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Rheological properties of starch and kaolin mixtures in water during and after heating

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Abstract

Gelatinization characteristics of the starch and kaolin mixtures were investigated with the Brabender viscograph at starch concentration of 40 g/l and different kaolin concentrations from 0 to 60 g/l. The properties of starch gelatinization were affected by the addition of kaolin. Above a kaolin concentration of 12 g/l had, an additional viscosity peak was seen at 73 °C. Further increase of kaolin concentration up to 60g/l led to an increase is the additional peak at 73 °C, while the value of maximum viscosity peak decreased.

The rheological features of gelatinized starch and kaolin mixtures were also investigated. Dependence of shear stress on shear rate was obtained with the use of Haake RS 100 rheometer with Z 38 AL sensor. Shear rate was first increased from zero to 200 s^{-1} and then decreased from 200 s^{-1} to zero within 400 s period. Those measurements were obtained at the 20, 40, 60 and 70 °C for the mixtures containing 10 or 20 g/l of starch and for kaolin concentrations ranging from 0 to 15 g/l. The results showed the independence between final viscosity and kaolin concentration in the mixtures. Viscosity was affected only by the temperature of the measurment and the starch concentration (10 or 20 g/l).

A mathematical model for the rheological behavior of gelatinized starch, was developed which contains the number and volume of macromolecule aggregates as well as the number of bonds between aggregates. According to this model, the bonds energy between the aggregates reached the value of 2–8 kJ/mol, which seemed to be close to the Van der Waals bonds energy.

Keywords: Starch kaolin mixtures; Gelatinization characteristics; Rheological properties; Mathematical model

1. Introduction

Rheological behavior of gelatinized starch is very important for the application of starch and its derivatives in food industry and many other areas. Therefore, the mechanism of starch gelatinization as well as the influence of the conditions of this process on viscosity and other rheological properties of water–starch mixtures have been investigated for many years.

The process of starch gelatinization is affected by many different organic and inorganic substances dissolved in water starch mixtures. Numerous previous studies have shown the dependence of the temperture of starch gelatinization and viscosity of gelatinized starch on the additon of saccharides (Abu-Jdayil, Azzam & Al-Malah, 2001; Beleia, Miller & Hoseney, 1996; Gudmundsson, Eliasson, Bengtsson, & Aman, 1991; Richardson, Langton, Bark & Hermansson, 2003), emulsifiers (Richardson, et al., 2003), surfactants (Harbitz, 1983) and inorganic substances (Sajeev, Moorthy, Kailaggan & Rani, 2003; Valles-Pamies et al., 1997).

The investigation of rheological properties of the mixtures of starch and insoluble inorganic substances (of submicronic sizes) in water are important for ceramic processing (Bonomi, Persay, & Schleiffer, 1989; Dmitriev, Podkovyrkin, Beloborodova, Kleshcheva & Gorskya 1990; Fagnelli, Silvers, Frei, Burlew & Marsh, 1989; Shanefield, 1995). Water suspensions of alumina powder in the presence of maltodextrin shows a high degree of clay-like plasticity (Goel, Schilling, Biner, Moore & Lograsse, 1996; Schilling, Biner, Goel, & Jane, 1995; Schilling, Tomasik, & Kim, 1999).

In the presence of non-spherical inorganic particles, the relation between the particles and polysaccharides depends

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on the kind of material, and particle charge and it increases when the particle concentration rises (Chow, 1980; Izak & Cioch, 2002; Kitano, Kataoka & Shirota, 1981; Metzner, 1985). The effect of the addition of clay substances on rheological properties of gelatinized starch is still not well understood. Investigations mentioned above very important for ceramic processing and obtaining biodegradable materials containing starch and kaolin (Carvalho, Curvelo & Agnelli, 2001).

The aim of this work was investigation of the process of starch gelatnisation in the presence of kaolin and rheological properties of the mixtures of gelatinized starch with kaolin.

2. Materials and methods

2.1. Materials

The materials involved commercial potato starch produced by Starch Factory Niechlow in 2001 and industrial kaolin trademark KOWS produced by Surmin-Kaolin with average diameter of 50% granules in population 11 μm containing 51.3% SiO_2, 34.3% Al_2O_3, and 0.5% Fe_2O_3 and $<0.8\%$ water.

