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Rheological Behavior of Concentrated Solutions EPDM of Some Viscosity Improvers in SAE 10W-40 Mineral Oil

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Author's contribution

This whole work was carried out by the author IS.

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ABSTRACT

Rheology of polymer concentrated solutions represents a cross-disciplinary field, using wide spectra of theoretical tools from physics and chemistry. They effectively thicken the oil at all temperatures, but the increase of viscosity is more pronounced at high temperatures. The lubricating effect is extends across a wider temperature range and the oil becomes thus a multi-grade one. Its viscosity still decreases logarithmically with temperature, but the slope representing the change is lessened. This slope is dependent on the nature and amount of additive to the base oil. The purpose of this study was to obtain automotive multi-grade oils. They have a number of advantages such as easy starting cold engine, reducing wear and decrease the formation of deposits in the engine. Multi-grade oil can be used longer than the engine base oils because they are more highly refined and contain large proportions of additives. The rheological behaviour of the solutions was determined using a Haake VT 550 Viscometers developing shear rates ranging between 3 and 1312 s⁻¹ and measuring viscosities from 10⁴ to 10⁶ mPa.s when the HV₁ viscosity sensor is used. Rheological measurements of 3; 6; 10 and 12% EPDM solutions in SAE 10W-40 mineral oil show non-Newtonian behaviour in the temperature range 313-370 K and shear rates ranging between 3 and 1312 s⁻¹. The lower slope is given by 3% solution, followed by 12%, and the highest one by 10% solutions. EPDM solutions present an increase of slope when passing from 3 to 6% and a decrease when the concentration exceeds the last value. The lowest slope was obtained for solution having the concentration 12%, followed by 3%, while 6% solution has the highest value.

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This suggests that EPDM can be a better viscosity improver for the mineral oil SAE 10W-40, both at low and high concentrations.

Keywords: Rheological; concentrated; solutions; viscosity; multi-grade oils.

1. INTRODUCTION

Rheology of polymer concentrated solutions represents a cross-disciplinary field, using wide spectra of theoretical tools from physics and chemistry [1-3]. For physicists, understanding the configuration and dynamics of long polymer chains has been a significant source of problems within statistical physics from the 1950's onwards. One of the reasons why physicists were drawn to the problem is the universality of polymer properties [4-8]. Within the time and length scales much exceeding the atomic ones, universal theories have been built, well describing the main features in the polymer behavior, insensitive to the details of the chemistry of the chains. Among these theories the most popular are the Rouse and Zimm models, in which the polymer is represented as a chain of beads under Brownian motion [9-12].

Additive effectively thickens the oil at all temperatures, but the increase of viscosity is more pronounced at high temperatures. The lubricating effect extends across a wider temperature range and the oil becomes thus a multi-grade one. Its viscosity still decreases logarithmically with temperature, but the slope representing the change is lessened. This slope is dependent on the nature and amount of additive to the base oil [13-15].

The purpose of this study was to obtain automotive multi-grade oils. They have a number of advantages such as easy starting cold engine, reducing wear and decrease the formation of deposits in the engine. Multi-grade oil can be used longer than the engine base oils because they are more highly refined and contain large proportions of additives. Rheological behavior and viscosity index properties are of great importance in terms of operation and fuel consumption of an engine: the oil viscosity increases the multi-grade oil consumption is lower [16-21].

The object of this paper is to determine the rheological behaviour of some concentrated solutions of copolymer EPDM produced by DSM Elastomers Europe B.V. and recommended as viscosity improvers for multi-grade mineral oils at shear rates ranging between 3 and 1312 s⁻¹ and temperatures between 40 and 100°C, to estimate their efficiency as lubricating additives for the low viscosity mineral oil SAE 10W-40.

2. MATERIALS AND METHODS

The following copolymer was used as viscosity improvers: ethylene-propylene-(ter) polymer (EPDM) product on DSM Thermoplastic Elastomers Europe B.V. The low viscosity oil SAE 10W-40 (INCERP, Romania) was used as mineral oil.

Copolymer EPDM is recommended for plastics modification and oil modification, for application in automotive, construction, wire and cable and general rubber good. The chemical and physical properties of Copolymer EPDM are: physical state - solid, form - bales or granulate, colour – natural opaque, brown in case of oil extended grades, odour – weak paraffinic, relative density 860-900 Kg.m⁻³, bulk density depending on bale or granulate

structure, insoluble in water, soluble in hydrocarbons such as (alkanes: hexane, heptanes, octane, decane, dodecane, iso-octane, isododecane, cycloalkanes: cyclo-octane, decaline, cyclododecane, aromatic substances: butyl benzene, octylbenzene, and oil: paraffinic naphthenic, aromatic. Typical of ethylene-propylene number-average molecular weight (4-20) 10000, molecular weight (20-40) 10000, visco-average molecular weight of the wind 10-40 million. Their ratio, which can be taken as a measure of copolymer polydispersity, is 2.37. The composition copolymer of a EPDM is: 45% propylene, 52.5% ethylene and 2.5% diene monomer.

