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A multiple-step slit die rheometer for rheological characterization of extruded starch melts

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ABSTRACT

Extrusion cooking is a very important process in the field of cereal and snack manufacturing. The rheological properties of the starch based matrix strongly influence this process. A newly developed online rheometer was mounted on a twin screw extruder in order to measure these properties. It was possible to obtain viscosity of wheat flour and corn grits at typical extrusion cooking conditions over a shear rate range of three decades. Flow curves for varying screw speed and water content at a constant thermomechanical history of the starch were measured. Also the temperature dependence of the apparent viscosities could be determined showing activation energies in the range found for synthetic polymers. Furthermore, additional pressure drops that occur at step changes of the slit height were detected. Thus, it was possible to evaluate visco-elastic properties of extruded starch melts at different extrusion cooking conditions.

1. Introduction

Today, many cereals and snacks are manufactured by the process of extrusion cooking. Sensorial properties, e.g. taste, crunchiness, crispness and 'mouth feel' of these products as well as rehydration rate of extrusion-cooked, ground instant powders highly depend on bulk expansion and the resulting porous structure (Meuser et al., 1982). The pressure drop at the extrusion die leads to the formation of vapour bubble nuclei, which then grow inside the plasticized starch matrix and significantly expand the product, as described in the review by Moraru and Kokini (2003). As the melt cools down and water evaporates outside the die, the melt solidifies which finally ends bubble growth and stabilizes the extruded starch based foams.

The vapour pressure of the fluid is the driving force of the nucleation and the bubble growth (Mao et al., 2006). The nucleation of bubbles starts when the vapour pressure of the fluid is reached inside the extruder die. Therefore, nucleation is determined by the pressure profile in the die, which in turn depends on the viscosity of the melt and the die geometry (Han, 1974). Since the bubble growth is governed by strain and extension of the surrounding matrix, elastic properties have an effect on the expansion of extruded products (Della Valle et al., 1997). Thus, knowing the viscous as well as the elastic behaviour of the plasticized starch matrices is of critical relevance in understanding and controlling expansion of extruded melts at extruder dies.

In contrast to synthetic polymer melts, the bio-polymer starch is very susceptible to molecular degradation due to high mechanical stresses or temperatures like they occur in an extrusion cooking process (van den Einde et al., 2004a). Nevertheless, such a thermo-mechanical treatment of starch is crucial to increase its digestibility through an increased gelatinization (Meuser et al., 1982). Thus, it is necessary to measure the rheological properties of starch at the exact extrusion process conditions.

Several investigations are related to the development of devices for the measurement of rheological properties of starch melts at extrusion conditions. While it still remains challenging to achieve the processing conditions of extrusion cooking with an offline approach (van den Einde et al., 2004b), online rheometry, on the other hand, is proposed to be the most accurate way to measure such properties (Steffe, 1996). Capillary and slit die rheometers mounted at the outlet of extruders have been widely used to measure the viscosities of plasticized starch matrices online. Slit die geometries are mostly preferred for online measurements, since flush-mounted pressure transducers allow measuring the pressure directly inside the flow channel without any disturbance of the flow. In consequence, the additional pressure loss at the die inlet, the well-known Bagley-pressure (Bagley, 1961), caused mainly by elastic but also by viscous properties, can be neglected. Thus,

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shear rate $\dot{\gamma}$, shear stress τ and, finally, the apparent viscosity η_{app} can be easily calculated by the measured pressure drop Δp at a certain volumetric flow rate $\dot{V}$ according to Eqs. (1)–(3). 'Apparent viscosity' in the relation means that there are some assumptions made in order to calculate the shear rate, especially Newtonian behaviour of the fluid (Pahl et al., 1991).

$$\dot{\gamma} = \frac{6 \cdot \dot{V}}{H^2 \cdot W} \tag{1}$$

$$\tau = \frac{\Delta p \cdot H}{2 \cdot L} \tag{2}$$

$$\eta_{app} = \frac{\tau}{\dot{\gamma}} \tag{3}$$

H and W are the height and width of the slit geometry, respectively. The length L defines the distance between two pressure measurement sites.

