Research Article

Sunflower Seed Cake and Larvae MassRheological Properties Analysis During Pressing WithVarying Temperature, Pressure and Oil Content

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Abstract. For the successful implementation of alternative protein sourcesforbiorefinery, optimization of the process parameters is crucial. Knowledge of the rheological propertiesis necessary for the design and development of appropriate equipment and process calculations.The objective of this research was to evaluate the effect of the following pre-treatments: temperature, pressure andeffect of initial oil content on the rheological properties of sunflower seedcake and larvae tissue. The rheological behavior of two protein sourceswas determined by using a rotational viscometer with a hydraulic system and thermostatic bath attached to the equipment. Using the mathematical apparatus and experimental data it was observed that the plastic viscosity of the sunflower seed cake corresponded to the viscosity of the vegetable oil, which confirmed the Bingham rheology assumption put forward in this work. For the larvae mass, a Hershey Buckley fluid model was proposed.A positive linear relationship was found for pressure and a negativelinear relationship was found for the oil content of the sunflower seed cake and larvae tissue on shear stress.

Keywords: rheological property, sunflower seed cake, larvae, pulsed electricaldischarge, viscoplasticity flow, Bingham model, modelling

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1. Introduction

Sunflower oil production is rapidly emerging in Russia, Ukraine, Turkey and other countries, filling an important niche of locally supplied protein and fat sources. Processing of sunflower seeds is becoming of outmost importance to fulfill the requirements for safe products and find efficient ways to reduce potential chemical and technological hazards [1].

Due to their potential dietary contribution regarded to be suitable for animals feeding and human consumption, and at the same time as a sustainable, along with isolation of relevant nutritional ingredients such as protein or fat, the number of insect varieties and farms increases steadily [2]. Insect biomass contains from 40 to 70% of crude protein and up to 36% of lipids that can be extracted and used for various purposes [3].

Studying rheological properties of the samples pre-treatedbytemperature, ovepressure and initial oil contentcan help to advance work and to develop projects for their industrial application. Such technology has great prospect in food and pharmaceutical industry, because it can increase the yield of extra virginvegetable oil during pressing. Some studies of the influence of differentpre-treatment on rheological properties on some materials have been reported in scientific literature [4-6].

Some studies of the influence of particle size and temperature on sunflower paste have also been reported [7-9]; however, there is a gap regarding the influence of overpressure and initial oil content on viscosity in the initial stages of oils processing.

The purpose of this paper is to study rheological parameters peculiar for the evaluation of influence oftemperature, hydrostatic pressure and material oil content for sunflower seed cake and larvae mass. Rheology study of five different protocols with parameters close to the conditions of the production cycle were carried out.

2. Materials and methods

2.1. Preparation of seed cake

Sunflower seed cakewas obtained from a local factory afterindustriallyflaking and cooking steps before pressing operation. Such industrial procedures allowed us to obtain sunflower mass close to the homogeneous structure. Initial oil content of seed cake according to the specification was 56.1 ± 0.3%. Initial moisture and ash content were 5.0 ± 0.7% and 10.2 ± 0.6% respectively.Protein content of seed cake according to the specification was 16.2 ± 0.8%.

Preparation of larvae mass

For the experiments live black soldier fly larvae (Hermetiaillucens) were purchased from a local German producer (Ahaus, Germany). The larvae were stored for 24 hours at 4 °C until the experiments. Prior to the trials, the samples were sieved to remove residual faeces and milled using shredder until a homogeneous mass was obtained. Initial oil content of larvae mass was 34.5 ± 1.3%. Initial moisture content was72.0 ± 1.7%. Protein content of larvae mass according to the specification was 60.8 ± 0.7%.