The properties physico-chemicals oil SAE10W-40 are: density $0.872 \cdot 10^3 \text{ kg.m}^{-3}$, kinematic viscosity at 40°C – 108.5 cSt, kinematic viscosity at 100°C – 15.4cSt, viscosity index – 149, viscosity-temperature coefficient (VTC) – 0.8580, CCS viscosity at -25°C – 6270 cP, flash point – 220°C , pour point - -37°C , sulfated ash – 0.86%, neutralization No. (TBN-E) – 7.1 and color 2.

The dissolution of the copolymer was performed at room temperature with continuous shaking for several weeks.

Solutions has the concentrations 3; 6; 10 and 12 g/dL were prepared EPDM requires much more time for complete dissolution. Concentration range indicated by the importing firm that is between 1 and 12% and temperature range have chosen to follow the behaviour of the polymer in the engine. The range of 1-3% concentrations or could not make determinations with Viscometer used.

The rheological behaviour of solutions was determined using a Haake VT 550 Viscotester developing shear rates ranging between 3 and 1312 s^{-1} and measuring viscosities from 10^4 to 10^6 mPa.s when the HV₁ viscosity sensor is used. The solutions concentrated of the copolymers were investigated in the temperature range of $40 - 100^\circ\text{C}$. The accuracy of the temperature was $\pm 0.1^\circ\text{C}$.

To calculate the value of the shear stress the following equation is used:

$$\sigma = Z \cdot \alpha \quad (1)$$

where represents the constant of the rotating cylinder. The z value depends on the sizes of the cylinders and on the constant of action for the spring, chosen from the apparatus constant sheet; α – factor that is read after each determination. The accuracy of measuring shear stress was $\pm 1\%$

3. RESULTS AND DISCUSSION

The Fig. 1 shows dependence dynamic viscosity on absolute temperature for oil SAE 10W-40 without additives. The dynamic viscosity of oil decreases exponentially with increasing absolute temperature by an equation of the form (2):

$$\eta = 16.25522 + 2.50576E11 \exp(-T/14.51402) \quad (2)$$

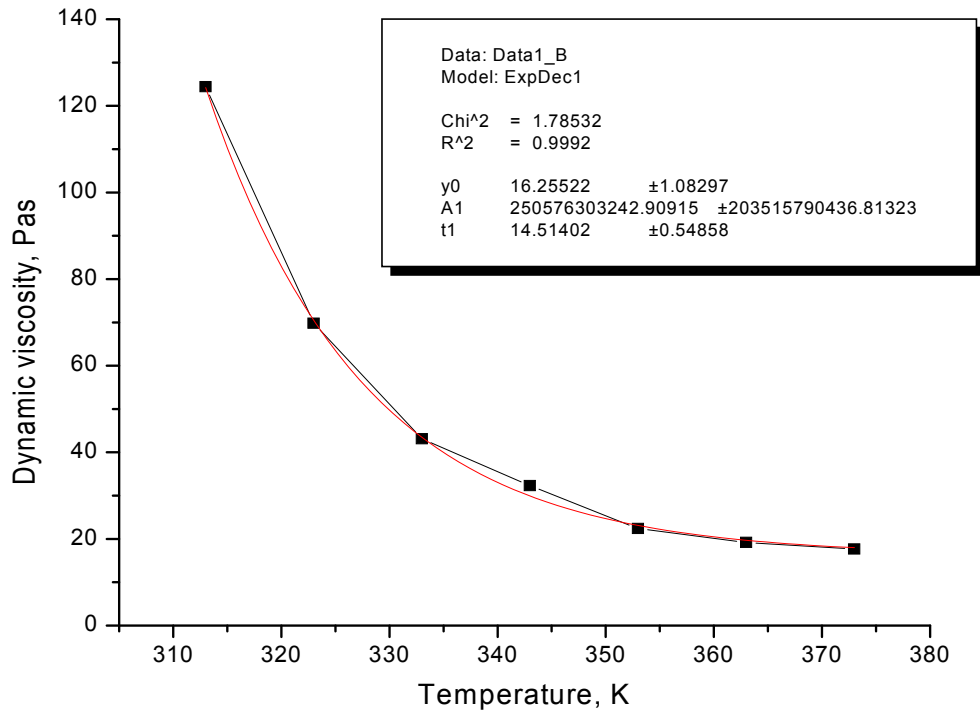


Fig. 1. The dependence dynamic viscosity – absolute temperature for oil SAE 10W-40

The correlation coefficient is $R^2 = 0.9992$.

