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Influence of structure-forming agent on rheological properties of polymer mixture based on low and high density polyethylene

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Abstract. *The paper considers the effect of a structure-forming agent (titanium dioxide) on the rheological characteristics of a polymer mixture based on low and high density polyethylene, taken in a 50/50 ratio. The titanium dioxide concentration was 1 wt%. The rheological behavior of melts of polymer composites was studied using a CEAST MF50 capillary rheometer (Instron, Italy) at temperatures of 190, 210, 230, 250 °C and loads of 3.8, 5.0, 10.0, 12.5, and 21.6 kg. The effect of temperature and shear stress on the regularity of changes in effective viscosity and shear rate has been established. According to the Arrhenius – Frenkel – Eyring model, the activation energy of the viscous flow of composites is determined. The "apparent" activation energy of the viscous flow varies within 16.04–33.10 kJ/mol for the initial polyethylene mixture and in the range of 6.96–33.10 kJ/mol for composites modified with a structurant based on a mixture of low and high density polyethylene. A universal temperature-invariant characteristic of the viscosity properties of polymeric materials has been constructed, which makes it possible, by extrapolating this dependence to the region of high shear rates, to predict the technological mode of their processing by injection molding and extrusion.*

Keywords: *rheology, polymer blend, structure-forming agent, titanium dioxide, shear stress, shear rate, temperature invariant characteristic*

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ХИМИЧЕСКИЕ НАУКИ

Научная статья
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Влияние структурообразователя на реологические свойства полимерной смеси на основе полиэтиленов низкой и высокой плотности

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Аннотация. *В работе рассмотрено влияние структурообразователя диоксида титана на реологические характеристики полимерной смеси на основе полиэтиленов низкой и высокой плотности, взятых в соотношении 50/50.*

Концентрация диоксида титана составляла 1% масс. Реологическое поведение расплавов полимерных композитов исследовано с использованием капиллярного реометра марки CEAST MF50 (Instron, Италия) при температурах 190, 210, 230, 250 °C и нагрузках 3,8, 5,0, 10,0, 12,5 и 21,6 кг. Установлено влияние температуры и напряжения сдвига на закономерность изменения эффективной вязкости и скорости сдвига. Согласно модели Аррениуса – Френкеля – Эйринга определена энергия активации вязкого течения композитов. «Кажущаяся» энергия активации вязкого течения варьируется в пределах 16,04–33,10 кДж/моль для исходной смеси полиэтиленов и в диапазоне 6,96–33,10 кДж/моль для композитов, модифицированных структурообразователем на основе смеси полиэтиленов низкой и высокой плотности. Построена универсальная температурно-инвариантная характеристика вязкостных свойств полимерных материалов, позволяющая путем экстраполяции этой зависимости в область высоких скоростей сдвига прогнозировать технологический режим их переработки методами литья под давлением и экструзии.

Ключевые слова: *реология, полимерная смесь, структурообразователь, диоксид титана, напряжение сдвига, скорость сдвига, температурно-инвариантная характеристика*

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INTRODUCTION

Polymer blends are widely used in various industries due to their strength, elasticity, low cost, lightness, transparency, and ease of processing [1]. Thus, these materials can be classified as promising materials. The morphology and final properties of polymer mixtures depend on the polymer structure, compatibility, rheological features, and the ability to be processed by injection molding and extrusion [2].

Since polyethylene is the most widespread commercial polymer in the world with an annual production volume of about 80 million tons [3], the study of the properties of composite materials based on a mixture of polyethylenes is of great interest [4, 5]. The main attractive characteristics of polyethylene are its low price, dielectric properties in a wide frequency range, good chemical resistance, manufacturability, impact strength, and flexibility [6]. There are several types of polyethylene: ultra high molecular weight polyethylene, high density polyethylene, low density polyethylene, linear low density polyethylene [7]. Blending different types of polyethylenes can provide the desired set of properties with improved processability. The use of polyethylene mixtures makes it possible to obtain a whole range of polymeric materials and products based on them with various combinations of density and crystallinity degree [8]. Since in the field of polymer processing it is necessary to properly control the flow and stability of polymer melts, it was interesting to study the influence of a structure-forming agent on the rheological properties of a melt of a polymer mixture based on low and high density polyethylene.

