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ОПРЕДЕЛЕНИЕ РЕОЛОГИЧЕСКИХ СВОЙСТВ РАСТВОРОВ ПОЛИЭТИЛЕНОКСИД - ВОДА

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В статье представлены результаты исследований реологических свойств водных растворов полиэтиленоксида. На основании измерений, проведенных с использованием ротационного вискозиметра, были определены значения касательных напряжений в относительно широком диапазоне скоростей сдвига, возникающих при смешивании ньютоновских и неньютоновских жидкостей в аппаратах с механическими перемешивающими устройствами. Реологические кривые аппроксимированы моделью Оствальда де Виля (или так называемым степенным законом). Коэффициенты модели: коэффициент консистенции жидкости k и индекс течения n были определены с использованием алгоритма Левенберга-Марквардта для нелинейной оценки. Также определено влияние температуры в диапазоне 15-40 °C на свойства и поведение исследуемых неньютоновских жидкостей. Для характеристики этой зависимости был определен параметр сдвига кривой a_t. Наибольшие значения a_t наблюдаются при самой низкой температуре, а наименьшие при 40 °C. Эксперименты показали значительное влияние концентрации полиэтиленоксида c_{PEO} на реологические свойства исследуемых растворов. Для самых низких концентраций (c_{PEO}=1,2%) растворы проявляли свойства, характерные для ньютоновских жидкостей, значения n были близки к 1. С увеличением концентрации полиэтиленоксида в воде (c_{PEO}=2,4-4,8%) растворы проявляли свойства псевдопластичных неньютоновских жидкостей без предела текучести. В этих случаях значения n были ниже единицы и для наибольшей концентрации (c_{PEO}=4,8%) находились в диапазоне n=0,5694-0,7536 в зависимости от температуры. Температура изменялась в диапазоне 15-40 °C. Результаты исследований могут быть использованы при численном моделировании, проектировании и оптимизации промышленного оборудования, работающего с жидкостями такого рода, включая смесительные емкости, колонны или теплообменники.

Ключевые слова: реологические свойства, неньютоновские жидкости, вязкость жидкости, полиэтиленоксид, модель Оствальда-де Виля

IDENTIFICATION OF RHEOLOGICAL PROPERTIES OF POLY(ETHYLENE OXIDE) – WATER SOLUTIONS

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In the paper results of investigations of rheological properties for selected PEO-water solutions are presented. On the basis of measurements, carried out with use of rotational viscosimeter values of shear stresses were determined in the relatively wide range of shear rates. Rheological curves were described by the Ostwald de Waele model (or so-called power-law). The model coefficients such as the fluid consistency coefficient k and the flow behavior index n were determined using Levenberg‒Marquardt algorithm for nonlinear estimation. The influence of temperature on properties and behavior examined non-Newtonian fluids was also determined. Results were processed in the curve shift parameter a_t***. Experiments shown a significant effect of poly(ethylene oxide) concentration*** c_{PEO} ***on rheological properties of examined solutions. For the lowest concentration (***c_{PEO}***=1.2%) solutions exhibited properties similar to Newtonian fluids with values of n close to 1. With increasing of PEO concentration in water (***c_{PEO}***=2.4-4.8%), solutions exhibited properties as non - Newtonian fluids, pseudoplastic, without yield limit. In these cases values of n were below unity and for the highest concentration (***c_{PEO}***=4.8%) belonged to the range of n=0.5694-0.7536, depending on the temperature. Results of investigations can be used during numerical simulations, design and optimization of industrial equipment, working with fluids of this kind, including mixing vessels, columns or heat exchangers.***

Key words: rheological properties, non-Newtonian fluids, fluid viscosity, poly(ethylene oxide), PEO, Ostwald de Waele model

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INTRODUCTION

The non-Newtonian fluids are widely used in the food, cosmetics, pharmaceutical and plastic industries [1, 2]. To correctly select the design and technical parameters of devices, in which processes with such fluids are carried out (e.g. mixers, columns, heat exchangers) it is important to found their rheological properties in the broad range of process parameters.