The rheograms obtained for the 3; 6; 10 and 12% EPDM solutions for shear rates ranging between 3 and 1312 s^{-1} were analysed according to the models that describe the deviations from the Newtonian behaviour [9,13]:

Bingham:

$$\tau = \tau_o + \eta(dy/dt) \tag{3}$$

Casson:

$$\tau^{1/2} = \tau_o^{1/2} + \eta^{1/2}(dy/dt)^{1/2} \tag{4}$$

Ostwald-de Waele:

$$\tau = k (dy/dt)^n \tag{5}$$

and Herschel-Bulkley:

$$\tau = \tau_o + k\eta(dy/dt)^n \tag{6}$$

where τ is the shear stress, τ_o – yield stress, η - viscosity, (dy/dt) - shear rate, n – flow index and k – index of consistency.

The rheograms of 3% EPDM solution at the specified temperatures and shear rates are shown in Fig. 2.

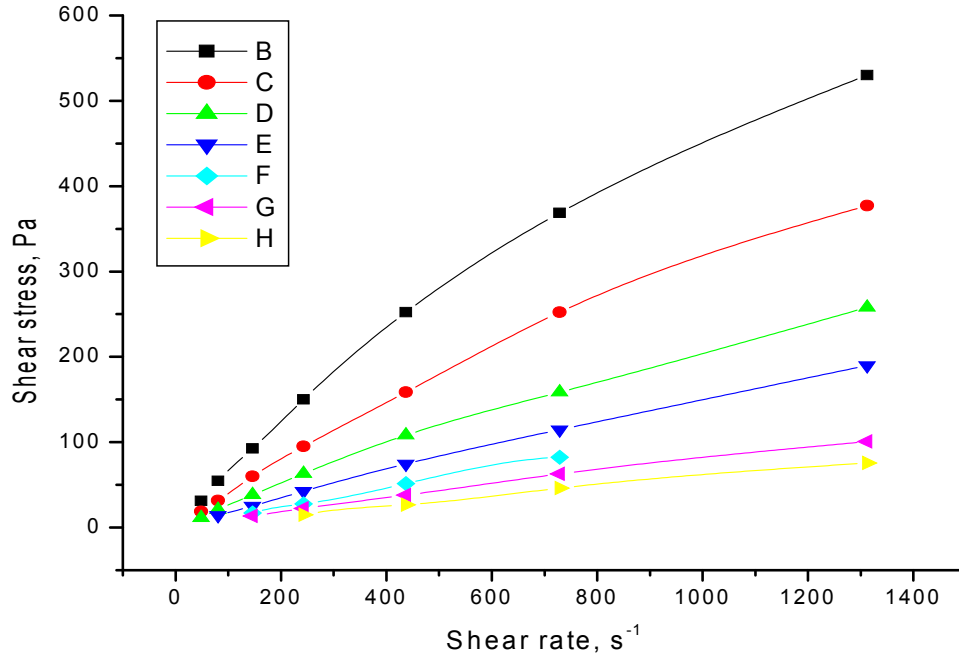


Fig. 2. Rheograms of 3% EPDM solution at: B – 313K; C – 323K; D – 333K; E – 343K; F – 353K; G – 363K and H – 370 K

The viscosities of EPDM solutions, the studied temperatures and shear rates behave as pseudoplastic fluids, following the Herschel-Bulkley model. Thus, the rheograms of 3% copolymer solution, shown in Fig. 1, indicate a pseudoplastic behaviour regardless the temperature. The higher the temperature, the less pronounced the pseudoplastic behaviour as expected, reflected in the value of the flow. The flow rate of the solution at the temperatures of 313 and 323 K, which indicates the amount of 0.93 - in addition - a more pronounced pseudoplastic behaviour.

The value of shear rate for thinning of EPDM solution 3 % for the temperatures are: 81 s⁻¹ for 343 K, 145.8 s⁻¹ for 353 K. The concentrate solution 3% EPDM are: z is 1.14 and then α is between 15.5 and 95.

Viscosity index oil SAE 10W-40 is 149 (ASTM 2270-93) and VTC for 0.8580. The kinematic viscosity of the concentrate solution was 131.3 cSt at 100°C and 900 cSt at 40°C.

Put the focus viscosity index of 3% solution is 106 times higher than SAE 10W-40 oil. VTC of the solution is 0.8541, down 0.0039 times.

Table 1 presented absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution of concentration 3 g/dL.

The model proposed to describe the dependence of shear stress vs. shear rate for the concentration of solutions of 3%, 6%, 10% and 12% is described by equation (7):

$$\tau = A + B(dy/dt) + C(dy/dt)^2 \quad (7)$$

The parameters A, B and C were obtained by fitting a polynomial solution concentration rheogram 3% absolute temperature of 313 K: A = 6.39202, B = 0.63535 and C = -1.80371E-4

Table 1 shows the data values of the correlation coefficients determined for each partial model rheological obtained by linear regression obtained from all seven rheograms temperatures at which the tests were performed. Although the values of correlation coefficients have values close enough for all four rheological models, the highest values are obtained Herschel-Bulkley model yet.