Twin-channel slit die rheometers, as designed by e.g. Vergnes et al. (1993), enable control of the flow in each channel separately by piston valves. However, the necessity for accurate measurement of the flow rate in each channel is a disadvantage of twin-channel rheometers. Therefore, single channel rheometers were used despite their lack of control over the backpressure in the extruder (Bindzus et al., 2002). The effort has been made to overcome that drawback by implementing a by-pass flow before the entrance to the single channel rheometer (Li et al., 2005). This, however, leads to the problem on quantifying the flow rate as in twin-channel rheometers described above. Hence, the necessity of a good control over the backpressure and thus over the thermo-mechanical history of the product while changing shear rates in a wide range is still a challenge in starch melt rheology.

In this work a modular single channel online rheometer was applied for the measurement of various rheological properties of molten starch-based matrices (i.e. corn grits and wheat flour). The rheometer had a modular design, with exchangeable inner geometry by using different slide-in modules. Therefore, this rheometer met the requirements for reliable measurements of a variety of rheological properties of plasticized starch melt with one device. First, the backpressure, which is a key parameter for starch structure, could be influenced by changing of either length or height of the flow channel. Secondly, a large shear rate range was covered (1–2000 s^{-1}) by changing the slit height. Furthermore, a multiple-step geometry enabled measuring the viscosity at three different shear rates during one experiment, assuring a flow curve at a constant thermo-mechanical history of the processed material.

2. Materials and methods

A co-rotating twin screw extruder (*Coperion Werner & Pfleiderer ZSK 26Mc*) with a screw diameter of 25.5 mm was used for the extrusion trials. With this new generation extruder, a very high screw speed range of 180–1800 rpm was accessible. The extruder barrel was divided into 7 sections with an overall length of 29 D. During the experiments, each barrel section, except the first one, was heated separately to 60 °C, 80 °C, 100 °C, 100 °C, 100 °C, and 100 °C, respectively.

As feed raw materials, commercially available wheat flour type 405 and corn grits (both delivered by *BÄKO*, Germany) with average particle sizes (median) of 85 μm and 350 μm, respectively, were used. The raw material specifications were available from a data sheet of the manufacturer. Starch content in wheat flour was 69% Wb and 58% Wb in corn grits. Protein content was at about 12% for wheat flour and 8% for corn grits.

The raw materials were fed into the first barrel by a gravimetrically controlled feeder (*Brabender DDW-DDSR 40*) which ensured constant feed rates between 5 kg/h and 75 kg/h. By using the gravimetric feeder, the feed rate could be controlled independently of screw speed, which was important for changing the screw speed of the extruder while maintaining a constant flow rate and therefore also a constant shear rate inside the rheometer. This way it was possible to observe the change of material properties due to higher mechanical energy input and therefore also higher shear stresses inside the extruder. For calculation of volumetric flow rate $\dot{V}$ a material density of 1450 kg/m^3 was used, which was determined by measuring the density of extruded and ground products by a helium gas pycnometer (*Quantachrom GmbH*). The measured value was also in accordance to literature values (Millauer, 1994).

Water was fed into the second extruder barrel and mixed with the raw material inside the extruder. The total moisture content was varied from 17% Wb to 30% Wb. The backpressure and the product temperature were measured in the last section of the extruder directly in front of the rheometer inlet.

The rheometer was mounted at the extruder outlet and heated by an electric heating jacket. It consisted of a single flow channel and had an overall length of 370 mm. The inner flow channel geometry had a height of 25 mm and width of 15 mm. By applying specially designed insertion modules of different sizes, different inner geometries of the flow channel could be implemented; therefore, several tasks could be performed in one device:

- Multiple-step flow channel for multiple viscosity and elasticity measurements at once (Fig. 1).
- Short flow channel for viscosity measurements at high shear rates (<2000 s^{-1})
- Long flow channel for more accurate viscosity measurements at lower shear rates (>4 s^{-1})

The rheometer had 9 sensor sites which could be equipped either with pressure or temperature sensors. In this work, 6 flush-mounted pressure sensors (*Gefran melt pressure sensor*, max. pressure 2 × 500 bar, 2 × 200 bar, 2 × 100 bar) were used and located according to the used inner geometry. Two pressure sensors were applied on one constant slit height to obtain viscosity data. The product temperature was measured on one site in each experiment.