2.2. Gelatinization characteristics

Gelatinization characteristics were investigated with Brabender viscograph equipped with a 700 cmg cartridge. The measuring tube was filled with 450 ml of water dispersion containing 40 g/l starch and 0–60 g/l kaolin. These mixtures were hydrated at 40 °C for 10 min at the constant rotation (75 rpm), then heated with the rate 1. 5 °C/min up to 94 °C and held at that temperature for 10 min. Afterwards the mixtures were cooled with the rate 1.5 °C/min up to 30 °C and held at that temperature for 10 min. All samples were measured in triplicate. The deviation between the repetitions was ±20 BU for viscosity and ±0.2 °C for temperature.

2.3. Microscopic investigations

The suspension of starch or the mixtures containing 40 g/l starch and 40 g/l kaolin were heated up to 63 and 73 °C. The samples of those dispersions were diluted by distilled water and placed on basic glass of microscope (‘BIOPOLAR B’ produced by PZO Warsaw) to prepare the microphotographs.

2.4. Investigation of rheological properties of the mixtures of gelatinized starch with kaolin

Mixtures containing 10 or 20 g/l starch and from zero to 15 g/l kaolin in water were heated in the water bath untill the starch gelatinized. Lump formation was prevented by constant stirring (70 rpm). Then the samples were boiled for the following 5 min. Afterwards the mixtures were cooled and the evaporated water was completed with distilled water. The samples were held 12 h at room temperature before undertaking viscosity investigations.

Dependence of shear stress on shear rate was obtained using reometer Haake RS 100 by using a co-axial cylinder system (diameters of cylinders: 38 and 43 mm) with the sensor Z38AL. Shear rate was changed from zero to 200 s^{-1} and from 200 to zero s^{-1} within 400 s (one cycle) at the temperature 20 °C.

Additionally, the samples containing 20 g/l of starch and 4 g/l kaolin were investigated at 20, 40 60 and 70 °C. The samples containing 20 g/l of starch and 2 g/l of kaolin were investigated only at the temperature of 20 °C but for 5 shear rate cycles.

Each analysis was performed in triplicate and the deviation of individual replication was ±20%.

3. Results and discussion

The addition of kaolin affected the process of starch gelatinization. As shown in Fig. 1 the samples of potato starch and starch–kaolin mixtures, with the addition of kaolin to concentration lower than 12 g/l gave typical 2-peak curve of gelatinization characteristics: the first peak of maximum viscosity was observed during heating (at the temperature of 92–94 °C), while the second during cooling (at 30 °C). The addition of 12 g/l or higher concentration of kaolin caused the emergence of an additional third viscosity peak at a temperature of approximately 73 °C. The additional peak increases with increasing kaolin concentration from 12 to 32 g/l, a further increase in kaolin concentration between 32 and 56 g/l did not affect the peak of viscosity, which rises again when the kaolin concentration reaches 60 g/l (Table 1). At the same time, maximum viscosity peak at 94 °C noticeably decreases from 830 to 670 BU with the change of kaolin concentration from 0 to 4 g/l, slightly decreases for 6–56 g/l and slightly rises at 60 g/l of kaolin concentration. Similar viscosity changes are observed after 10 min at 94 °C (Table 1).

Decrease in the viscosity of mixtures containing up to 4 g/l of kaolin in comparison to the samples of starch suspension is probably caused by interaction of kaolin particles with unfolded chains of gelatinized starch. Kaolin particles have electrostatic charge and in low kaolin concentrations it could be responsible for the decrease of gel viscosity in a similar manner to that of the addition of electrolytes (Rueggeberg, 1953; Sajeev et al., 2003).

At kaolin concentration higher than 4 g/l interactions between kaolin particles and swollen starch granules can occur.

Dramatic increase of viscosity at temperature higher than initial pasting temperature is connected with both