Table 1. Absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution 3 g/dL

Value coefficient correlation, R ²					
Temperature, K	Model Bingham calculated with eq. (3)	Model Casson calculated with eq. (4)	Model Ostwald-de Waele calculated with eq. (5)	Model Herschel-Bulkley calculated with eq. (6)	Model proposed calculated with eq. (7)
313	0.9870	0.9869	0.9919	0.9922	0.9997
323	0.9929	0.9955	0.9928	0.9979	0.9997
333	0.9958	0.9964	0.9958	0.9974	0.9992
343	0.9981	0.9985	0.9980	0.9992	0.9996
353	0.9994	0.9966	0.9996	0.9998	0.9963
363	0.9979	0.9987	0.9994	0.9998	0.9997
370	0.9978	0.9982	0.9976	0.9987	0.9995

Table 2 shows the yield stress, flow index, index of consistency and correlation coefficients for the solution of 3% in the temperature range 313-370 K determined by equation (6).

Doubling of copolymer concentration reduce very much the shear rate range on which the measurements can be done, excepting the last two temperatures, as can be seen in Fig. 3. In the temperature range 363-370 K is not observed significant differences in rheological behaviour of concentrated solutions of copolymer. Melt index is much higher at 363K compared to that obtained at 370 K. Solution viscosity decrease with increasing shear rate can be explained by the alignment of the polymer molecules in the direction of shear force to shear velocities mentioned, which has the effect of thickening when they are randomly distributed. The higher the temperature, the greater the force required to align molecules for temperature. Decreasing thinning with increasing shear rate viscosity returns to the previous value and rheograms obtained with increasing shear rate overlap. The value of shear rate for thinning of solution EPDM 6 g/dL for the temperatures are: 48.6 s⁻¹ for 333 and 343 K, 145.8 s⁻¹ for 363 K. The concentrate solution 6% EPDM are: z is 1.14 and then α is between 24 and 97.5.

Table 2. Absolute temperature, yield stress, flow index, index of consistency and value coefficient correlation of the model described by equations (6) for the solution 3 g/dL

Temperature, K	Yield stress, τ_0	Flow index, n	Index of consistency, k	Correlation coefficient, R^2
313	1.3841	0.8442	2.2361	0.9922
323	2.5455	0.5190	2.5435	0.9979
333	2.3701	0.5459	2.3721	0.9974
343	2.4870	0.2837	2.4868	0.9992
353	2.4985	0.1830	2.4335	0.9998
363	2.6329	0.1045	2.6331	0.9998
370	2.4183	0.1366	2.4247	0.9987

The kinematic viscosity of the concentrated solution was 1110 cSt at 100°C and 7498.7 cSt at 40°C.

The viscosity index of concentrated solution 6% is 236 times higher than SAE 10W-40 oil. VTC of the solution is 0.8520, down 0.006 times.

Table 3 presented absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution of concentration 6 g/dL.

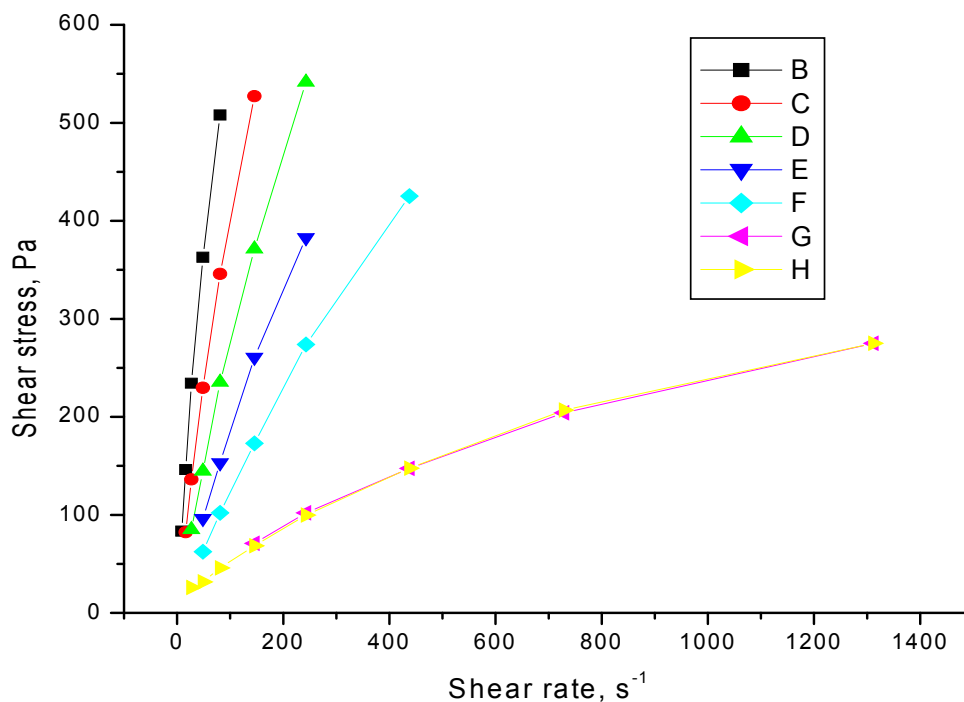


Fig. 3. Rheograms of 6% EPDM solution at: B – 313K; C– 323K; D – 333K; E – 343K; F – 353K; G – 363K and H– 370 K

In the case of 10% EPDM solution the temperature range on which the measurements were possible narrowed to 323-363 K and the shear rate one to 3-48.6 s⁻¹, as Fig. 3 shows, because of the great increase of solution viscosity. Its pseudoplastic behaviour is more accentuated compared with a 6% solution (the flow index is decreased from 0.93 to 0.87). The concentrated solution 10% EPDM are: z is 1.14 and then α is between 32 and 98.3.

