Study of the rheological characteristics of the potato-based fish and vegetable mixture melt in the pre-die zone of a single-screw extruder

O I Aksenova, G V Alexeev and D D Temershin

ITMO University, 9, Lomonosova st., St-Petersburg, 191002, Russia

E-mail: oksi280491@yandex.ru

Abstract. This paper presents study of the rheological characteristics of the melt of the fish and vegetable mixture biopolymer in the pre-die zone of a single-screw extruder. The analysis of the flow index, being less than one, and that of the flow curves of the fish and vegetable mixture biopolymer melt, intended for production of expanded potato snacks, showed that the melt in a certain range of deformation rates can be attributed to pseudoplastic fluids. The results of the study represent important data in the design of the extruders molding units, on which the stability of the finished products quality depends. The results of the study can be used for mathematical modelling of the flow of the biopolymer melt in the pre-die zone of the extruder and can serve as justification of the screw rotational speed range in order to obtain extrusion foodstuffs of stably high quality.

1. Introduction

Extruded foodstuffs represented all over the world with a wide range of products, from breakfast cereals to protein textures, have recently been in great demand. The extrusion allows to process a variety of raw materials of plant and animal origin, mixtures and by-products of the food industry, which characterizes extrusion as a universal process.

The variety of processed raw materials, which qualitatively changes its properties during thermoplastic extrusion, causes problems of stabilization of the product yield and of obtaining a product of a predicted quality level, as well as problems associated with the creation of new designs of extruder molding units that would increase production efficiency, problems associated with the development and implementation of methods for assessing the quality of finished extrudates in the production. Rheological research methods can play a significant role in solution of the above-mentioned problems.

Among the complex of physical properties, rheological ones are main, since they determine the behavior of the biopolymer melt in the technological process. They are an external expression of the internal essence of objects, that is, they characterize the aggregate state, dispersion, composition, structure and type of interactions within the product [1]. The rheological properties of the biopolymer melt from fish-vegetable raw materials depend on the moisture content of the feedstock, on the temperature mode of the extrusion process and on the pressure in the pre-die zone of a single-screw extruder [2].

The aim of the paper is to research the rheological properties of the melt of the fish and vegetable mixture in order to predict the behavior of the material in the forming channel of a single-screw extruder.
extruder.

An analysis of rheological studies [3-9] shows that, as for the nature of the flow, melts into which mixtures based on starch raw materials or mixtures with a predominant content of starch raw materials when processed in the field of high temperatures and pressures are transferred are abnormally viscous fluids obeying the power law of Oswald-de-Ville:

$$\tau = k \cdot \dot{\gamma}^n,$$

(1)

$\tau$ is the shear stress at the capillary wall, Pa; $\dot{\gamma}$ is the shear rate near the capillary wall, s$^{-1}$; $k$ is the consistency coefficient, Pa$\cdot$s$^n$; $n$ is the flow index. At the same time, the consistency coefficient is a measure of the liquid viscosity, and the flow index characterizes non-Newtonian behavior. Based on the value of the flow coefficient, power-law fluids are divided into three groups: into pseudoplastic and thixotropic ones for $n < 1$, into Newtonian one for $n = 1$, into dilatant one for $n > 1$.

In extrusion processing, the coefficients $k$ and $n$ included in equation (1) can be determined experimentally based on capillary viscometry. Traditionally, capillary viscometry is used in order to measure the viscosity of melts at medium and high shear rates and at high temperatures up to 773 K [10]. The rheometer in this measurement is the rheometer-channel system with means for measuring the pressure difference, which is proportional to the shear stress and to the volumetric flow rate. At the same time, the extruders used to conduct the study should provide a sufficiently high pressure for forcing the biopolymer melt through long capillaries of small radius.

2. Materials and methods

The methodology for determination of the flow index $n$ and of the consistency coefficient $k$ is based on the direct use of the power law of the flow which is realized through the equation for calculating the pressure loss arising from the viscous flow of the polymer melt in any given simple channel (a capillary), and on the application of the two capillary and the least square methods.

The method of two capillaries allows to eliminate the determination of the pressure loss at the input to the forming channel, because capillaries are geometrically similar systems with different lengths of the forming channel, to neglect the turbulence arising in the initial section of the measuring capillary, as well as the final effects [10].