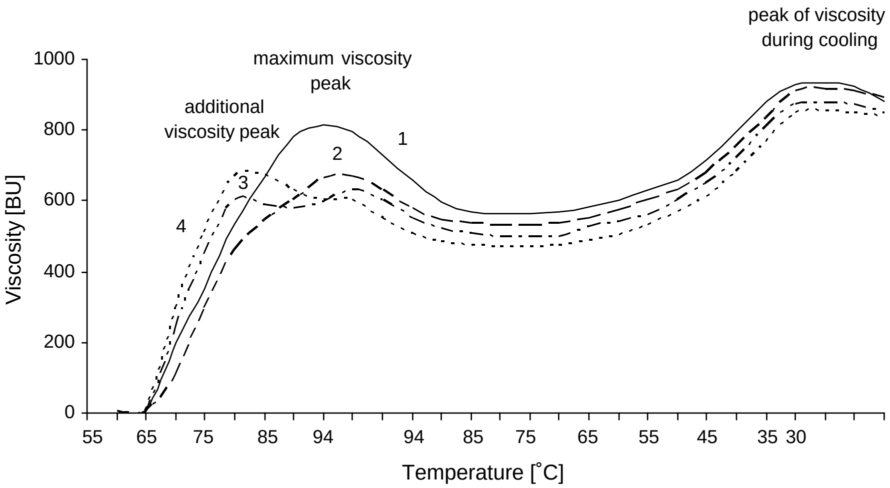

Fig. 1. Gelatinization characteristics of 40 g/l potato starch dispersion without kaolin (1); and the mixture of starch and 4 (2), 24 (3) and 40 (4) g/l of kaolin.

the increased volume of starch granules swollen in water and friction forces between them (Fig. 2). The swollen starch granules move in suspension without destruction in the absence of kaolin particles. The friction between granules in a suspension, which is permanently stirred, increases up to the temperature of maximum viscosity and then the granules are disrupted, while formation of an entangled solution of macromolecule net with elastic polymer loops and tails can be observed.

At kaolin concentration in the mixture higher than 12 g/l, kaolin particles are adsorbed on the surface of starch granules (Fig. 3), particularly on the membranes of the large granules, which swell earlier than granules of a middle or a low size. Fiction forces between starch granules with adsorbed kaolin particles are greater than the granules without kaolin which leads increase of viscosity, which is visualized as an additional viscosity peak. Further increase of temperature leads to the destruction of swollen starch granules with the kaolin particles on the surface, formation of flexible aggregates of starch macromolecules and to the decrease of viscosity (Fig. 1).

During heating up to 94 °C the mixtures containing more than 12 g/l of kaolin, held at that temperature for 10 min, behaved like liquids, however during cooling down to 30 °C they started to form a solid-like net. Rheological behavior of these mixtures is not liquid-like and can be described as the behavior of cross-linked polymer swelling in a good solvent. Therefore, the results of viscosity measurments, made with Brabender viscograph, are not reliable because these mixtures now longer show liquid like behaviour. For this reason rheological properties of cooled gelatinized

Table 1
Temperature of starch gelatinization and viscosity of starch and kaolin mixtures

Kaolin concentration (g/l)	Initial pasting temperature (°C)	Additional viscosity peak		Maximum viscosity peak		Viscosity (BU)			
		Temperature (°C)	Viscosity (BU)	Temperature (°C)	Viscosity (BU)	After 10 min at 94°C	Minimum	At 30°C	After 10 min at 30°C
0	65.0	–	–	92.0	830	580	570	930	880
0.8	64.2	–	–	93.1	750	560	550	930	870
2	63.6	–	–	94.0	720	540	540	900	870
4	63.7	–	–	94.0	690	540	530	910	890
6	63.5	–	–	94.0	690	540	540	900	870
8	63.6	–	–	94.0	685	545	535	900	875
12	63.1	73.0	560	94.0	680	540	520	860	840
16	63.3	73.0	595	94.0	670	535	525	875	855
20	63.2	73.0	610	94.0	655	530	520	875	855
24	63.6	73.3	600	94.0	630	520	515	870	850
28	63.5	73.3	640	94.0	650	530	520	880	865
32	63.7	73.4	650	94.0	650	530	520	915	905
36	63.4	73.3	645	94.0	640	520	510	905	895
40	63.6	73.0	660	94.0	635	525	510	925	910
52	63.7	73.0	670	94.0	600	480	470	850	840
56	63.6	73.0	670	94.0	600	480	460	850	840
60	63.7	72.0	765	94.0	640	510	480	925	910

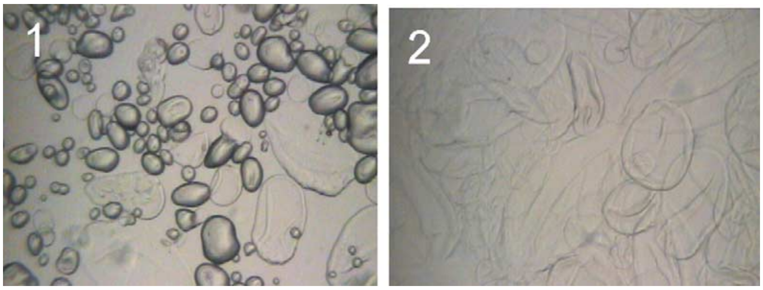

Fig. 2. Microphotographs of potato starch heated up to 63 °C (1), 73 °C (2) (magnification: 290×).