All data was recorded at equilibrium of the process determined by constant backpressure, torque and temperature inside the extruder and repeated 2 times.

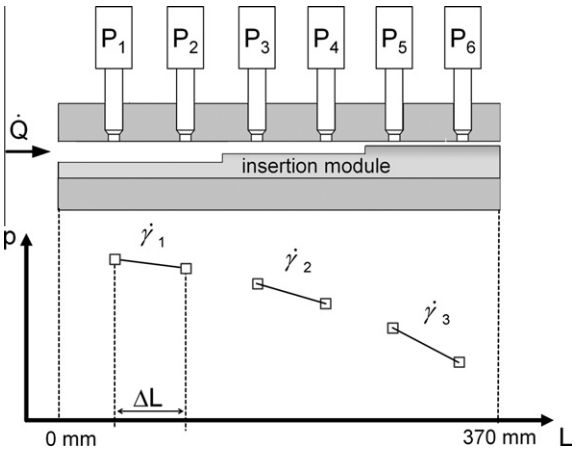

Fig. 1. Slit-die rheometer with a triple-step insertion module generating three different shear rates in the flow channel. Two flush-mounted pressure sensors are applied on each stage to measure pressure drop.

3. Results and discussion

First, trials were conducted to determine the accuracy and reproducibility of the measurements. Fig. 2a shows the pressure values measured in the flow channel at a constant slit height as a function of time for extruded corn grits at one parameter setup. The values for other conditions as well as for wheat flour are not shown but correspond to these qualitatively. Although the fluctuation of the values is increasing with increasing pressure, the resulting standard deviation is lower than 10% (mostly between 2% and 5%) in all experiments. For calculation of shear stress, the average pressure value over a time range of minimum 80 s was taken. In Fig. 2b, typical calculated shear stresses are shown as a function of shear rate for different extrusion parameters. In order to change shear rate at constant screw speed and mass flow rate, the slit height was varied. The error bars in the graph contain the deviation of pressure fluctuation as well as of two extrusion experiments. The calculated shear stresses have a standard deviation of 10% maximum, although in most cases it is about 5%.

In Fig. 3, a linear pressure decrease measured by 6 pressure transducers in the rheometer at different process parameters is illustrated. Due to the linear decrease of pressure, first, it can be assumed that the length between the slit die entrance and the first pressure sensor is chosen long enough to achieve a fully developed flow. Secondly, it shows that there is no change in starch viscosity during its flow in the rheometer. This leads to the assumption that the molecular degradation of the starch is completed before the entrance into the rheometer. Starch-based materials, such as corn grits and wheat flour consist of two major fractions of glucose-based molecules. There are linear amylose chains, with a molecular weight of about 10^2–10^5 Da, and highly-branched amylopectin molecules, with a molecular weight of about 10^5–10^7 Da (Bertoft, 2005). During the extrusion process, amylopectin molecules in particular are degraded by thermal and mechanical stresses (i.e. shear and elongational stresses) (van den Einde et al., 2004a,b; Igura et al., 1997; Steffe, 1996). However, van den Einde et al. (2004b) found that molecular degradation mainly depends on the maximum shear stress applied. We assume, that the maximum shear stress was reached inside the extruder. Further molecular degradation inside the rheometer would result in a continuous change of the viscosity (Della Valle et al., 1997), and thus in the pressure drop along the rheometer. However, no bended pressure curve was observed in Fig. 3.