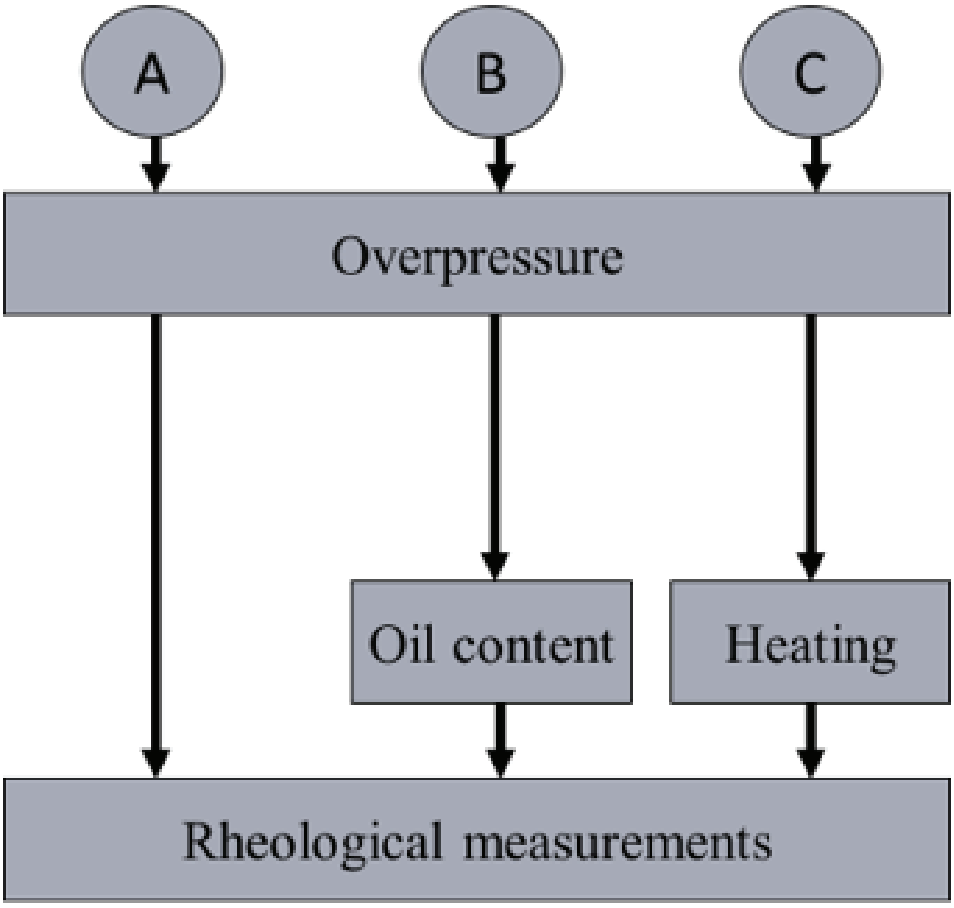

Figure 1: Different protocols of rheological measurements.

2.2. Rheological measurements

2.2.1. Measurement protocols

Rheological measurement was produced according to the scheme shown in Figure 1. Different protocols of rheological measurement involving heating, overpressure and different oil content were compared:

1. Protocol A: overpressure(in range from 980 up to 2800 Pa) + rheological measurements;
2. Protocol B: overpressure (at 981 Pa for sunflower seed cake and 2700 Pa for larvae mass) + variation of oil content (from 40% up to 56% for sunflower seed cake and from 25% up to 34.5% for larvae mass) + rheological measurements;
3. Protocol C: overpressure (at 1805 Pa) + heating(in range from 301 up to 318 K) + rheological measurements;

2.2.2. Rheological measurements

The rheological behavior of sunflower seed cake was determined by using a rotational viscometer Fungilab One Pro (Fungilab, Spain) with cylindrical spindle L4. Rheological analyses were obtained with shear rate from 1 to 10 -1.