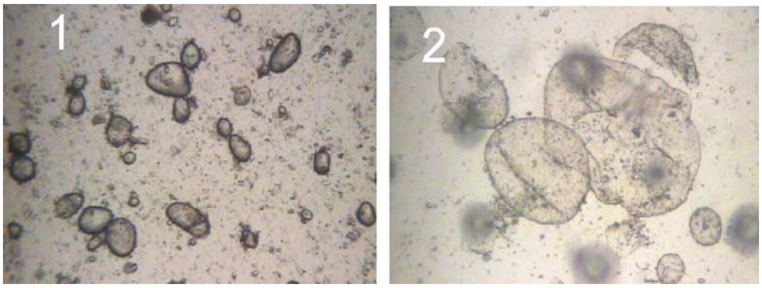

Fig. 3. Microphotographs of potato starch (40 g/l) and kaolin (40 g/l) mixtures heated up to 63 °C (1), 73 °C (2) (magnification: 290×).

starch/kaolin mixtures were determined using pastes of lower concentrations of starch (10 or 20 g/l) and kaolin (0–15 g/l).

The data (not shown) indicate that for the mixtures containing 10 g/l of starch and zero, 4, 10 and 15 g/l of kaolin shear stress varies from 8 to 12 Pa, at shear rate 200 s^{-1}, at 20 °C, and it does not depend on kaolin concentration. Calculated viscosity values of these mixtures were quite similar to each other (0.04–0.06 Pas). The globular size of gelatinized starch is much greater than the size of kaolin particles and evidently swelling starch globules include the kaolin particles without changing of their hydrodynamic volume. This data conforms with the change of the complex viscosity of gelatinized starch with increasing of kaolin concentration at 30 °C (Table 1) after gelatinization of their mixtures in the amylograph. The mixtures of gelatinized starch with kaolin behaved similar to Newtonian liquids only at a starch concentration lower than 5 g/l. In this case, the dependence of shear stress on shear rate is practically linear if we neglect of the change of flow viscosity at shear rates closed to zero where the experimental error is high. At the starch concentrations greater than 10 g/l, the behavior of the investigated systems is pseudoplastic.

An increase in the starch concentration up to 20 g/l leads to approximately a 9-fold increase of shear stress at all shear rates; for example: 70 Pa, at the shear rate 200 s^{-1}, at 20 °C. Shear stress decreases when the temperature of the mixture rises (Fig. 4). The relation between shear stress value and shear rate decreases gradually in each the following cycles (Fig. 5). The hysteresis loops on the rheological curves observed during 4–5 cycles for all samples of the mixtures of gelatinized starch. The flow viscosity in the linear region of the curve of the relationship between shear stress and shear rate decreased from cycle to cycle. However, the difference between these two neighbouring loops decreases when the number of cycle increases. These experimental data are close to the results obtained by other scientists (Tattiyakul & Rao, 2000). It can be associated with irreversible changes in the structure of solution as the result of destruction of hydrogen bonds between aggregates of starch macromolecules and their formation of hydrogen bonds within the aggregates, as well as the result of

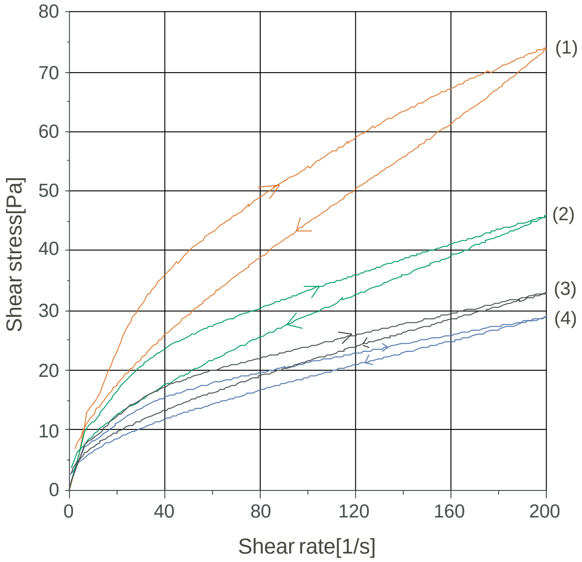

Fig. 4. Dependence of shear stress on shear rate of gelatinized potato starch (20 g/l) and kaolin (4 g/l) mixtures, at temperature 20 °C (1), 40 °C (2), 60 °C (3) and 70 °C (4).