The kinematic viscosity of the concentrated solution was 2414 cSt at 100°C and 16321 cSt at 40°C.

The viscosity index of concentrated solution 10% is 285 times higher than SAE 10W-40 oil. VTC of the solution is 0.8520, down 0.006 times.

Table 5 presented absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution of concentration 10 g/dL.

Table 3. Absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution 6 g/dL

Temperature, K	Value coefficient correlation, R ²				
	Model Bingham calculated with eq. (3)	Model Casson calculated with eq. (4)	Model Ostwald-de Waele calculated with eq. (5)	Model Herschel-Bulkley calculated with eq. (6)	Model proposed calculated with eq. (7)
313	0.9902	0.9937	0.9902	0.9972	0.9995
323	0.9930	0.9958	0.9930	0.9987	0.9998
333	0.9943	0.9952	0.9942	0.9954	0.9996
343	0.9962	0.9974	0.9962	0.9986	0.9999
353	0.9950	0.9965	0.9948	0.9984	0.9999
363	0.9775	0.9774	0.9979	0.9988	0.9995
370	0.9809	0.9876	0.9926	0.9949	0.9992

Table 4 shows the yield stress, flow index, index of consistency and correlation coefficients for the solution of 6% in the temperature range 313-370 K determined by equation (6).

Increasing of concentration at 12% reduces more the temperature range of measurements, to 333-363 K, as can be seen in Fig. 5 and increases the pseudoplastic behaviour, the flow index decreasing at 0.84. Table 7 presented absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution of concentration 12 g/dL.

The kinematic viscosity of the concentrate solution was 3065 cSt at 100°C and 20798 cSt at 40°C.

The concentrated solution EPDM 12% are: z is 1.14 and then α is between 15 and 102.

The viscosity index of concentrated solution 12% is 299 times higher than SAE 10W-40 oil. VTC of the solution is 0.8518, down 0.0062 times.

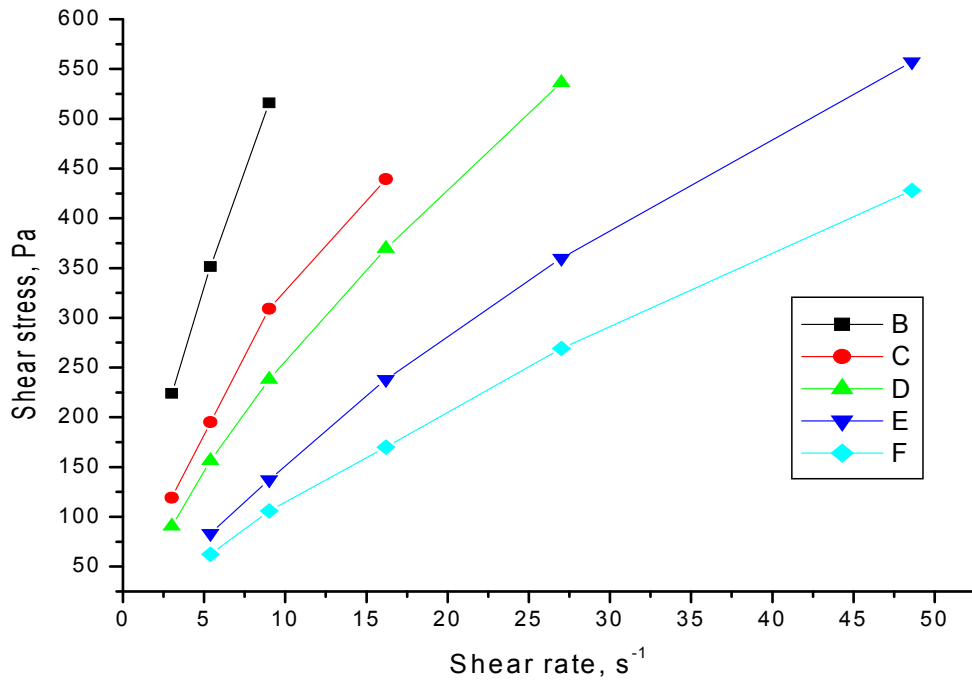


Fig. 4. Rheograms of 10% EPDM solution at: B– 323K; C– 333K; D – 343K; E– 353K and F - 363K

Table 4. Absolute temperature, yield stress, flow index, index of consistency and value coefficient correlation of the model described by equations (6) for the solution 6 g/dL

Temperature, K	Yield stress, τ_0	Flow index, n	Index of consistency, k	Correlation coefficient, R^2
313	2.6692	0.8240	0.0693	0.9972
323	2.1027	0.8458	0.1221	0.9987
333	1.6876	0.8459	0.1849	0.9954
343	1.2334	0.8626	0.2913	0.9986
353	0.7559	0.8777	0.4695	0.9984
363	1.2067	0.6193	0.1405	0.9988
370	1.0425	0.6436	0.3525	0.9949

Table 6 shows the yield stress, flow index, index of consistency and correlation coefficients for the solution of 10% in the temperature range 313-370 K determined by equation (6).