2.2.3. Rheological measurements with adjusting temperature, overpressure and oil content

The choice of a rotational measurement method is based on the ability to measure the viscosity of both Newtonian and non-Newtonian media [10]. To assess the effect of pressure, the viscometer cell was equipped with a hydraulic system of controlled overpressure on the viscometer rotor [11]. Under the influence of gravity, the piston pressed on the mass under study. The value of excess pressure was determined as overpressure = Fg/S, where Fg – gravity, H; S - area of the piston, m^2. The gap between the rotor and the piston was 2 mm. To minimize the measurement error with and without the hydraulic system, the removal of the effective viscosity indicators from the device was performed with three times repetition. The reliability of experimental data was evaluated by analyzing the variance index and the average absolute deviation. To change the temperature, the viscometer cell was equipped with a flexible coil, which was supplied with hot water from the thermostat. The cell along with the coil was located in a foam body to ensure uniform heating of the pulp by volume. To change the oil content, the pulp was subjected to preliminary mechanical pressing on a hydraulic press, thoroughly mixed and sent to the measured cell of the viscometer. Thus, the experimental study of the effective viscosity of the pulp was carried out with a variation in the values of overpressure in the range from 980 to 2,700 Pa, temperature in the range from 28 to 45 °C and oil content of sunflower seed cake at values of 40, 48.5 and 56 %.The values used to fitting the data to the model related to the downward curve of the shear rate. The model adjustment was performed by using the Microsoft Excel software.

2.3. Microstructure

Sunflowerseed cake samples were examined using a scanning electron microscope (SEM) JEOL SEM 6360LA (A*Star, IMRE, Singapore) at the accelerating voltage of 10 kV and at the medium magnification of 170×.

Whole H. illucens larvae were fixed in a SEM sample holder with special optimal cutting temperature compound (OCT compound) glue consisting of glycols and resins, cryogenic frozen in super-cooled liquid nitrogen and inserted in a cryogenic preparation system (Emitech K 1250, UK), where the samples were broken down, and the free water was removed by sublimation. The prepared samples were transferred into the SEM (Jeol JSM 6460 LV, Japan) at approximately –180 °C and analyzed.

2.4. Statistical analysis

To minimize the magnitude of the measurement error with and without a hydraulic system, measurements of the apparent viscosity from the device was performed with triplicate. The results expressed as means ± standard deviation. Statistical significance was declared at $p < 0.05$ tested by analysis of variance (ANOVA).All statistical analyses including ANOVA were calculated using IBM SPSS Statistics Subscription software.

3. Results and discussion

3.1. Sunflower seed cake microstructure

The structure of rheological flows in the oil press auger is largely determined by the choice of the rheological flow equation, which affects the volumetric capacity of the oil press. When analyzing the microphotography of the pulp, oil globules (O) in the dark and light zones are clearly visible on its surface, as well as the oil film (F), expressed as light homogeneous areas (Fig. 2). The membrane of the oilseed cells (M) is represented as ringed light fibers surrounding the oilseed cell. At rest, the pulp is a loose coagulation structure [12]. Adhesion between dispersed particles occurs mainly due to free oil released as a result of heat treatment. In this way, the contacting particles of the pulp form a frame in a stationary oily film, linked to the walls of the screw channel.

The cross section cut of larvae sample is shown on Figure 2. The insect intestines can be observed from Figure 2. When analyzing SEM-images of larvae tissue, oil globules (O) are clearly visible on the surface of the sample. The size of oil globules ranges from a few microns up to 40 μm.