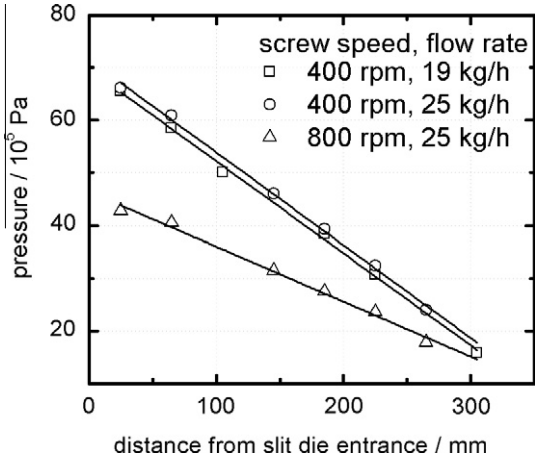

Fig. 3. Linear pressure loss inside the slit die rheometer at different extrusion parameters. Slit height $H = 4$ mm, moisture content 30%, shear rate (□) $\dot{\gamma} = 100\ s^{-1}$, (○,△) $\dot{\gamma} = 132\ s^{-1}$.

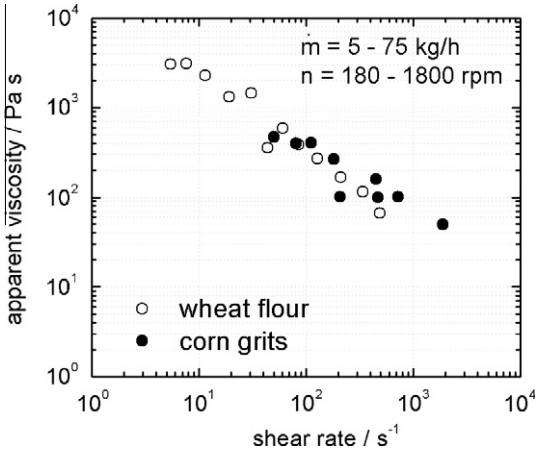

Fig. 4. Viscosities of extruded corn grits and wheat flour at different extrusion conditions to achieve a wide range of shear rates.

Fig. 4 shows the range within which viscosity data was determined. With the interchangeable inner geometry of the rheometer

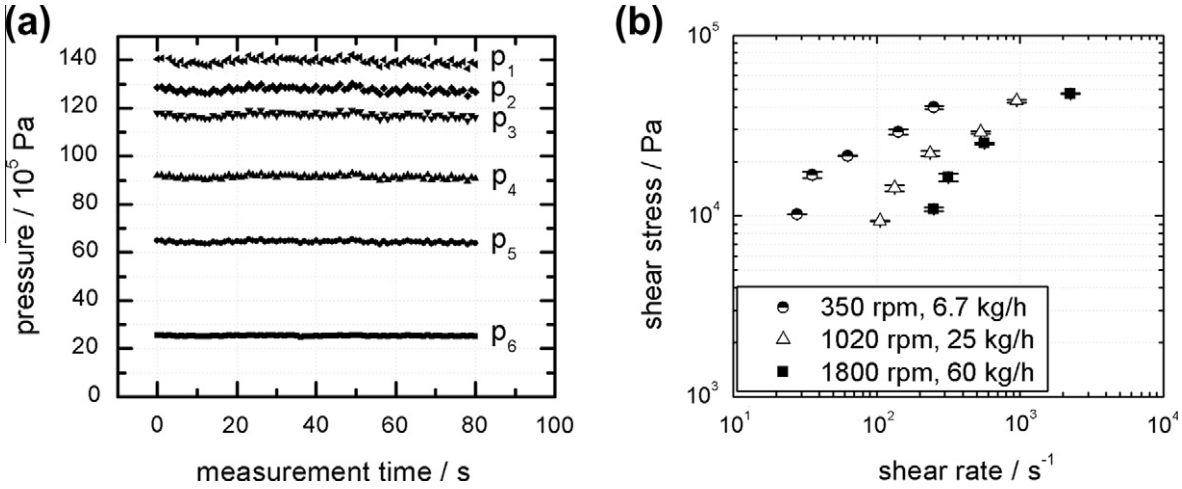

Fig. 2. (a) Measured pressure values inside the rheometer at constant extrusion parameters and constant slit height of 4 mm. (b) Measured shear stresses as function of shear rate at varying extrusion parameters.