The dynamic viscosity of most materials, including polymer solutions and polymer melts well above their glass transition temperatures, decreases with temperature in accordance to Andrade equation [13]:

$$\eta = A \cdot 10^{B/T} \tag{8}$$

where A and B are constants characteristic of the polymer and T is the absolute temperature.

Table 5. Absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution 10 g/dL

Value coefficient correlation, R ²					
Temperature, K	Model Bingham calculated with eq. (3)	Model Casson calculated with eq. (4)	Model Ostwald-de Waele calculated with eq. (5)	Model Herschel-Bulkley calculated with eq. (6)	Model proposed calculated with eq. (7)
323	0.9991	0.9969	0.9969	0.9992	1.0000
333	0.9887	0.9927	0.9886	0.9963	0.9998
343	0.9953	0.9969	0.9953	0.9986	0.9993
353	0.9950	0.9965	0.9951	0.9982	0.9999
363	0.9974	0.9973	0.9989	0.9998	0.9991

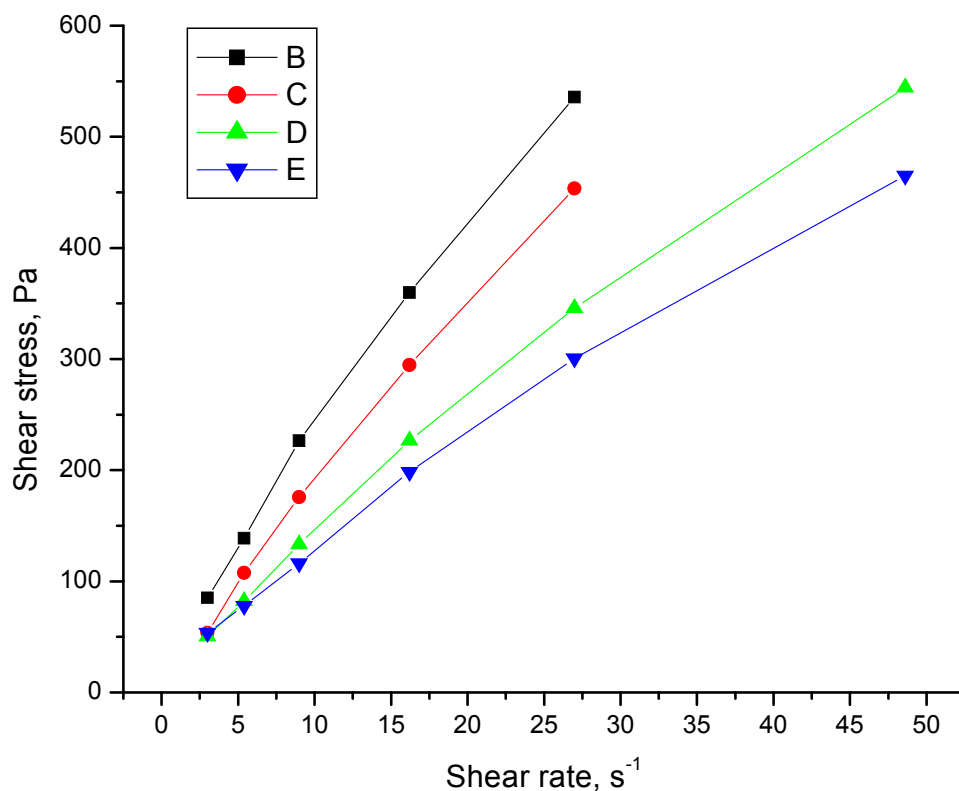


Fig. 5. Rheograms of 12% EPDM solution at: B – 333K; C– 343K; D – 353K and E– 363K

Table 6. Absolute temperature, yield stress, flow index, index of consistency and value coefficient correlation of the model described by equations (6) for the solution 10 g/dL

Temperature, K	Yield stress, τ_0	Flow index, n	Index of consistency, k	Correlation coefficient, R^2
323	4.5780	0.7598	0.7853	0.9992
333	3.8508	0.8477	0.8457	0.9963
343	3.6673	0.8039	0.9135	0.9986
353	3.0002	0.8694	0.8464	0.9982
363	2.7012	0.8700	0.8845	0.9998

Table 7. Absolute temperature, value coefficient correlation of the model described by equations (3-7) for the solution 12 g/dL

Value coefficient correlation, R^2					
Temperature, K	Model Bingham calculated with eq. (3)	Model Casson calculated with eq. (4)	Model Ostwald-de Waele calculated with eq. (5)	Model Herschel-Bulkley calculated with eq. (6)	Model proposed calculated with eq. (7)
333	0.9969	0.9982	0.9970	0.9993	0.9997
343	0.9967	0.9971	0.9973	0.9998	0.9999
353	0.9963	0.9980	0.9965	0.9994	0.9996
363	0.9963	0.9985	0.9963	0.9998	0.9999

Table 8 shows the yield stress, flow index, index of consistency and correlation coefficients for the solution of 12% in the temperature range 313-370 K determined by equation (6).