For the study, a laboratory unit based on a SFE-2 extruder was used. A pair of matrices was made, shown in Figure 1. The forming channels of the matrices had a circular cross section of equal diameter of 0.003 m and different lengths of 0.027 m for the short capillary and of 0.078 m for the long one. A ratio of capillary lengths to diameter equal to 9 and 26, respectively, was chosen so that in one of the cases a high ratio of capillary lengths to diameter was observed. The use of a high ratio completely eliminates the influence of the most significant input effects due to the stored elastic energy in the biopolymer melt subjected to shear at a high speed, the non-laminar flow in the capillary entrance area and the unsteady flow conditions when the flow is accelerated at the entrance into a small diameter capillary [10].

Figure 1. Dies with short and long round capillaries: 1 – die, 2 – capillary, 3 – pressure sensor PD100-DI12.0-111-1.0, 4 – boss, 5 – incompressible fluid, 6 – pressure transmission piston, 7 – barrel.
In order to determine the pressure at the input to the capillary, a pressure measuring system was installed, based on a membrane-pressure transducer. The pressure measurement system is a multilayer construction in which pressure is transferred to the sensor membrane from the biopolymer melt in the pre-die zone by a piston through a layer of incompressible fluid. Direct connection of the pressure sensor to the extruder pre-die zone is complicated by the constant sticking of the biopolymer melt flow and by the limited diameter of the extruder pressing case. Also, during rheometry in systems with a round capillary, we estimated the pressure difference in the extruder body and at the exit of the die (the atmospheric pressure), since the pressure difference inside the capillary cannot be determined, due to the difficulty to install pressure sensors in a round capillary of such a small diameter.

The medium used was a fish and vegetable mixture intended for the production of potato snacks with an increased content of protein and of dietary fiber. The recipe mixture had the following percentage of components: 79% of dehydrated ground potatoes, 13% of dry malt grains, 8% of fish powder from by-products of salmon processing (growths, flanks, napes, fish bellies, cuts from ridges, slices and non-standard pieces of salmon).

In the experiment, pressure losses Δp1 and Δp2 were determined for each capillary at a fixed volume flow and at variable temperature values in the pre-die zone of a single-screw extruder in the range from 373 to 433 K and at the initial humidity of the mixture from 12 to 18%. The selected range of temperature and humidity is typical of the hot extrusion mode.

The constancy of the flow rate was set by the rotational speed of the screw shaft of the extruder.

Below is the calculation procedure.

The difference between the obtained pressure loss values is calculated as follows:

$$\Delta p_{1,2} = \Delta p_1 - \Delta p_2$$

where Δp1, Δp2 are the experimental values of pressure loss at the exit of the extruder at an equal volume flow, Pa.

It is known that the equation for a power-law fluid using capillaries of circular cross section can be written as follows:

$$\Delta p_{1,2} = 2k \cdot (L_1 - L_2) \cdot Q^n \cdot \left(\frac{A}{4} \cdot r^A\right)^{-n} \quad A = \frac{3n+1}{n},$$

$$L_1, L_2$$ are the lengths of the long and short capillaries, respectively, \(m\); \(r\) is the radius of the capillary, \(m\); \(Q\) is the volumetric flow rate, \(m^3/s\). We denote:

$$N = 2k \cdot (L_1 - L_2) \cdot \left(\frac{A}{4} \cdot r^A\right)^{-n} \quad \Delta p_{1,2} = N \cdot Q_n.$$  

Let us suppose that in processing the experimental data, a system of points \(Q_n, \Delta p_{1,2}, k = 1 - m\), was compiled, which theoretically correlates with the equation (4). Then, using the least squares method, we can find estimates of the pair \((N, n)\):

$$\ln N = \left[\sum_{k=1}^{m} \ln^2 Q_k \cdot \sum_{k=1}^{m} \ln \Delta p_{1,2} - \sum_{k=1}^{m} \ln Q_k \cdot \sum_{k=1}^{m} \ln \Delta p_{1,2}\right] \cdot \left[m \cdot \sum_{k=1}^{m} \ln^2 Q_k - \left(\sum_{k=1}^{m} \ln Q_k\right)^2\right]^{-1}$$

$$n = \left[m \cdot \sum_{k=1}^{m} \ln Q_k \cdot \ln \Delta p_{1,2} - \sum_{k=1}^{m} \ln Q_k \cdot \ln \Delta p_{1,2}\right] \cdot \left[m \sum_{k=1}^{m} \ln^2 Q_k - \left(\sum_{k=1}^{m} \ln Q_k\right)^2\right]^{-1}$$