3.2. Flow characteristics

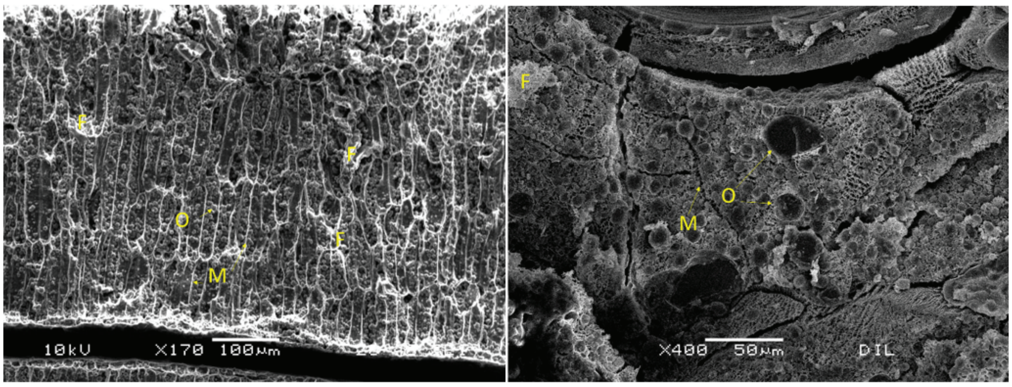

Figure 2: SEM-image of sunflower tissue (left) and larvae tissue (right) at x170 and x400 magnification.

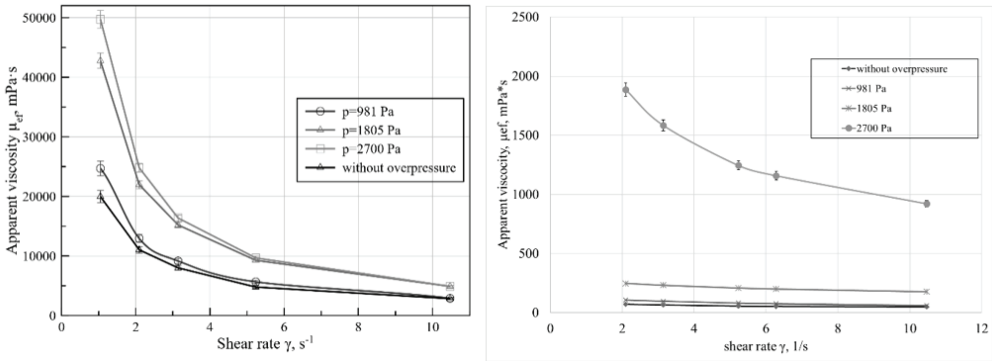

Figure 3: Apparent viscosity and shear rate dependence of seed cake (left) and larvae mass (right) at 981, 1805 and 2700 Pa over-pressure.

3.2.1. Effect of PEDtreatment (protocol A)

Structurally seed cake is a complicated dispersed system consisting of a disperse phase bubbles, oil globules with husk droplets and disperse medium as a protein shell.

The steady-shear rate flow of sunflower seed cake with different overpressure value was evaluated. Figure 3 shows the viscosity for protocol A sunflower seed as a function of the shear rate. Non-Newtonian shear-thinning behavior from the flow curves sunflower seed cake (pre-treated and non-treated) exhibit is observed. A similar shear-thinning behavior has also been observed for sesame seed [13] and sage seed solutions [14].

Because oilseed material had inhomogeneous loose structure in comparison with cellular structure of fruits and vegetables, only a few layers of surface oil cells were damaged and oil droplets released on the surface. Flow curve μ_{ef} (γ) showed that apparent viscosity decreased with decreased value ofoverpressure (981,1805 and 2700 Pa).

TABLE 1: Rheological model parametersfor non-treatedsunflowerseed cake

Shear rate γ, s^{-1}	Shear stress τ, Pa	Standard deviation	Bingham model τ_{lin} Pa	Engineering model τ_R, Pa	Discrepancy of Bingham model (τ-τ_{lin})/τ	Discrepancy of engineering model (τ-τ_R)/τ	Confidence interval δ_τ/τ
10.5	25.5	0.4	25.5	25.5	0.0%	0.0%	1.8%
5.2	25.0	0.6	25.0	24.9	0.0%	0.5%	2.4%
3.1	24.1	0.5	24.8	24.1	3.0%	0.1%	2.2%
2.1	23.1	0.8	24.7	23.2	7.0%	0.5%	3.3%
1.0	20.9	2.0	24.6	20.9	17.7%	0.2%	9.6%