Table 8. Absolute temperature, yield stress, flow index, index of consistency and value coefficient correlation of the model described by equations (6) for the solution 12 g/dL

Temperature, K	Yield stress, τ_0	Flow index, n	Index of consistency, k	Correlation coefficient, R^2
333	3.5206	0.8449	0.4296	0.9993
343	3.1034	0.8877	0.5423	0.9998
353	2.9751	0.8565	0.4378	0.9994
363	2.8932	0.8564	0.4289	0.9998

The statistical correlation coefficients calculated at each temperature experimental data obtained for solutions of concentration 3%, 6%, 10% and 12% are given in Table 9.

The dynamic viscosity solutions EPDM copolymer similarly decreases with increasing temperature, and in Fig. 6 is the decrease of viscosity with temperature for a solution with a concentration of 3 g / dL.

From the figure it can be seen that the first order exponential decrease is observed for the solution on the entire temperature range.

Table 9. Absolute temperature, value statistical coefficient correlation for the solution 3 g/dL, 6g/dL, 10g/dL and 12g/dL

Value coefficient correlation, R ²				
Temperature, K	Solution concentration 3%	Solution Concentration 6%	Solution Concentration 10%	Solution concentration 12%
313	0.9744	0.9809	-	-
323	0.9859	0.9888	-	-
333	0.9950	0.9902	-	0.9938
343	0.9964	-	0.9890	0.9900
353	0.9524	0.9924	0.9902	0.9904
363	0.9956	0.9683	0.9968	0.9948
370	-	0.9575	-	-

The same can be seen in the correlation coefficient of 0.9872. For solutions with other concentrations are lower correlation coefficients, ranging between 0.9769 solution concentration of 10 g / dL and 0.9930 for the given concentration of 12 g / dL.

Log dependence on the value of the dynamic viscosities of the solutions of the inverse of the temperature of EPDM is shown in Fig. 7.

The figure shows that this copolymer are linear dependencies in the range of concentrations for all temperatures studied, but that the slopes are not similar, in particular the concentration of 12 g / dL, which value is very small compared to the other. At the same time, viscosity values are very close to concentrations to 10 and 12 g / dL, but the more concentrated solution viscosity less dependent on temperature. The dependence log dynamic viscosities on temperature for concentrated solutions were obtained the equation (8).

In order dependencies temperature viscosities of the four solutions were obtained the following equation (9-12):

$$\log \eta = - 3.4660 + 1946.97/T \quad (9)$$

$$\log \eta = - 3.0122 + 2171.59/T \quad (10)$$

$$\log \eta = - 1.8091 + 2099.44/T \quad (11)$$

$$\log \eta = - 0.2991 + 1560.91/T \quad (12)$$

The values of the correlation coefficients of between 0.9996 and 0.9998 .

The values of the constants A and B are given in table 10 to facilitate discussion of their variation .

Comparing the decrease of viscosity of solutions of the copolymer with increasing temperature it can be seen that EPDM is able to generate multi-grade oils with wider temperature ranges, that is it is a better viscosity improver at all the studied concentrations.