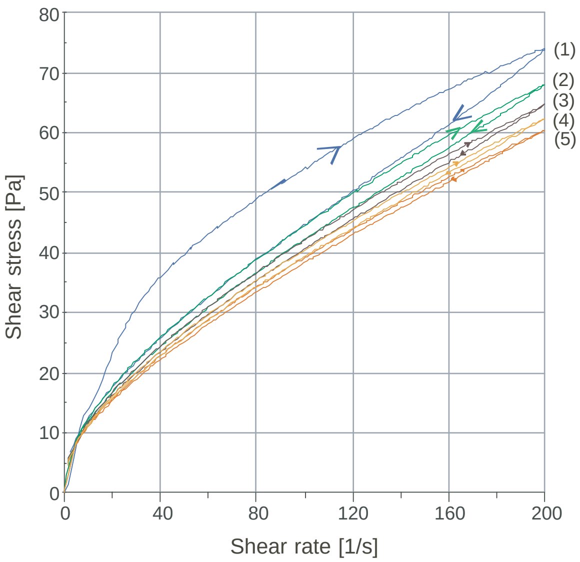

Fig. 5. Dependence of shear stress on shear rate of gelatinized starch (20 g/l) and kaolin (2 g/l) mixtures during 5 cycles.

the decreased number of loops and tails protruding from such an aggregate. The concentration of bonds between starch aggregates in the network decreases.

Rheological behavior of the mixtures of gelatinized starch with kaolin particles can be explained by the mathematical model proposed below.

The dependence of the viscosity of the liquid containing particles on the volume concentration of particles can be described by Eq. (1) when there is no interaction between particles.

$$\eta_1 = \eta_0 + K_1\psi \tag{1}$$

where η_0 is viscosity of the solvent, K_1 is constant, ψ is volume concentration of particles.

$$\psi = V_{\mathrm{ag}}N_{\mathrm{ag}} \tag{2}$$

where V_{ag} is the volume of macromolecule aggregate, N_{ag} is the number of aggregates in a volume unit.

When the aggregate of particles or macromolecules has a fractal structure the dependence of the aggregate volume on the number of initial particles can be described by the following equation:

$$V_{\mathrm{ag}} = V_0 i^{\alpha} \tag{3}$$

where V_0 is the volume of initial particle or macromolecule in solution, i is the number of particles in one aggregate, α is the fractal number that determines the structure of aggregate contained in particles or macromolecules.

$$N_{\mathrm{ag}} = \frac{N_0}{i} \tag{4}$$

where N_0 is the initial number of particles or macromolecules in a volume unit.

$$N_0 = \frac{c_0}{m_{\mathrm{p}}} \tag{5}$$

where c_0 is the mass concentration of the dispersed phase in solution, m_{p} is the mass of one particle or the initial aggregate of starch macromolecules.

From Eqs. (1)–(5) an equation can be obtained for viscosity of low concentrated dispersions when the formation of bonds between initial starch macromolecules or their aggregates does not proceed.

$$\eta_1 = \eta_0 + K_1 V_0 i^{(\alpha-1)}\frac{c_0}{m_{\mathrm{p}}} \tag{6}$$

When the starch concentration rises, the bonds between macromolecule aggregates form under the condition $V_0N_0 > 1$ and the network structure forms in a solution. The number of hydrogen bonds between two aggregates formed on a unit surface of one aggregate can be described to a first approximation by the equation:

$$n = K_2 c_0^{(1-1/(V_{\mathrm{ag}}N_{\mathrm{ag}}))} \tag{7}$$

where K_2 is proportional coefficient.

Eq. (7) shows that the number of bonds between aggregates in the network remains close to zero at low starch concentrations and increases when the starch concentration rises.