it was possible to measure viscosity values over a shear rate range of three orders of magnitude. Nontheless, it must be noted, that in this experiment, extrusion parameters (incl. mechanical energy input) were changed to achieve a wide range of shear rates. Thus, molecular degradation may have happened to a different degree and the data shown may not be interpreted as flow curves. Further reduction of the slit height enables the measurement of shear rates up to $10^4\ s^{-1}$, which is in the range of shear rates applied within the extruder as well as in extrusion dies. Since the backpressure increases drastically with decreasing slit height, the slit length has to be adjusted (decreased) such that the backpressure is kept in a technically relevant range. Common extrusion cooking processes are performed at backpressures up to 200 bar. Superior control on backpressure generally is offered by twin-channel rheometers only. They enable direct control of the flow in each channel by piston valves as shown by Vergnes et al. (1993). The authors pointed out that controlling the flow in a slit die rheometer helps to maintain the backpressure in the extruder and thus avoids changes in the thermomechanical history of the starch. The approach of maintaining backpressure can be even improved by making the slit channel heights adjustable during the process, as proposed e.g. by Robin et al. (2010). In order to increase the shear rate in one channel, the slit height can be decreased. However, at the same time the slit height of the second channel has to be increased – to maintain overall backpressure – which, in turn, leads to a decreased flow rate in the first channel and therefore smaller shear rates (max. $30\ s^{-1}$). Hence, due to the modular design of the single channel rheometer used in our study, we are able to control backpressure and also apply shear rates higher than in twin-channel rheometers. The modular design even offers the possibility to mount additional circular dies at the end. This way, expansion of cylindrical products can be observed and evaluated in the same experiment, thus, ensuring identical thermo-mechanical history of the molten starch.

In contrast to experiments shown in Fig. 4, multiple-step geometry enabled to measure flow curves at constant extruder conditions (Fig. 5). The flow curve with 3 viscosity values was measured at once applying an inner geometry with 3 different slit heights of 10 mm, 5 mm and 3 mm, respectively. The results indicate a shear thinning behaviour of plasticized wheat flour, as also reported by Wang et al. (1990). Furthermore, the influence of different water contents and also screw speeds applied (i.e. variation of mechanical energy input) is depicted. Higher water content led to lower viscosities, due to the plasticizing effect of water (Fig. 5a). By increasing screw speed the viscosity also decreased (Fig. 5b). Higher screw speed generally increases the specific mechanical energy input, which enhances starch degradation. It is well established that smaller polymer molecules with lower molecular weight show lower viscosity (Fox and Flory, 1948; Schaefgen and Flory, 1948). However, the impact of screw speed which has been varied between 300 rpm and 800 rpm on the viscosity was not as significant as the effect of water which has been increased from 18% up to 30%. Therefore, the plasticizing effect of water on the viscosity was more pronounced than the impact of molecular degradation in the range of the parameters applied in our study. Nonetheless, the viscosity can be altered by variation of either screw speed or water content (Della Valle et al., 1997). Increased screw speed leads to higher mechanical stresses inside the extruder. Especially for snacks and breakfast cereals high mechanical stresses are not desirable, since product properties such as taste and crispness are impaired by dextrin formed during molecular degradation. Hence, manipulation of viscosity for these products is performed via water content favourably. However, in a manufacturing process excessive water addition can have an adverse effect on the expansion (Della Valle et al., 1997) limiting the control of product characteristics by variation of water content to a certain extent.

In addition to viscosity, elastic properties were investigated in our study. The data depicted in Fig. 5a and b were obtained using channels with heights of 10 mm, 5 mm and 3 mm corresponding to height ratios of 2 and 1.6, respectively. Under these conditions no pressure drop at the channel edges indicating elastic effects was observed, presumably due to the low contraction ratio. Therefore, in a further experiment the rheometer was equipped with a two-step inner geometry with heights of 10 mm and 1.5 mm (ratio 6.7). The additional pressure drop was evaluated by linear extrapolation of the measured pressure values to the location of the slit height change. An additional pressure drop of about 5 bar was observed as shown in Fig. 6. This pressure drop is assumed to be related to elongational properties of the extruded starch similar to the "Bagley-pressure" or entrance pressure drop in cylindrical dies (Cogswell, 1972). By extrapolating the measured pressure values to the outlet of the rheometer at the length of 370 mm, the exit pressure was determined to be 6.5 bar. For this quantity a correlation to the first normal stress difference N_1 has been suggested in the literature (Padmanabhan and Bhattacharya, 1991).