Literature [3, 4] describes methods for measuring of rheological properties of non-Newtonian fluids. A number of models describing their rheological characteristics was also presented.

Issues related to the practical application of selected rheological models have been the subject of many papers for a long time [5-18]. This is mainly due to the large variety of non-Newtonian fluids (emulsions [5-8], suspensions [8, 9], solutions [10], polymer mixtures [11] etc.), their various properties [11-14] which often change with parameters of technological process, e.g. temperature [10, 11, 14, 15], flow conditions [7, 11, 16], mixing intensity [17, 18] etc. Publications contain important information regarding non-Newtonian fluids, especially theoretical foundations their rheology and phenomena occurring both in liquids and during their flow and processing.

As previously mentioned, non-Newtonian liquids can have different properties (pseudoplastic, yield-pseudoplastic, Bingham-plastic, dilatant fluids) and exhibit different behaviors, e.g. in flow processes. One of such liquids, often used as an emulsifier in the production of emulsions and as an agent for increasing the viscosity of liquid cosmetics and medicines, is an aqueous solution of poly(ethylene oxide) (PEO). It is a completely biodegradable liquid, which is not insignificant, especially for environmental protection.

Experimental studies with the use of PEO solutions are described, e.g. in [19-21]. In [19] various mixtures of polycarbonate and poly(ethylene oxide) were tested, with a PEO content in a relatively wide range (5 to 30% by weight). The impact of its addition on the polycarbonate flow properties was analyzed. It has been found that viscosity of the mixtures decreases as the shear rate increases, and the decrease is the greater for the higher poly(ethylene oxide) content. The authors of [20] studied rheology of emulsions with a temperature-sensitive microstructure, which were produced using star polymers of poly(ethylene oxide) as emulsifiers. It was confirmed that the addition of PEO was an effective and efficient emulsifier that stabilizes emulsions. In paper [21] results of studies on rheological properties of suspensions of PEO and carboxymethyl cellulose (CMC) with high molecular weight were presented. The authors analyzed the influence of solid particles with various particle volume fractions on properties of mixtures. Their rheological properties were described with the Cross model.

As analysis of literature showed that non-Newtonian fluids have a much more complicated structure than classic, Newtonian liquids. The variability of their properties and strong dependence on process conditions significantly complicates the description of their behavior and greatly limits – and sometimes even prevents – the use of classic theories and rules, e.g. in relation to momentum, mass and energy transport processes.

The use of theoretical rheological models for some non-Newtonian fluids does not bring the expected results. In many cases, the only solution is to carry out laborious, laboratory tests to determine individual rheological characteristics. Only on this basis, we can choose the right rheological model, describing properties of the tested liquid in a relatively wide range.

RESEARCH MOTIVATION

The paper presents results of the first stage of research on mixing non-Newtonian fluids in mechanical mixers [22, 23]. The developed correlations and rheological characteristics, describing properties of aqueous solutions of poly(ethylene oxide) in a relatively wide range of temperatures, shear rates and concentrations, will allow – in the next stages of research – to select of appropriate control and measuring equipment and also the creation of correct theoretical, numerical and empirical models for processes of non-Newtonian fluids mixing in mechanical mixers.

EXPERIMENTAL PART

The measurement of properties of poly(ethylene oxide) – water solutions were carried out for PEO concentration: c_{PEO} = 1.2; 2.4; 3.6 and 4.8%, and in the temperature range of t = 15-40 °C. The density of solutions ρ (kg·m^{-3}) was measured using density meter Anton Paar DMA-38A. Determination of viscosity dependencies in form of relation between shear stress τ (Pa) and shear rate $\dot{\gamma}$ (s^{-1}) was conducted with the use of rotational viscometer Haake VT 550. The temperature was stabilized by thermocontroler Thermo Haake DC 10. Shear rates were changed in the range of $\dot{\gamma}$ = 50-2000 s^{-1}. This range fully covers shear rates occurring during mixing Newtonian as well as Non-Newtonian fluids in mechanically agitated mixing vessels [24-27].