Titanium dioxide is widely used as a structure-forming agent and filler in polymer composites [9–11]. This is due to its useful properties, for example, low cost, bactericidal action, large specific surface area per particle size, non-toxicity for humans and animals [12, 13]. It is obvious that the solid dispersed substance loading into the composition of the polymer matrix significantly affects the rheology of the composite. The rheology of composites plays an important role in evaluating their processability by injection molding and extrusion. In this regard, the task of this study was to find out how the mixing an $LDPE_{(50)}/HDPE_{(50)}$ polymer composition with titanium dioxide can affect the pattern of change in their rheological characteristics.

MATERIALS AND METHODS

The object of study was a mixture of low density polyethylene (LDPE) and high density polyethylene (HDPE).

LDPE is characterized by the following properties: ultimate tensile stress 15 MPa, elongation at break 764%, melt flow rate at 5 kg load and 190 °C temperature 8.9 g/10 min.

HDPE is characterized by the following properties: ultimate tensile stress 30 MPa, elongation at break 50%, melt flow rate at 5 kg load and 190 °C temperature 17 g/10 min.

Titanium dioxide (analytical grade) TiO_2- grade P-02 (the mass fraction of the rutile form is not less than 95%) was used as a structure-forming agent. Inorganic finely dispersed crystalline substance with a melting point of 1843 °C. The titanium dioxide concentration was 1 wt%.

To obtain nanosized particles, an A-11 basic analytical mill (IKA, Germany) was used. TiO_2 particle size was determined on a Mastersizer 3000 laser analyzer (Malvern Instruments, England). The measurement range of the instrument is 0.01–3500 μm. The particle size varied in the range of 95–118 nm.

Polymer compositions with $LDPE_{(50)}/HDPE_{(50)}$ and $LDPE_{(50)}/HDPE_{(50)}$ + 1 wt% TiO_2 were obtained in the process of mixing on laboratory rollers at a temperature of 160–170 °C within 8–10 minutes. Further, at a pressing temperature of 180 °C, the plates were molded from which the corresponding samples were cut out for testing.

The choice of the 50/50 ratio of the $LDPE_{(50)}/HDPE_{(50)}$ polymer mixture was due to the fact that when equal amount of mixture components are used the phase inversion and the best technological compatibility and miscibility are achieved [8].

The rheological properties of the polymer composition melt were measured on a CEAST MF50 capillary rheometer (Instron, Italy), which determines the following properties: effective viscosity, shear rate, and shear stress. The rheometer is equipped with a nozzle having a ratio of the length (23.400 mm) of the capillary to the diameter (1.048 mm) equal to – 24. The measurements were carried out at four temperature s (190, 210, 230, and 250 °C) and five loads (3.8, 5.0, 10.0, 12.5, and 21.6 kg).

The activation energy of the samples under consideration was determined from the tangent of the slope of the curves according to the following formula [14]:

$$E = 2.3R(dlg(d \times (1000\ /\ T))),$$

where R = 8.31 J/mol×K is the universal gas constant, $dlg(d \times (1000/T))$ is the tangent of the slope of the lgh versus $1000/T$ curve.