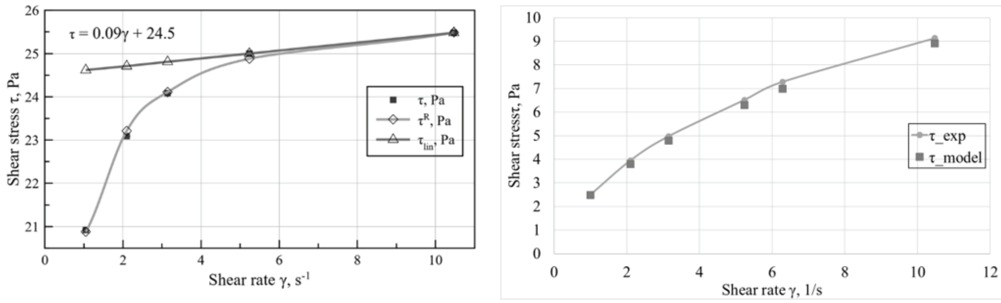

Figure 4: Shear stress vs. shear rate diagram for sunflower seed cake (left) and larvae mass (right).

Binghamand engineering model for oil presses were fitted to experimental shear-rate/shear-stress data fornon-treated sunflower seed cake (table 1). A linearization of initial rheological parameters in the inverse value of 1/γ and 1/τ was used to approximate data in Table 1.The engineering rheological model for sunflower seed cake in oil press can be represented as [15]:

$$\tau^R(\dot{\gamma}) = \frac{1}{b_0 + \frac{b_1}{\dot{\gamma}}}$$

Here b_0, b_1 are linear approximation coefficients of the reciprocals flow parameters of the material in the oil press channel (b_0 = 0.04 Pa^{-1}) and (b_1 = 0.01 Hz/Pa).

Considering that apparent viscosity showed nearly Newtonian character at higher shear rates to determine the exact relationship between viscosities and shear rates, shear stress vs. shear rate dependence of sunflower seed cake was plotted (Figure 4). At low shear rate (γ <5 s^{-1}) the external particles of sunflower seed cake slide along the channel walls, and with an increase in the shear rate on the oil film.

For larvae mass the flow curves ofshear rate and stress demonstrates sheardiminishing conduct ($n < 1$) with a yield value (Figure 4).The yield stress characterized as slightest shearstress important to start product flow, linkedwith the breaking of the material's innerstructure.

Table 2: Values for the parameters of the Herschel–Buckley model for the larvae mass

Temperature, K	$\tau_{0,}$ Pa	K, (Pa*s^n)	n	R^2
301	1.03	2.35	0.43	0.979

Herschel–Buckley's (HB) modelcan be represented as [16]:

$$\tau = \tau_0 + K \bullet \gamma^n$$

where K - the consistency index; n - the flow index.

Elastic deformation happens underneath the yield stress making it act like an elastic solid; however material flows over the yield stress making it act like a viscous liquid. If there should arise an occurrence of multiphase material like vegetable puree/paste, which is formed by a dispersion of insoluble parts (materials of cell wall) in a water solution (serum, containing sugars, minerals, proteins, and solvent polysaccharides), is having a yield stress [16]. Table 2, describes the estimations of the Herschel–Buckleymodel parameters for larvae mass. A higher estimation of R^2 (more than 0.97) was obtained. A nearby value for yield stress, flow index (n), and consistency index (K) were acquired tentatively when contrasted with values from the literature for vegetable items.

The rheological engineering model of sunflowerseed cake flow represented by eq.(1)allows to determine the infinite stress value τ_∞ as asymptote to stress curve, determined by the following equation:

$$\tau_\infty = \lim_{\dot{\gamma}\to\infty} \left[\tau^R(\dot{\gamma})\right] = \frac{1}{b_0}$$