RESULTS AND DISCUSSION

The values of PEO-water solutions density, ρ, for PEO concentration in the range of c_{PEO} = 1.2-4.8% and temperature t = 15-40 °C are listed in Table 1.

Table 1

Values of density ρ for PEO-water solutions

***Таблица 1.* Значения плотности ρ растворов полиэтиленоксид-вода**

PEO concentration, c_{PEO} (%)	PEO-water solution density, ρ (kg·m^{-3})					
	Temperature, t (°C)					
	15	20	25	30	35	40
1.2	999.1	998.6	997.8	996.9	995.6	993.3
2.4	999.2	998.7	998.2	997.2	996.2	995.3
3.6	999.4	998.9	998.2	997.1	996.2	995.1
4.8	999.5	999.1	998.5	997.5	996.6	995.2

The measurements showed that changes in the density of tested solutions with PEO concentration and temperature are small. They have a similar qualitative and quantitative tendencies as for water. The effect of temperature is more visible (decrease in density with increase in t), while the effect of concentration is not significant (slight increase in density with increase in c_{PEO}).

Viscosity characteristics for solutions are more complex. Flow curves were created on the basis of measurements. Data set was described with relation $\tau = f(\dot{\gamma})$, for various PEO concentrations in different solution temperatures. Selected trends of this relationship are presented in Fig. 1.

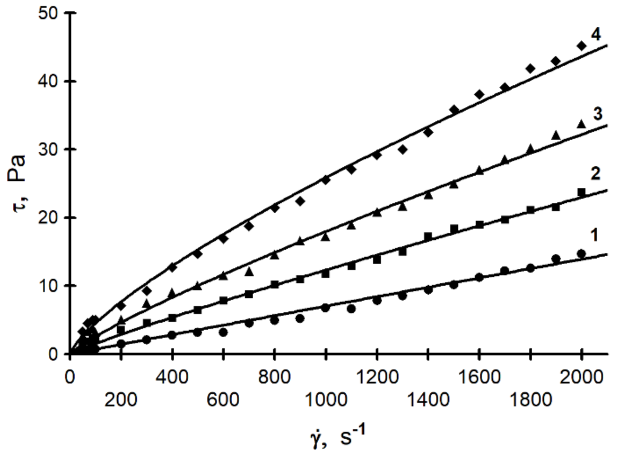

Fig. 1. Relation τ vs. γ for selected PEO-water solutions: 1 – c_{PEO}=1.2; 2 – c_{PEO}=2.4; 3 – c_{PEO}=3.6 and 4 – c_{PEO}=4.8 (%) (symbols – measurement, lines model, t=20 (°C))

Рис. 1. Зависимость касательного напряжения τ от скорости сдвига γ для растворов полиэтиленоксид-вода различных концентраций: 1 – c_{PEO}=1,2; 2 – c_{PEO}=2,4; 3 – c_{PEO}=3,6 и 4 – c_{PEO}=4,8 % (символы – опытные данные, линии – расчет по модели, t=20 °C)

An analysis of all measurement data showed, that solutions of poly(ethylene oxide) in water are non-Newtonian liquids and their properties depend on the concentration of PEO. For concentrations in the range of c_{PEO} = 2.4-4.8%, the flow curves show characteristic courses for pseudoplastic fluids (without yield value limit), while the exception is the concentration of c_{PEO} = 1.2%, for which measuring points lie down along a straight line.

Quantitative data analysis was performed using the empirical Ostwald de Waele (the power-law) model. He is one of the basic mathematical, rheological models. This simple, two-parameter power relationship, with high accuracy describes flow curves of non-Newtonian liquids, in a relatively wide range of their variability and shear rates.

$$\tau = k\dot{\gamma}^n \tag{1}$$

where: k and n are empirically determined the fluid consistency coefficient and the flow behavior index, respectively.