With the introduced modular rheometer, it was also possible to measure the viscosity of starch based melts as a function of tem-

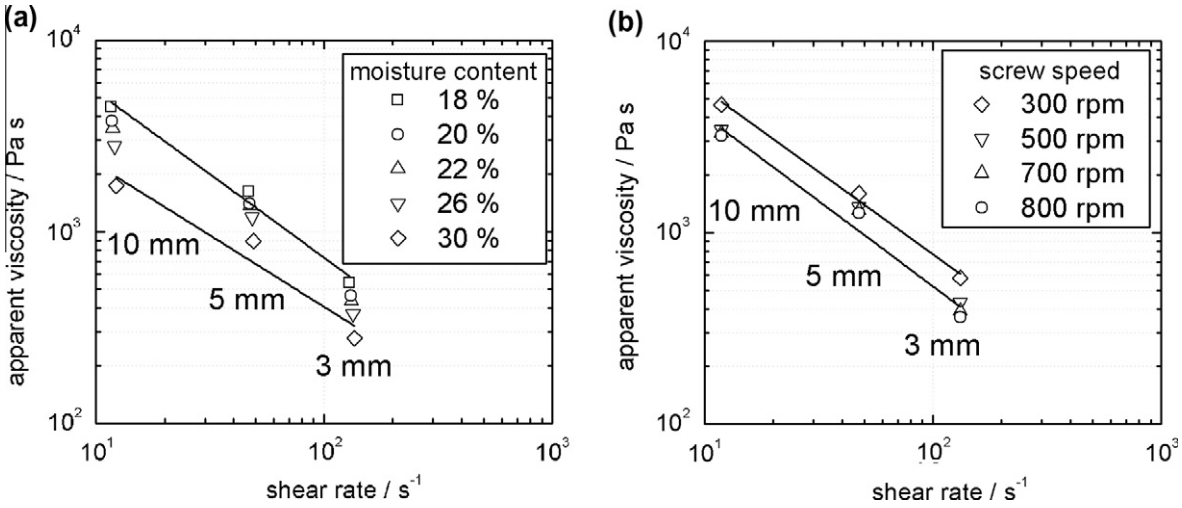

Fig. 5. Viscosity curves of wheat flour at varying moisture content in (a) and screw speed in (b). The corresponding slit heights are shown below the data points.

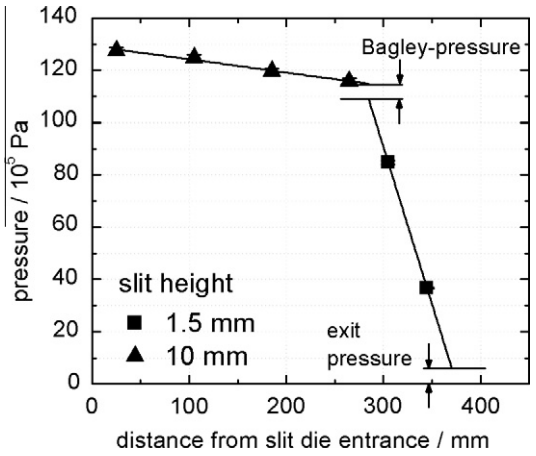

Fig. 6. Pressure drop along the slit including the step change in height from 10 to 1.5 mm.