To clarify the parameters of rheological engineering model (1)a smooth functional relationship in the form of spline approximation at the points of shear stress vs. shear rate diagram (Figure 4) on the interval [a = 1.0; b = 10.5] Hz is required (Table 1). Cubic spline used to approximate flow curve. Cubic spline represents benefits ofa function that:

on each segment is a polynomial of degree higher than three;

has continuous first and second derivatives on the whole interval [a, b];

in the experimental points, the equality of the spline interpolation function is done. For the unambiguous assignment of the spline impose additional requirements on the borders of the spline:$\tau''(a) = \tau''(b) = 0$. In this case, according to Schoenberg-Whitney about the conditions of existence of a spline interpolation there is only one spline $\tau_s(\gamma)$

satisfying the above conditions. In this case the relative discrepancy of rheological engineering model can be represented by the objective function Z (b_0, b_1):

$$Z\left(b_0, b_1\right) = \int_a^b \left[\frac{\tau^R(\gamma) - \tau_s(\gamma)}{\tau^R(\gamma)}\right]^2 d\gamma$$

Minimization of the functional (3) allowed us to refine the parameters of the engineering model in comparison with their quasi–linear approximation (b0 = 0.0356 Pa^{-1}) and (b1 = 0.0043 Hz/Pa). The realistic flow equation that is typical for an oil press in the process of extracting oil from the pulp is determined by the interval of shear rates: from 5 to 11 rad/sec. In this case, to determine the rheological parameters of the sunflower seed cake flow in the auger channel, the most realistic flow equation is the ideal-plastic Bingham model:

$$\tau(\dot{\gamma}) = \tau_0 + \mu_{pl} \cdot \dot{\gamma}$$

where τ_0 - Bingham yield stress; μ_{pl} - plastic viscosity. The parameters of the equation(5) can be determined on the basis of a linear approximation in the specified range of shear rates found with the asymptotes from eq.(3) of the parameters of engineering models (1). From the graphs of linear approximations (Figure 4) parameters τ_0 = 24.5 Pa andμ_{pl} = 0.09Pa·s for non-treated seed cake and τ_0 = 22.9 Pa; μ_{pl} = 0.11 Pa•s for PED-treated seed cakewere found. From an initial approximation of $\tau_0$and$\mu_{pl}$for shear rates in range from 5.2 to 10.5by relative discrepancy of ideal Bingham plastic model(5) can beobtained:

$$Z_B\left(\tau_0, \mu_{pl}\right) = \int_{5.2}^{10.5} \left[\frac{\tau^R(\dot{\gamma}) - \tau(\dot{\gamma})}{\tau^R(\dot{\gamma})}\right]^2 d\dot{\gamma}$$

Minimization of $Z_B(\tau_0, \mu_{pl})$ from equations (6) allowed to specify the parameters of the ideal Bingham-plastic model with respect to engineering rheological functions ($\square_0$ = 24.4 Pa; μ_{pl} = 0.12 Pa•s) for non-treated sunflower seed cake and ($\square_0$ = 22.7 Pa; μ_{pl} = 0.11 Pa•s) for PED-treated sunflower seed cake. Considering the fact that the apparent viscosity of sunflower seed cake corresponds to the viscosity of vegetable oil the Bingham model for sunflower seed cake was confirmed.

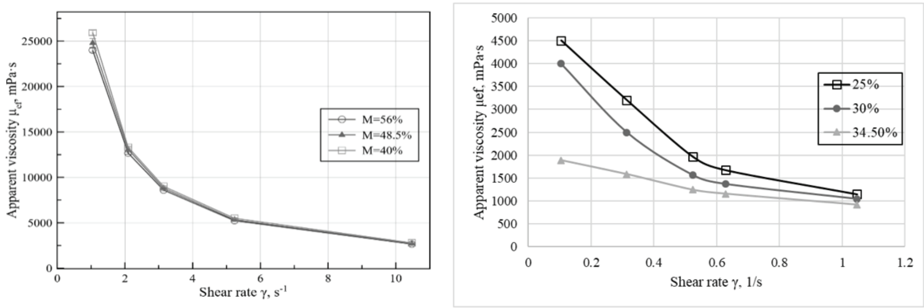

Figure 5: Apparent viscosity and shear rate dependence of sunflower seed cake at 56, 48,5 and 40% oil content at p=981 Pa (left) and larvae mass at 34.5, 30 and 25% oil content at p=2700 Pa (right).