It should be noted, that the relationship Ostwald de Waele (Eq. 1) is not a rheological equation of state, but an empirical interpolation formula (mathematical model). However, its uncomplicated forms, high accuracy of description of the rheology for different types of liquids in a wide range of shear rates as well as flexibility of use, makes it often used to characterize and describe flow of non-Newtonian fluids in various industrial devices, including mixers [24, 27, 28].

In Table 2 are listed values of coefficients in Eq.1, determined for PEO-water solutions for various values and PEO concentrations and temperatures.

The values of constants and exponents in correlation were determined with Statistica 9.1 software and the Levenberg–Marquardt algorithm (nonlinear estimation). The loss function was estimated using the least-squares method.

Estimation precision was assessed by calculating the coefficient of determination (R) and the mean relative error ($\bar{\Delta}$). For all cases R was in the range of R = 0.98-0.99. The relative error did not exceed 15 (%) and most of values were in the range of $\bar{\Delta}$ = 5-10%, which testifies to the correctness of chosen mathematical model.

Data presented in Table 2 confirms previous qualitative analysis. Values of the flow behavior index n are various and depend on PEO concentrations and temperature. For the lowest concentration c_{PEO} = 1.2% the values of the flow behavior index are close to 1 (n = 1.0317-0.9496), which indicates, that the liquid has similar properties to Newtonian fluid. In the range of concentrations c_{PEO} = 2.4-4.8% values n are below 1 and decrease both with increasing PEO concentration and temperature. Solutions show typical properties for pseudoplastic non-Newtonian fluids, with the largest deviations from Newtonian fluid for the highest PEO concentration (n = 0.7536-0.5694. A consistency coefficient k takes values the order of 10^{-3}-10^{-1} and increases with increasing PEO concentration.

Table 2

Values of coefficients in Eq. (1)

***Таблица 2.* Значения коэффициентов в уравнении (1)**

PEO concentration, c_{PEO} (%)	Temperature, t (°C)	Fluid consistency coefficient, k (Pa·s^n)	Flow behavior index, n	Mean value of relative error, $\bar{\Delta}$ (%)
1.2	15	0.0079	1.0317	10.7
	20	0.0082	0.9811	14.9
	25	0.0079	0.9792	14.3
	30	0.0081	0.9496	8.7
	35	0.0057	0.9518	10.1
	40	0.0049	0.9610	11.5
2.4	15	0.0292	0.9291	11.8
	20	0.0251	0.8973	13.8
	25	0.0448	0.7805	8.2
	30	0.0576	0.7300	5.2
	35	0.0531	0.7297	5.6
	40	0.0495	0.7291	4.5
3.6	15	0.0707	0.8543	13.1
	20	0.0545	0.8396	12.8
	25	0.0996	0.7280	10.1
	30	0.1392	0.6633	5.6
	35	0.1480	0.6439	5.1
	40	0.1369	0.6455	4.6
4.8	15	0.2159	0.7361	9.1
	20	0.1420	0.7536	13.6
	25	0.2440	0.6505	10.5
	30	0.3254	0.5931	6.7
	35	0.3570	0.5694	5.1
	40	0.2999	0.5845	8.6

Visible deviations from presented rules for some cases are small and result from a two-parameter approximation of experimental data with a mathematical model, so the strength of their impact in each case should be considered together, which is also suggested in the literature [1, 3].

The influence of temperature on viscosity properties of solutions was characterized by the curve shift parameter a_t, determined as [2]:

$$a_t = \frac{\tau(\dot{\gamma})}{\tau(\dot{\gamma})_R} \tag{2}$$

where: $\tau(\dot{\gamma})$ are values obtained at a given temperature, and $\tau(\dot{\gamma})_R$ are values determined at a reference temperature (for this study t_R = 20 °C).

Example changes in flow curves for different temperatures are shown in Fig. 2, while mean values of the curve shift parameter are listed in Table 3.

As it is seen, for the smallest temperatures (t = 15 °C) values of shear stresses are correspondingly higher than those at the reference temperature in proportion a_t = 1.25-1.39, while for higher temperatures correspondingly lower, reaching for t = 40 °C even values of the order of a_t = 0.63-0.72.