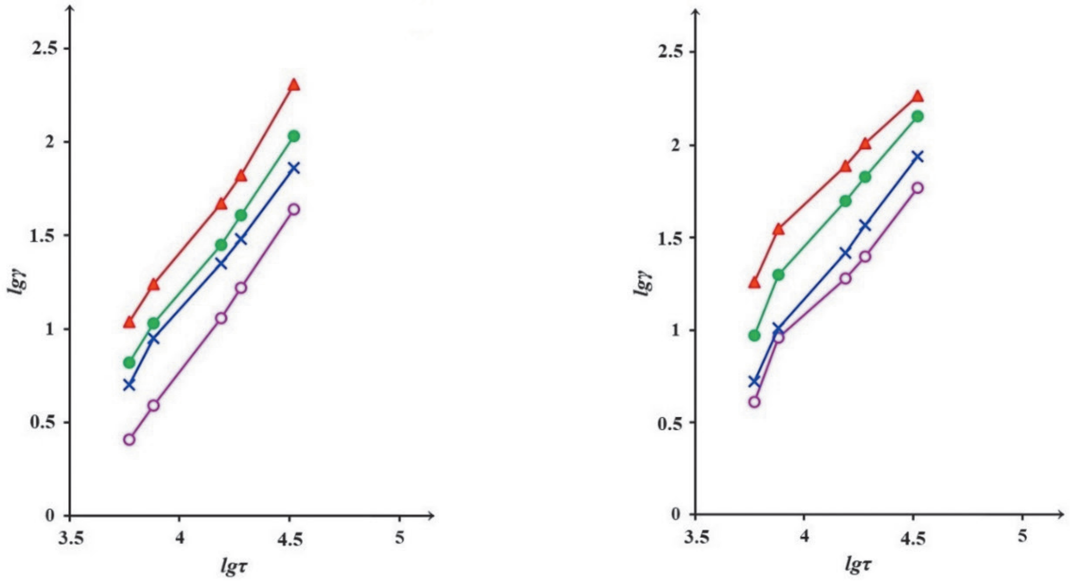
RESULTS AND DISCUSSION

For comparison, the flow curves of the initial LDPE$_{(50)}$/HDPE$_{(50)}$ mixture and the LDPE$_{(50)}$/HDPE$_{(50)}$ composite with 1 wt% TiO$_2$ were presented simultaneously. Fig. 1 shows the flow curves of these polymer composites. As can be seen from Fig. 1, *b*, up to the shear stress $lg\tau = 3.88$, in the sample containing the structure-forming agent (TiO$_2$), the region of the lowest Newtonian viscosity was recorded. At higher shear stresses, a non-Newtonian region of melt flow takes place.

A comparative analysis of the curves shows that the composite with LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ has a higher shear rate at 5 different shear stress values (5824 Pa ($lgt = 3.77$); 7664 Pa ($lgt = 3.88$); 15328 Pa ($lgt = 4.19$); 19161 Pa ($lgt = 4.28$); 33110 Pa ($lgt = 4.52$)). This is due to the fact that, with the inclusion of a structure-forming agent into LDPE$_{(50)}$/HDPE$_{(50)}$ mixture, an increase in melt fluidity is observed, which contributes to the movement of macrosegments associates relative to each other [15].

The rheological properties of polymers can be characterized only by establishing the dependence of the effective viscosity of the melt on the shear rate. Knowledge of viscosity versus shear rate curves at various temperatures is essential for process design, optimization, and troubleshooting. Fig. 2 shows in logarithmic coordinates plots of the effective viscosity of melts of polymer compositions based on LDPE$_{(50)}$/HDPE$_{(50)}$ and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ on shear rate, at various temperatures and loads. Comparative analysis of the curves in Fig. 2 allows us to establish their similarity and difference. The similarities lie in the fact that the flow curves of the considered composites indicate the non-Newtonian nature of the melt flow, while their viscosity decreases with increasing shear rate. Such fluids are commonly referred to as “pseudoplastic” in nature, which is a specific example of a non-Newtonian fluid. Long macromolecules under the influence of the applied shear stress are straightened and oriented in the flow. However, their orientation is disturbed by the thermal motion of other macromolecules, so the degree of orientation depends on the intensity of the thermal motion and the magnitude of the applied shear stress or rate gradient. As the shear rate increases, the particles gradually orient themselves relative to the flow. Since the dissipation of energy because of particles friction depends on the degree of their orientation in the direction of the fluid flow, they tend to orient themselves in such a way as to provide the least resistance to the fluid flow. Therefore, the effective viscosity decreases as the melt flow rate increases [16].

