaction. Temperatures indicated on the curves are die exit temperatures for starch without and with reaction.

**Figure 11 (right):**  
Comparison of viscosities calculated from Eq. 8 (full lines) and measured with slit die (symbols) for products extruded with the screw profile 2. Temperatures indicated on the curves are die exit temperatures.

Eq. 8 can be used for the simulation of the cationization process using a twin screw extruder, both for the native and the cationized starch, whatever the processing conditions and the reagent concentrations.

To validate Eq. 8, we have performed experiments on the second screw profile. We have measured the viscosity of starches extruded under different conditions and, using Eq. 8 with the previously defined data, we have calculated the theoretical viscosities. The screw profile being more restrictive, SME is higher for same N/Q ratios (see Figure 3) and consequently viscosities are lower than for screw profile 1. Moreover, the temperatures measured at die exit are higher, due to a higher viscous dissipation. Figure 11 shows that, even if the agreement is not perfect for all conditions (the maximum error is around 28%), the proposed model allows a good evaluation of the wheat starch viscosity, including the main parameters of the extrusion process (water content, specific energy, temperature).

## 5 CONCLUSION

Starting from published data, we have adapted a twin channel slit die to a laboratory twin screw extruder, in order to measure in-line the viscous behaviour of a molten wheat starch, modified or not by a cationization reaction. The results confirm the general form of the rheological law for these materials, with a strong dependence not only on temperature and water content, but also on specific mechanical energy, directly responsible of starch transformation. We have defined the parameters of this law for a wheat starch and we have shown its applicability, at least in the explored experimental window. Finally, we have shown that, for the DS range that we explored, the cationization reaction has no effect on the rheological behaviour of the molten starch.

## ACKNOWLEDGMENTS

This work was funded by Région Champagne-Ardenne (Conseil Général de la Marne, France) through the research program Amival, devoted to the starch valorization for non-food applications.

## REFERENCES

- [1] Tomasik P, Wang YJ, Ames JJ: Facile route to anionic starches. Succinylation, maleination and phtalation of corn starch on extrusion, *Starch/Stärke* 47 (1995) 96-99.
- [2] Tara A, Berzin F, Tighzert L, Vergnes B: Preparation of cationic wheat starch by twin screw reactive extrusion, *J. Appl. Polym. Sci.* 93 (2004) 201-208.
- [3] Ayoub A, Berzin F, Tighzert L, Bliard C: Kinetic study of the thermoplastic wheat starch cationisation reaction under molten condition, *Starch/Stärke* 56 (2004) 513-519.
- [4] Berzin F, Tara A, Tighzert L, Vergnes B: Computation of starch cationization performances by twin screw extrusion, *Polym. Eng. Sci.* 47 (2007) 112-119.
- [5] Miladinov VD, Hanna MA: Starch esterification by reactive extrusion, *Industrial Crops and Products*, Elsevier 11 (2000) 51-57.
- [6] Barron C, Della Valle G, Colonna P, Vergnes B: Energy balance of low hydrated starch transition under shear, *J. Food Sci.* 67 (2002) 1-12.
- [7] Vergnes B, Villemaire JP: Rheological behaviour of low moisture molten maize starch, *Rheol. Acta* 26 (1987) 570-576.
- [8] Fletcher SI, McMaster TJ, Richmond P, Smith AC: Rheology and extrusion of maize grits, *Chem. Eng. Commun.* 32 (1985) 239-262.
- [9] Senouci A, Smith AC: An experimental study of food melt rheology. I: Shear viscosity using a slit die viscometer and capillary rheometer, *Rheol. Acta* 27 (1988) 546-554.
- [10] Altomare RE, Anelich M, Rakos R: An experimental investigation of the rheology of rice flour dough with an extruder-coupled slit die rheometer, In: Kokini JL, Ho CT, Karwe MV Eds., *Food Extrusion, Science and Technology*, Marcel Dekker, New York (1992).
- [11] Brouillat-Fourmann S, Carrot C, Mignard N, Prochazka F: On the use of an internal mixer for the

This is an extract of the complete reprint-pdf, available at the Applied Rheology website  
<http://www.appliedrheology.org>

- rheological characterization of maize starch, *Appl. Rheol.* 12 (2002) 192-199.
- [12] Roos H, Bolmstedt U, Axelsson A: Evaluation of new methods and measuring systems for characterisation of flow behaviour of complex foods, *Appl. Rheol.* 16 (2006) 19-25.
- [13] Vergnes B, Villemaire JP, Colonna P, Tayeb J: Inter-relationships between thermo-mechanical treatment and macromolecular degradation of maize starch in a novel rheometer with preshearing, *J. Cereal Sci.* 5 (1987) 189-207.
- [14] Vergnes B, Della Valle G, Colonna P: Rheological properties of biopolymers and applications to cereal processing, In: Kaletunc G, Breslauer KJ Eds., Characterization of Cereal and Flours; Properties, Analysis and Application, Marcel Dekker, New York (2003).
- [15] Parker R, Ollett AL, Lai Fook RA, Smith AC: The rheology of food melts and its application to extrusion processing, In: Carter RE Ed., Rheology of Food, Pharmaceutical and Biological Materials, Elsevier, London (1990).
- [16] Vergnes B, Della Valle G, Tayeb J: A specific in-line rheometer for extruded starchy products. Design, validation and application to maize starch, *Rheol. Acta* 32 (1993) 465-476.
- [17] Della Valle G, Boché Y, Colonna P, Vergnes B: The extrusion behavior of potato starch, *Carbohydr. Polym.* 28 (1995) 255-264.
- [18] Della Valle G, Colonna P, Patria A, Vergnes B: Influence of amylose content on the viscous behavior of low hydrated molten starches, *J. Rheol.* 40 (1996) 347-362.
- [19] Della Valle G, Vergnes B, Colonna P, Patria A: Relations between rheological properties of molten starches and their expansion behaviour in extrusion, *J. Food Eng.* 31 (1997) 277-296.
- [20] Della Valle G, Buléon A, Carreau PJ, Lavoie PA, Vergnes B: Relationships between structure and viscoelastic behavior of plasticized starch, *J. Rheol.* 42 (1998) 507-525.
- [21] Banks W, Greenwood CT. Starch and its Components. Edinburgh University Press, (1975).
- [22] Agassant JF, Avenas P, Sergent JP, Vergnes B, Vincent M: La mise en forme des matières plastiques, Lavoisier, Tec & Doc, Paris (1996).
- [23] Della Valle G, Koslowski A, Colonna P, Tayeb J: Starch transformation estimated by the energy balance of a twin-screw extruder, *Lebensm. Wiss. u-Technol.* 22 (1989) 279-286.
- [24] Guy RCE, Horne AW: Extrusion and coextrusion of cereals, In: Blanshard JMV, Mitchell JR Eds., Food Structure. Its Creation and Evaluation, Butterworths, Londres, (1988).
- [25] Vergnes B, Della Valle G: Propriétés thermophysiques et rhéologiques des substrats utilisés en cuisson-extrusion, In: La cuisson-extrusion, Lavoisier, Tec & Doc, Paris, (1994).
- [26] Willett JL, Jasberg BK, Swanson CL: Rheology of thermoplastic starch: effects of temperature, moisture content and additives on melt viscosity, *Polym. Eng. Sci.* 35 (1995) 202-210.



This is an extract of the complete reprint-pdf, available at the Applied Rheology website  
<http://www.appliedrheology.org>

This is an extract of the complete reprint-pdf, available at the Applied Rheology website  
<http://www.appliedrheology.org>

Applied Rheology  
 Volume 17 · Issue 2

2122-7