perature. Therefore, extrusion trials at two different parameter sets were performed, first a screw speed of 560 min^{-1} with a flow rate of 10 kg/h and secondly a higher screw speed of 1020 min^{-1} with a flow rate of 25 kg/h. During each trial, the extrusion parameters were kept constant and the temperature in the rheometer was changed by changing the temperature of the heating jacket. The lowest possible measurement temperature was the temperature of the material leaving the extruder at the specified extrusion cooking conditions. For the two parameter sets described above, these lowest material temperatures were 130 °C and 152 °C, respectively. The temperature in the rheometer then was varied on three levels between this lowest value and 170 °C. When equilibrium in pressure and temperature was reached, pressure data were recorded to calculate the viscosities which are shown in Fig. 7. An Arrhenius-type equation (Eq. (4)) was applied to describe these data:

$$\eta = \eta_{ref} \cdot \exp\left[\frac{E_A}{R_G \cdot T}\right] \tag{4}$$

In Eq. (4), η_{ref} is a reference viscosity at a reference temperature, R_G is the ideal gas constant and T the actual temperature. E_A is called the flow activation energy, which is a material property characterising the temperature dependence of the viscosity. The range of calculated activation energies shown in Fig. 7 is within the range of the values for some synthetic polymers, such as polyethylene or polybutadiene, reported in literature (Wang and Porter, 1995). The shear rates were varied by changing either the flow rate in the extruder or the slit height in the rheometer. E_A seems to change at varying shear rates or extrusion conditions, which might be due to different degradation and therefore different chain length and branching of starch molecules. However, since the experimental error for the apparent viscosity measurement is about 5–10%, error propagation results in an experimental uncertainty for the activation energy E_A of about 20%. Thus, the activation energies determined in our study under different experimental conditions have to be treated as equal within the experimental error and we specify an average value $E_{A,mean}$ = 25.5 kJ/mol.

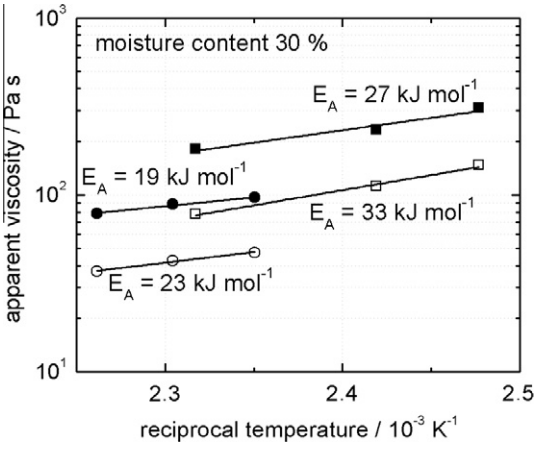

Fig. 7. Temperature dependence of viscosity at different screw speeds and flow rates (shear rates). At slit height H = 4 mm, shear rate (■) $\dot{\gamma}$ = 53 s^{-1}, (●) $\dot{\gamma}$ = 143 s^{-1}, at slit height H = 2 mm, (□) $\dot{\gamma}$ = 211 s^{-1}, (○) $\dot{\gamma}$ = 535 s^{-1}.

4. Conclusions

An online slit die rheometer was attached to the exit of a high-speed twin-screw extruder to measure rheological properties of plasticized starch based melts, like wheat flour and corn grits. With the interchangeable insertion module, the inner geometry of the slit die could be designed such that backpressure and therefore thermomechanical history of the starch melt could be kept constant during measurements of the rheological properties.

The accuracy as well as the reproducibility of the measured data were determined experimentally. The error of the calculated shear stresses based on two extrusion experiments was well below 10%. The viscosity of the starch based melt was measured at extrusion cooking conditions over a shear rate range of 3 decades (up to 2000 s^{-1}). In the range of shear rates applied in literature with slit rheometers of different geometries the corresponding viscosity values in our study showed good agreement with the results from previous studies. Using a multiple-step geometry in the rheometer, additional pressure drops, which occur upon step changes of the slit height, were also detected. Thus, it was possible to evaluate the viscosity function and a measure of the elastic properties of a starch based melt online at the same time.

The temperature dependence of viscosity was also measured and could be described by an Arrhenius-type equation. Due to the experimental error a relation of the activation energies to process parameters or shear rate could not be proven. However, the calculated average activation energy E_A = 25.5 kJ/mol was in the same range as that of well-known synthetic polymers.

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