The difference lies in the fact that for LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ composites, the transition from the region of the highest Newtonian viscosity to the non-Newtonian one occurs at a load of more than 5.0 kg. At that time,



a **b**

○ 190 °C × 210 °C ● 230 °C ▲ 250 °C

Fig. 1. Dependence of shear rate on shear stress for LDPE$_{(50)}$/HDPE$_{(50)}$ (a) and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ (b) polymer composites at various temperatures

Рис. 1. Зависимость скорости сдвига от напряжения сдвига для полимерных композитов из LDPE$_{(50)}$/HDPE$_{(50)}$ (a), а также LDPE$_{(50)}$/HDPE$_{(50)}$ и 1% масс. TiO$_2$ (b) при различных температурах

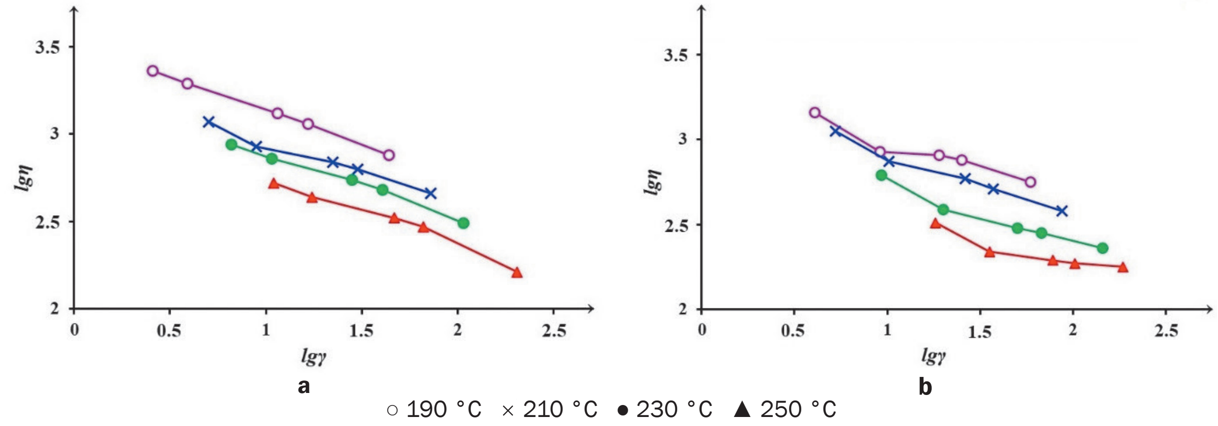


Fig. 2. Dependence of effective melt viscosity on shear rate for LDPE$_{(50)}$/HDPE$_{(50)}$ (a) and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ (b) polymer composites at various temperatures

Рис. 2. Зависимость эффективной вязкости расплава от скорости сдвига для полимерных композитов из LDPE$_{(50)}$/HDPE$_{(50)}$ (a), а также LDPE$_{(50)}$/HDPE$_{(50)}$ и 1% масс. TiO$_2$ (b) при различных температурах

this region was not visible in the initial polymer mixture. Also, as temperature and shear stress increase, the slope of the flow curves decreases.

In addition to shear rate, viscosity also depends on temperature. There are several models to describe this dependence. One of them is the Arrhenius equation, which makes it possible to accurately describe the dependence of viscosity on temperature [14, 17]. Fig. 3 shows the results of a study of the effect of reverse temperature on the effective viscosity of composites based on LDPE$_{(50)}$/HDPE$_{(50)}$ and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$. For most polymers, the viscous flow activation energy is the minimum energy required for a macrochain segment to overcome the energy barrier and move from an *in situ* position to a nearby "hole" during flow. It is a sign of the sensitivity of the apparent activation energy of a viscous flow to temperature.

Processing the results of experimental studies in the coordinates *lnη-1000/T* (Fig. 3) makes it possible to determine the point or apparent activation energy of a viscous flow. The activation energy of a viscous flow determines the energy required to move a macrochain segment from one position to another in the process of thermal energy fluctuations in the melt.