It is known that for an annular forming channel, the consistency coefficient is defined as follows:

$$k = \frac{N}{2(L_1 - L_2)} \cdot \left(\frac{A}{4} \cdot r^A\right)^{-n}.$$
3. Results and discussion

The results of calculating the coefficients $k$ and $n$, according to the procedure described above, based on the experimental data are shown in Table 1.

Table 1. Results of calculating the coefficient of consistency and of flow index.

| Temperature in the pre-die zone $T$ (K) | Initial humidity of the mixture $H$ (%) | Flow index $n$ | Consistency coefficient $k$ (Pa·s$^n$) |
|---------------------------------------|----------------------------------------|---------------|----------------------------------------|
|                                       |                                        |               |                                        |
| 373                                   | 12                                     | 0.522         | 492507                                 |
| 373                                   | 14                                     | 0.556         | 398121                                 |
| 373                                   | 16                                     | 0.589         | 327810                                 |
| 373                                   | 18                                     | 0.621         | 275341                                 |
| 383                                   | 12                                     | 0.532         | 420725                                 |
| 383                                   | 14                                     | 0.566         | 341960                                 |
| 383                                   | 16                                     | 0.599         | 283231                                 |
| 383                                   | 18                                     | 0.631         | 239417                                 |
| 393                                   | 12                                     | 0.542         | 359989                                 |
| 393                                   | 14                                     | 0.576         | 294234                                 |
| 393                                   | 16                                     | 0.608         | 245176                                 |
| 393                                   | 18                                     | 0.640         | 208604                                 |
| 403                                   | 12                                     | 0.552         | 308532                                 |
| 403                                   | 14                                     | 0.586         | 253621                                 |
| 403                                   | 16                                     | 0.618         | 212643                                 |
| 403                                   | 18                                     | 0.649         | 182138                                 |
| 413                                   | 12                                     | 0.562         | 264878                                 |
| 413                                   | 14                                     | 0.595         | 219015                                 |
| 413                                   | 16                                     | 0.627         | 184791                                 |
| 413                                   | 18                                     | 0.658         | 159368                                 |
| 423                                   | 12                                     | 0.572         | 227795                                 |
| 423                                   | 14                                     | 0.605         | 189484                                 |
| 423                                   | 16                                     | 0.637         | 160914                                 |
| 423                                   | 18                                     | 0.667         | 139754                                 |
| 433                                   | 12                                     | 0.582         | 196252                                 |
| 433                                   | 14                                     | 0.615         | 164249                                 |
| 433                                   | 16                                     | 0.646         | 140412                                 |
| 433                                   | 18                                     | 0.676         | 122830                                 |

An analysis of the results of calculating the flow index $n$ in Table 1 shows that for all humidity and temperatures in the considered range, the biopolymer melt can be assigned to abnormally viscous fluids (pseudoplastic or thixotropic), since $n < 1$.

Data from Table 1 were processed using mathematical statistics methods, on the basis of which the following regression equations were obtained:

$$k = 105.34 \cdot 10^4 - 22.39 \cdot 10^3 \cdot H + 3.59 \cdot 10^3 \cdot T - 2.84 \cdot H^2 - 0.409 \cdot T^2$$

(8)

$$n = 0.253 + 1.61 \cdot 10^2 \cdot H + 0.95 \cdot 10^3 \cdot T.$$  (9)

$T$ is the melt temperature of the biopolymer in the pre-die zone of a single-screw extruder, K; $H$ is the initial moisture content of the fish mixture, %.

An analysis of the regression equations shows that humidity has a greater effect on the rheological properties of the melt than temperature, which is consistent with the results of studies [3, 5, 6, 9]. The direct dependence of viscosity on humidity in the raw material and the inverse one on the temperature of the barrel are explained by the plasticizing effect of moisture during the extrusion of starch-containing raw materials, which reduces the degree of degradation during shear.