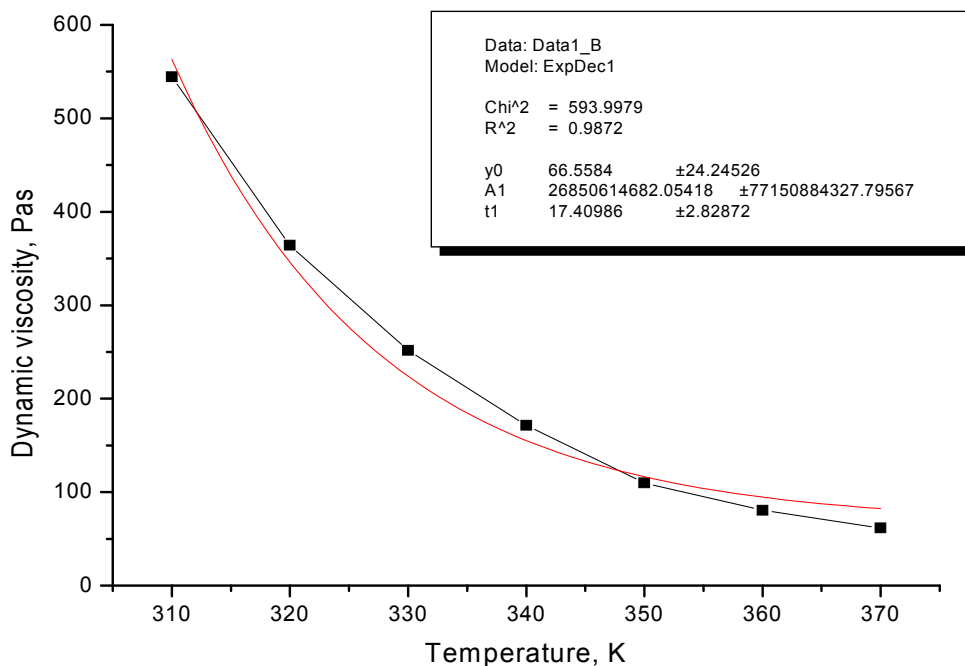


Fig. 6. Dependence of dynamic viscosity – absolute temperature for solution of concentration 3 g/dL

Table 10. Constans A and B in the Andrade equation for EPDM solutions

Concentration, %	EPDM	
	log A	B
3	-3.4660	1946.97
6	-3.0122	2171.59
10	-1.8091	2099.94
12	-0.2991	1560.91

As can be seen from the table, the values for EPDM solutions vary with the concentration of high values.

In respect of the slope, the smaller value is obtained for a concentration of 12 g / dL, followed by the concentration of 3 g / dL, and the higher the concentration of 6 g / dL.

Lower values obtained from the change in the slopes of the lines representing the viscosity of the solution of the copolymer with EPDM at all concentrations temperatures means that the viscosity of solutions of the copolymer is less dependent on temperature.

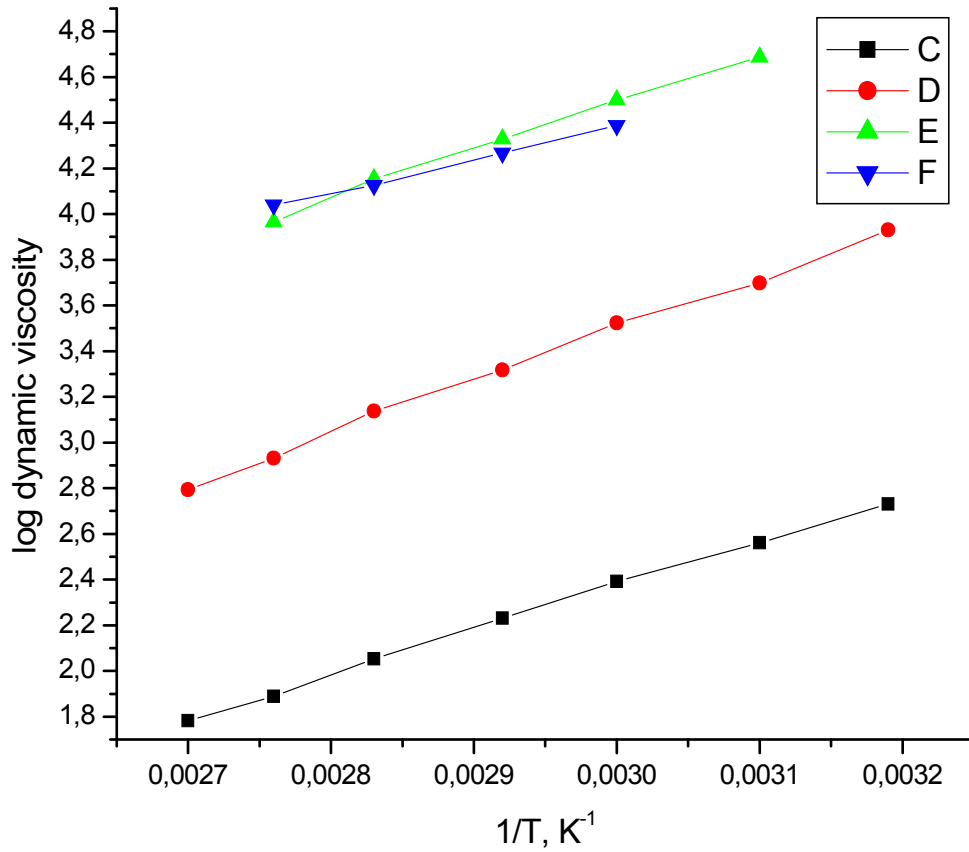


Fig. 7. Dependence $\log \eta = f(1/T)$ for solutions of EPDM: C – 3%; D – 6%; E – 10%; F – 12%

4. CONCLUSION

EPDM solutions in SAE 10W-40 mineral oil are more viscous. In the case of EPDM solutions the slope increases when passing from the copolymer concentration 3 to 6%, but decreases when concentration exceeds this value. The lowest slope was obtained for solution having the concentration 12%, followed by that of 3% solution, while 6% solution has the highest value. This suggests that EPDM is a better viscosity improver at all the concentrations for the mineral oil SAE 10W-40.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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