A comparative analysis of the curves in Fig. 3 shows that the dependence of the logarithm of effective viscosity on temperature is predominantly non-linear. The figure shows that the activation energy of LDPE$_{(50)}$/HDPE$_{(50)}$ is higher than that of LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$. This indicates that the melt viscosity of LDPE$_{(50)}$/HDPE$_{(50)}$ is more temperature sensitive compared to LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$. In these samples, the "apparent" viscous flow activation energy varies within 16.04–33.10 kJ/mol for LDPE$_{(50)}$/HDPE$_{(50)}$

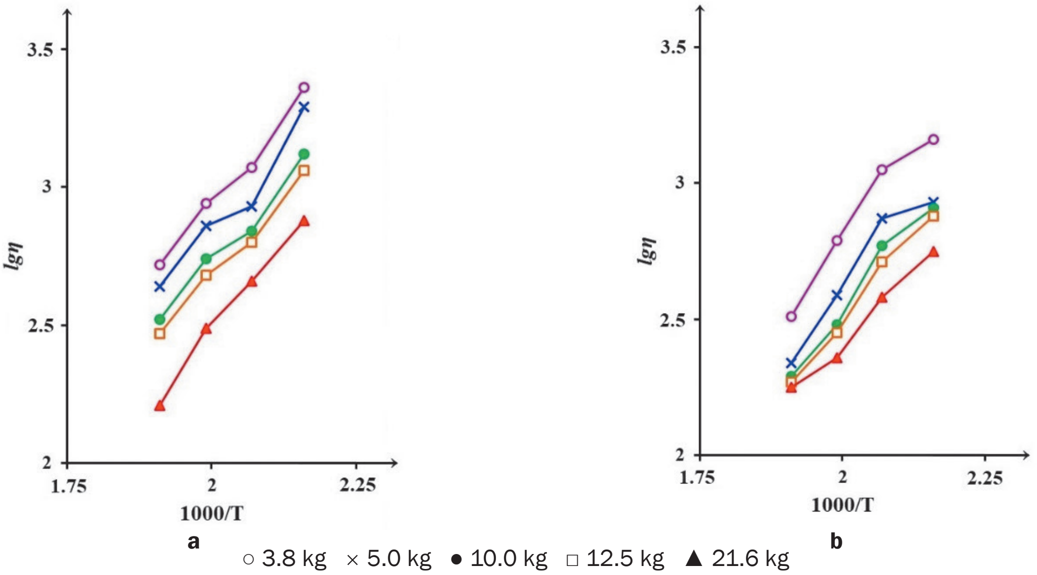


Fig. 3. Dependence of melt viscosity on reciprocal temperature in Arrhenius coordinates for LDPE$_{(50)}$/HDPE$_{(50)}$ (a) and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ (b) polymer composites at various loads

Рис. 3. Зависимость вязкости расплава от обратной температуры в аррениусовских координатах для полимерных композитов из LDPE$_{(50)}$/HDPE$_{(50)}$, а также LDPE$_{(50)}$/HDPE$_{(50)}$ и 1% масс. TiO$_2$ (b) при различных нагрузках and in the range 6.96–33.10 kJ/mol for LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$.

The results obtained are in good agreement with the Arrhenius equation, according to which the activation energy of a viscous flow tends to somewhat decrease in the presence of a structure-forming agent. The results of the study also affect the processability of polymeric materials. The relatively low temperature dependence of LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ melts can, to a certain extent, simplify the choice of the temperature regime for processing composites. Fig. 3 shows that the viscosity of LDPE$_{(50)}$/HDPE$_{(50)}$ and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ decreases with increasing temperature. However, the melt viscosity of LDPE$_{(50)}$/HDPE$_{(50)}$ changes with temperature much more distinctly than that of LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$. Therefore, it can be assumed that the rheological behavior of LDPE$_{(50)}$/HDPE$_{(50)}$ is more sensitive to temperature than LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$. The decrease in viscosity with increasing temperature is because of the fact that with increasing temperature, the free volume of the melt increases, which leads to a decrease in the forces of interchain interaction. In other words, an improvement in the freedom of movement of macrochains in the melt as the temperature of the melt increases, the kinetic energy of macromolecules necessary for their movement increases in a viscous system [18].