Table 3

Oil content, M / 100	Yield stress, τ_0	Plastic viscosity, μ_{pl}
40 %	28.5 Pa	42.8 mPa·s
48.5 %	27.5 Pa	54.1 mPa·s
56 %	27.1 Pa	60.3 mPa·s

3.3. Effect of oil content (Protocol B)

In Fig. 5 the viscosity diagrams (mPa·s) vs. shear rate (s^{-1}) are represented for seed cake measured at oil content of 40, 48,5 and 56% at overpressure p=981 Pa and for larvae mass at oil content of 25, 30 and 34.5% at overpressure p=2700 Pa.

To assess the influence of the oil content of sunflower seed cake on the parameters of the Bingham rheological model, a statistical analysis of the coefficients of linear approximation of experimental data was performed (Fig. 5), which showed the dependence only of the yield strength and effective viscosity on the oil content of the pulp. Reducing the oil content leads to an increase in the expended pressing energy and, as a result, an increase in the temperature of the pulp and the pressure on the matrix of the oil press. The presence of free oils in the form of a film on the surface of the pulp provides a powerful lubricating effect [17]. In addition, the presence of even 1 % oil on the surface of the pulp provides stabilization and normalization of the pressing process, which indicates the importance of the process of heat treatment. The parameters of equation (4) are determined from the graphs of linear approximations for the oilseeds m = 40, 48.5 and 56%, which are listed in table. 2. at the same time, the plastic viscosity values that characterize the presence of an oil film are in good agreement with the viscosity values of sunflower oil [18].

Table 3

Bingham equation parameters for different oil content in seed cake

Oil content effect on parameters of equation (4) can be represented as:

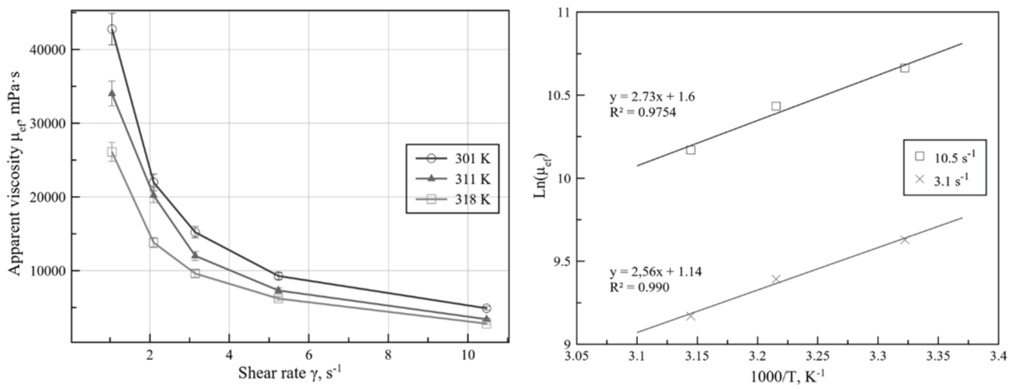

Figure 6: Apparent viscosity and shear rate dependence of sunflower seed cake at 301, 311 and 318 K (left) and temperature ramp dependence on natural logarithm at p= 1805 Pa.

$$\tau(\dot{\gamma}, M) = (-0.09 \cdot M \ + \ 31.9 Pa\ ) + (0.0011 \cdot M \ - \ 0.0005 \cdot Pa \cdot s) \cdot \dot{\gamma}$$

3.3.1. Effectof temperature(Protocol C)

Of practical interest is the study of the influence of temperature on the rheological model of the flow of sunflower pulp. During the pressing process, the temperature of the pulp can reach 140 °C. The change of rheological properties of pulp at different temperatures is due to the spatial-temperature structural changes in the volume of pulp, as well as the dynamic process of structure formation. In this regard, the preheating process of the pulp is an important preparatory stage that affects the efficiency of the pressing process.In fig. 6 the viscosity diagrams (mPa·s) vs. shear rate (s^{-1}) are represented for seed cake measured at temperature of 301, 311 and 318 K. All samples from protocol C were under 1805 Pa overpressure because of the more stable apparent viscosity data.