In this case, the viscosity increases when the number of bonds between aggregates rises and it is proportional to the surface area of aggregate, S_{ag}, and the number of aggregates in a volume unit.

$$\eta_2 = K_2 n N_{\mathrm{ag}} S_{\mathrm{ag}} \tag{8}$$

where

$$S_{\mathrm{ag}} = 36\pi V_{\mathrm{ag}}^{2/3} \tag{9}$$

Taking into account Eqs. (2)–(5) and (7)–(9) we obtain the following equation:

$$\eta_2 = 36\pi K_2 \frac{V_0^{2/3}}{m_{\mathrm{p}}} i^{(2/3\alpha-1)} c_0^{(2-m_p i^{(1-\alpha)}/(V_0c_0))} \tag{10}$$

From Eqs. (6) and (10) we can obtain the equation for the change of viscosity of gelatinized starch over a wide concentration range.

$$\eta = \eta_0 + K_1 V_0 i^{(\alpha-1)}\frac{c_0}{m_p} + 36\pi K_n K_2 \frac{V_0^{2/3}}{m_{\mathrm{p}}} i^{(2/3\alpha-1)} c_0^{(2-m_p i^{(1-\alpha}/(V_0c_0))} \tag{11}$$

When the starch concentration is high $\eta_2 \gg \eta_1$, Eq. (10) can be transformed into the equation:

$$\log(\eta_2) - 2\log(c_0) = \log(36\pi K_n K_2 \frac{V_0^{2/3}}{m_{\mathrm{p}}} i^{(2/3\alpha-1)} - i^{(1-\alpha)}\frac{m_{\mathrm{p}}}{V_0}\log(c_0)/c_0 \tag{12}$$

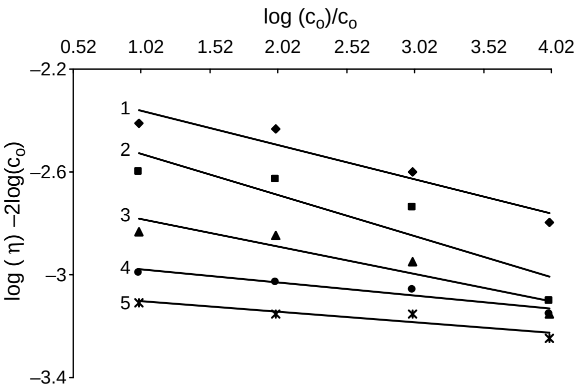

Fig. 6. Relationship between viscosity and starch concentration for gelatinized cross-linked waxy maize starch when plotted according to Eq. (12) at shear rate 10(1), 20(2), 40(3), 80(4) and 122 (5) s^{-1}. Experimental data are taken from Tattiyakul & Rao (2000).

At constant shear rate, the forces compressed starch macromolecules as well as the number of macromolecules in one aggregate is constant, i = constant. Therefore, experimental data on the dependence of viscosity, η_2, on the initial starch concentration, c_0, can be described by the linear relationship according to Eq. (12).

Fig. 6 shows that experimental data (which are taken from Tattiyakul & Rao, 2000) for gelatinized cross-linked waxy maize starch lay on the straight line when plotted according to Eq. (12). Correlation coefficients, the values of intercepts on ordinate axis and the tangents of slope angles of straight lines are displayed in Table 2. The decrease of intercepts on ordinate axis shows that the number of macromolecules in one aggregate decreases according to Eq. (10) when the shear rate increases.

When the value of 'a' is constant and the value of 'i' depends on shear rate, we can obtain a linear dependence of the intercept on the ordinate axis $\log((36\pi K_n V_0^{2/3}/m_p) + (2/3\alpha - 1)\log(i)$, on the logarithm of the tangent of the slope angle of straight lines $\log(m_p/V_o) + (1-\alpha)\log(i)$, calculated for different shear rates according to Eq. (12) and obtained from Fig. 6.

Fig. 7 shows that there does exist a linear relationship with correlation coefficient 0.976. The tangent of slope angle of straight line equals $(2/3\alpha - 1)/(1-\alpha) = 2.6 \pm 0.4$ and $\alpha = 1.1$. Therefore, the macromolecules in an aggregate are close to each other and form structures of high density.