The great importance of the temperature dependence of the effective viscosity of polymers is due to the fact that temperature change is one of the main means of regulating the technological process of their processing. It is of interest to find the relationship between the effective viscosity and other rheological properties independent of temperature. The presence of such a temperature-invariant characteristic would facilitate the construction of flow curves in very wide ranges of rate and shear stresses from a relatively small number of experimental points. Among the formulas proposed for this purpose, the Vinagradov method tested on a large experimental material can be noted [19]. Using the temperature-invariant characteristics of the viscosity of non-Newtonian fluids, it is possible to determine the viscosity values at any rate gradient and at any temperature in those intervals of their change that took place during the experiment. Fig. 4 shows the temperature-invariant dependence of the reduced viscosity (η_e/η_o) on the reduced shear rate ($\gamma\eta_o$), where η_o is the highest Newtonian viscosity, and η_e is the effective viscosity of the melt.

The horizontal section on this curve corresponds to the condition when $\eta_e = \eta_o$, which is typical for the Newtonian region at very low shear rates. It has been established that, regardless of temperature and shear stress, the dependence of the reduced viscosity on the reduced shear rate changes according to the same pattern. The existence of a universal characteristic of the viscosity properties of polymeric materials is explained by the cooperative nature of the deformation of macrochain agglomerates in the melt flow [15, 20].

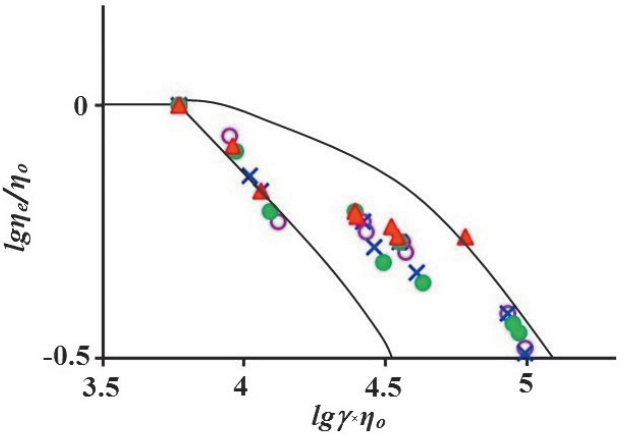


Fig. 4. Universal temperature-invariant characteristic of the viscosity properties of LDPE$_{(50)}$/HDPE$_{(50)}$ and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$.

Рис. 4. Универсальная температурно-инвариантная характеристика вязкостных свойств композитов из LDPE$_{(50)}$/HDPE$_{(50)}$, а также LDPE$_{(50)}$/HDPE$_{(50)}$ и 1% масс. TiO$_2$

The use of temperature-invariant characteristic greatly simplifies calculations for measuring the melt viscosity in a wide range of temperatures, velocities, and shear stresses, and thus makes it possible to characterize the state of polymers in the steady flow regime. Extrapolation of these curves to the region of high shear rates makes it possible to give you rough estimate the effective viscosity of the melt, which is close to the conditions of polymer processing.

CONCLUSIONS

The effect of a structure-forming agent (1 wt% TiO$_2$), temperature, and shear stress on the regularity of changes in the effective viscosity and shear rate of an LDPE$_{(50)}$/HDPE$_{(50)}$ polymer blend has been studied. It has been established that when a structure-forming agent former is loaded into the polymer mixture, the effective viscosity decreases to some extent. This circumstance can facilitate the processing polymeric materials and increase the productivity of the unit. The “apparent” activation energy of the viscous flow is determined. The activation energy of the viscous flow was found to decrease with the inclusion of a structure-forming agent. To obtain more complete information about the polymer state in relation to their processing by injection molding and extrusion, a universal temperature-invariant characteristic of the viscosity of LDPE$_{(50)}$/HDPE$_{(50)}$ and LDPE$_{(50)}$/HDPE$_{(50)}$ + 1 wt% TiO$_2$ has been constructed.

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