The activation energy was determined and the temperature influence on the rheological properties of the pulp was estimated using the Frenkel-Eyring equation:

$$\mu = A \exp(\frac{E_a}{RT}) \cdot \gamma^{-n}$$

where A is the pre-exponential multiplier (Pa·s), Ea is the activation energy (j·mol-1),R is the universal gas constant (8,314 j·mol-1·K-1), T is the thermodynamic temperature (°K), γ is the numerical value of the strain rate; n is the rate of destruction of the structure.

Under experimental conditions, the strain rate γ is a constant value, practically independent of temperature, so the Arrhenius equation was used to determine the energy:

$$\mu = A \exp(\frac{E_a}{RT})$$

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Logarithm of the equation (9) received:

$$Ln(\mu) = Ln(\mu_{\infty}) + \frac{E_a}{R}\frac{1}{T}$$

By entering the notation: y = Ln(μ); a = Ln(μ∞); b = Ea/R; x = 1/T, we obtained the linearized equation y = a + bx. We determined the coefficients a and b, and calculated the pre-exponential multiplier AandEa activation energy. To predict the maximum shear stress of the mezga an attempt was made to determine the type of functional curve from the temperature by reducing the latterto the linear view. The problem was solved by selecting a semi-logarithmic scale (Fig. 7). After analyzing the obtained graphical dependencies in semi-logarithmic scales, we can conclude that equation (9) is applicable for this dependence. The tangent of the angle of inclination of this line determines the activation energy of the process.

Based on mathematical processing of experimental dependencies Ln(μ) from 1/T, the activation energy and the pre-exponential multiplier are determined. The rectilinear form of the dependence of Ln (μ) on 1/T indicates the formation of connections of one type of fluctuation grid. The model flow equation in the framework of the Bingham rheological model taking into account the established temperature influence can be represented by the following dependence:

$$\mu = A\,\text{exp}(\frac{E_a}{RT}) = -1.98 \cdot \text{exp}(\frac{26.2 \cdot kJ}{RT})$$

The activation energy of the sunflower seed cake for the deformation rate of 3.14 s-1 is 21.19 kJ/mol, the pre-exponential multiplier A = -1.98 Pa.

4. Conclusion

Taking into account the fact that the effective viscosity of the pulp corresponds to the viscosity of vegetable oil [18], which is part of this viscoplastic material, we have received confirmationBingham's rheology of sunflower pulp. As can be seen from the presented data, the consistency graph is a Bingham viscoplastic liquid.

Larvae mass demonstrates shear-thinning conduct and found to display Harshley-Buckley flow behavior with yield stress decrease with decreasement in oil content. The larvae mass viscoelastic conduct was described as a weakgel.

Experimental studies have shown the presence of structure formation of the pulp entering the oil press. As the shear rate increases, the effective viscosity decreases. The consistency of the pulp corresponds to Bingham's rheology. It is established that the yield strength of sunflower pulp changes linearly depending on the excess pressure, and the obtained dependence τ(γ, p) allows identifying the flow of the plastic layer as an oil filmat the boundary of the piston flow of the sunflower seed cake. The influence of the increase in oil content of the pulp on the flow rheology is characterized by a decrease in the value of the maximum shear stress of the pulp from 28.5 to 27.11 Pa, which reduces the load on the pressing process.

The structural mechanical and energy properties of sunflower pulp depend on a number of factors: the component composition of the pulp and the temperature conditions of processing. The influence of temperature is characterized by a decrease in the plastic viscosity of the pulp from 0.0985 to 0.0917 Pa•s for samples at an excess pressure of 1.8 kPa.

The information obtained is conceivably helpful for future examinations on the properties of food and process design.

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