Table 2

Dependence of correlation coefficients, the intercept on ordinate axis and the tangent of slope angle of straight lines according to Eq. (12) for gelatinized cross-linked waxy maize starch

Shear rate (s^{-1})	R	$\log\left(36\pi K_n K_2 \frac{V_0^{2/3}}{m_p} i^{(2/3\alpha-1)}\right)$	$I^{(1-\alpha)}(m_p/V_0)$
10	0.983	−1.4±0.3	40±6
20	0.967	−1.6±0.5	40±9
40	0.970	−2.0±0.4	33±6
80	0.983	−2.6±0.2	15±4
122	0.999	−2.79±0.04	13.6±0.6

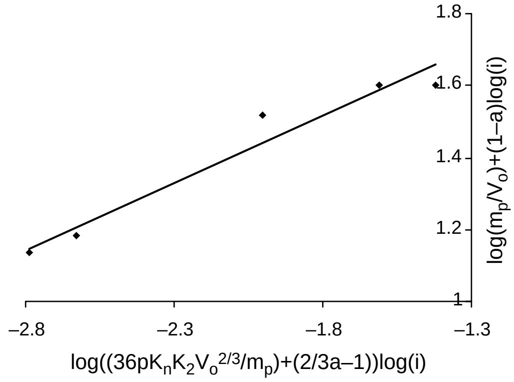

Fig. 7. Dependence of the $\log((36\pi K_n V_0^{2/3}/m_p) + (2/3\alpha - 1)\log(i)$ on the $\log(m_p/V_0) + (1-\alpha)\log(i)$ at different shear rates.

The temperature dependence on viscosity describes the constant K_2 in Eq. (12). According to Arrenius rules we can calculate activation energy of network destruction. As it is shown on Fig. 8 the dependence of viscosity on the temperature in Arrenius coordinates is linear. Correlation coefficients and activation energies for of starch–kaolin mixtures and for cross-linked waxy starch are presented in Table 3 (experimental data 1,2 are taken from Tattiyakul & Rao, 2000). The activation

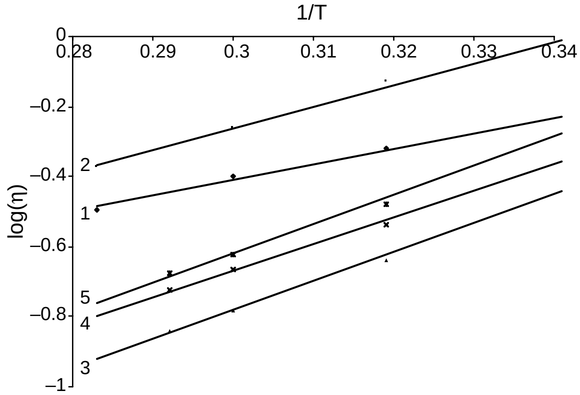

Fig. 8. Relationship between viscosity and temperature in Arrenius coordinates at shear rate 500 (1), 200 (2, 3), 100 revers (4) and 100 s^{-1} (5). Experimental data of curves (1,2) are taken from Tattiyakul & Rao (2000).

Table 3

Correlation coefficients and activation energies at different shear rates for gelatinized cross-linked waxy maize starch (1, 2) (experimental data from Tattiyakul & Rao 2000) and the mixture of gelatinized starch with kaolin (3–5) when shear rate increases (1–3, 5) and shear rate decreases (4)

N	Shear rate (s^{-1})	R	E (kJ/mol)
1	500	0.995	3.7±0.6
2	200	0.999	5.6±0.1
3	200	0.997	6.8±0.4
4	100 revers	0.998	6.2±0.3
5	100	0.996	6.8±0.4

energy is close to the energy of Van-der–Waals bond (2–8 kJ/mol).

4. Conclusions

Characteristics of starch gelatinization was affected by kaolin addition, and it depended on the kaolin concentration. Kaolin addition 0.8–4.0 g/l caused the increase of initial pasting temperature, as well as that of maximum viscosity. The addition of 12 g/l or higher kaolin concentration caused the emergence of additional viscosity peak at the temperature of approximately 73 °C. Further increase of kaolin concentration up to 60 g/l led to an increment of additional peak, while the value of maximum viscosity peak decreased.

Rheological properties of starch and kaolin mixtures (10 or 20 g/l of starch; 2–15 g/l of kaolin) were not affected by kaolin addition. Viscosity was affected only by the temperature of the measurment and the starch concentration.

A mathematical model for the rheological behavior of gelatinized starch, was developed which contains the number and volume of macromolecule aggregates, and the number of bonds between aggregates. According to that model, the bonds energy between aggregates reached the value of 2–8 kJ/mol, which seemed to be close to Van der Waals bonds energy.

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