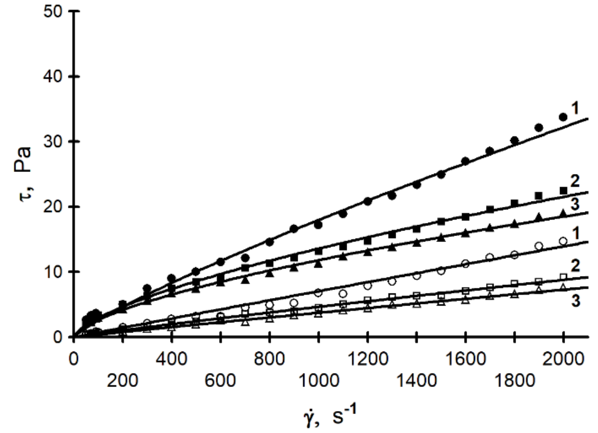

Fig. 2. Relation τ vs. γ for selected PEO-water solutions (c_{PEO}=1.2 (%) - empty symbols, c_{PEO}=3.6 (%) - filled symbols). Temperature, °C: 1 – 20; 2 – 30; 3 – 40

Рис. 2. Зависимость касательного напряжения τ от скорости сдвига γ для выбранных растворов полиэтиленоксид-вода при различных температурах: (c_{PEO}=1,2 % – пустые символы, c_{PEO}=3,6 % – залитые символы). Температура, °C: 1 – 20; 2 – 30; 3 – 40

Table 3

Mean values of the curve shift parameter, a_t

***Таблица 3*. Средние значения параметра сдвига кривой, a_t**

PEO concentration, c_{PEO} (%)	Mean values of the curve shift parameter, a_t					
	Temperature, t (°C)					
	15	20	25	30	35	40
1.2	1.32	-	0.95	0.83	0.68	0.63
2.4	1.39	-	0.88	0.82	0.77	0.71
3.6	1.31	-	0.86	0.78	0.73	0.67
4.8	1.25	-	0.88	0.81	0.76	0.72

It should also be noted that in this study results were processed in the form of a standard functional relation $\tau = f(\dot{\gamma})$. However, on their basis, it is very easy to determine the dependence on the apparent viscosity η_a, in the form of:

$$\eta_a = k\dot{\gamma}^{n-1} \quad (3)$$

which in some cases may be more useful.

In addition to obvious regularities, e.g., such as an increase in shear stresses and apparent viscosities with an increase of PEO concentration, it is difficult to unequivocally indicate exact theoretical basis of above described changes in rheological characteristics of tested solutions. This is mainly due to the unusual properties of PEO – water solutions – especially their structure and clustering behavior [29]. Some authors stated that depending on the concentration in solutions polymer clusters (or aggregates) coexist in equilibrium with free polymer coils [30]. Others found that PEO can exists as two phases, with different polymer concentration. The polymer reach phase organizes into a liquid crystalline fibrillary network that leads to properties such as shear thinning end elasticity [31].

With such a complex internal structure of poly(ethylene oxide) – water solutions, seems to be a reasonable physical interpretation, which assumes that as the shear rate increases, the asymmetrical particles or molecules undergo gradual ordering and arrange along the flow line, which results in a decrease in apparent viscosity. However, creating a correct, theoretical description of the rheology of PEO-water solutions still requires a number of studies and analyzes, because – so far – results of experimental studies of such solutions significantly differ from the theoretical relationships commonly used in the theory of polymers [29].

CONCLUSION

The goal of the study was to determine rheological properties of poly(ethylene oxide) - water solutions, for various PEO concentrations and at various temperatures. Test results were processed in the form of viscosity curves, using the two-parameter Ostwald de Wale model. The values of determined regression coefficients show that solutions are non-Newtonian fluids (pseudoplastic, shear-thinning), with the exception of the solution with the lowest concentration, for which the flow curve, is similar to that of Newtonian fluids. The results will be used during investigations of mixing non-Newtonian fluids in mechanical mixers.

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