The obtained equations describe the change in rheological constants with a 95% probability level and make it possible to identify patterns of change in the rheological characteristics of the biopolymer.
melt in a given range of the studied parameters.

An analysis of the dependences of the rheological constants on the mixture humidity and temperature in the pre-die zone shown in Figure 2 showed that an increase in temperature from 373 K to 433 K leads to an exponential decrease in the consistency coefficient, which may be due to the overcoming of the viscosity by the kinetic energy of the molecules.

**Figure 2.** Dependence of the flow index \( n \) (a) and of the consistency coefficient \( k \) (b) on the humidity \( H \) of the fish and vegetable mixture and the temperature \( T \) in the pre-die area.

Based on the obtained values of the flow index \( n \) and of the consistency coefficient \( k \), the change in shear stress near the capillary wall at various humidity and temperature values in the pre-die zone of a single-screw extruder was determined by the power-liquid equation (1). The flow curves for the calculated parameters at a constant temperature and at a constant humidity are shown in Figure 3. An analysis of the flow curves in Figure 3 shows that the structure of the biopolymer melt breaks down in the range of shear rates of \( 10^{-2} \text{ s}^{-1} \), which indicates the rearrangement occurring in the melt of the product under study. Figure 3b shows that an increase in temperature causes a decrease in the shear stress \( \tau \) of the mixture melt, which is also consistent with studies [6, 9, 10].

**Figure 3.** Curves characterizing the currents: a - at a constant temperature of 403 K and at various humidity \( H \) values; b - at a constant humidity of 12% and at different temperatures \( T \).
4. Conclusion
In a series of experiments, it was confirmed that all components of the recipe mixture, for the production of potato snacks, when processed in the field of high temperatures under high pressure, pass into the melt, which refers to abnormally viscous liquids obeying the Oswald-de-Ville power law. As a result of studying the rheological properties of the melt of the fish and vegetable mixture located in the pre-die zone of the extruder, based on the calculation results at $n < 1$ and the nature of the flow curves, it was determined that the melt in a certain range of deformation rates can be attributed to pseudoplastic fluids. It was found that the maximum deformation rates necessary to obtain extrudable products of the required quality correspond to the smooth transition to the region of the destroyed structure and are of $10^{-20}$ s$^{-1}$.

The obtained results of the study of the rheological properties of the biopolymer melt helped to better understand the ongoing changes in the raw material during its transformation into a finished product and the influence of extrusion process factors on the formation of the finished product quality.

The authors will direct further research to study the relationship between the rheological properties of the melt and the microstructure of the finished product and the design of a unified forming unit for single-screw extruders.

References
[1] Vlachopoulos J and Strutt D 2003 Extrusion Minitec and Conference: From Basics to Recent Developments 1-25
[2] Chen Y, Struble L J and Paulino G H 2008 Int. J. Appl. Ceram. Tech. Applied. 5 513-21
[3] Chen H, Xie F, Chen L and Zheng B 2019 J. Food Eng. 244 150-58
[4] Alam M S, Kaur J, Khaira H and Cupta K 2016 Crit. Rev. Food Sci. 56 445-73
[5] Thandavathi Y L N, Wassen S and Kadar R 2019 J. Food Eng. 245 112-123
[6] Xie F, Yu L, Su B, Lui P, Wang J, Liu H and Chen L 2009 J. Food Eng. 49 (3) 371-77
[7] Wani I A, Farooq G, Qadir and Wani T A 2019 Int. J. Biol. Macromol. 131 (15) 850-57
[8] Martinez-Bustos F, Aguilar-Palazuelos E and Galicia-Garcia T 2012 Thermoplastic Behavior of Biopolymers during Extrusion Biopolymer Engineering in Food Processing (CRC Press, Taylor & Francts Group) chapter 7 pp 245-78
[9] Cheyne A, Barnes J and Wilson D I 2005 J. Food Eng. 66 1–11
[10] Schramm G 1994 A Practical Approach to Rheology and Rheometry (Karlsruhe: Gebueder HAAKE GmbH)