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FEATURES OF THE SWELLING KINETICS OF POLYMER MATERIALS IN BLENDED DIESEL FUEL

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Abstract. The work is devoted to determining the features of the swelling kinetic patterns of polymer materials in contact with blended fuel containing fatty acid esters. Kinetic equations for predicting the behavior of polymer materials in contact with blended fuel at different temperatures were obtained. Peculiarities of the swelling kinetics of polymer materials with different chemical structures in blended diesel fuel were studied. It was found that the process proceeds in two stages for any temperature conditions. The effect of fatty acid esters in the composition of blended diesel fuel on the stability of polymer materials depending on their structure and temperature factor was determined. An approach to the selection of polymer materials resistant to the effects of fatty acid esters is proposed. The patterns of changes in the swelling resistance of polymer materials upon contact with blended diesel fuel, depending on their structure, were determined.

Keywords: blended diesel fuel, fatty acid esters, polymer materials, swelling degree, kinetic equations, activation energy.

1. Introduction

The problem of expanding raw materials with the involvement of industrial and agricultural fat-containing waste is urgent in the context of a critical shortage of traditional mineral fuels.[1]

An alternative to diesel fuel is fatty acid esters (FAE), which are produced by transesterification of fat-containing raw materials.[2] According to the physicochemical and operational characteristics, FAE is close to diesel fuel and can be used as an additive to blended fuel or in its pure form.[3,4] In addition, this fuel is more ecological, since it does not contain sulfur and aromatic substances.[5,6]

There are many sources for FAE production, such as plant raw materials (soybean, palm, rapeseed oil), animal fats (beef, chicken, pork fat), and various fat-containing wastes, in particular frying oil and fuzz, the physicochemical properties of which depend on the raw materials.[7]

Fuel interacts with various materials in a diesel engine. Several studies have shown the stability of seals, hoses, and gaskets in diesel fuel.[8,9] However, there is much less available information on the stability of fuel system polymer materials in contact with FAE and blended fuels. Diesel fuel and FAE interact differently with structural materials due to differences in chemical composition.[10] Numerous studies have shown that structural materials destruction is a significant problem due to the instability of structural materials, particularly elastomeric, during FAE use.[11,12]

An elastomeric material based on nitrile butadiene rubber is highly resistant to mineral oils and non-polar solvents due to the presence of nitrile groups in its structure. It is used to manufacture parts constantly in contact with fuel in the engine.[13] The destruction of nitrile rubber occurs in various ways, including changes in the network of crosslinks, reactions with double bonds of the elastomer, and also acceleration of the thermal oxidation process of rubber in the presence of carbon black.[14-16]

Stability studies of any polymer materials with FAE and blended fuels are carried out by observing changes in swelling and mechanical properties after static and/or dynamic immersion in various types of the medium at different temperatures.[17]

Bessee and Fay[18] presented a study on the influence of blended diesel fuel with the addition of fatty acid methyl esters (FAME) on such characteristics of common polymer materials as tensile strength, elongation at break, hardness, and swelling. The elastomeric material based on nitrile butadiene rubber, nylon, and high-density polypropylene showed changes in physical properties, while Teflon, Viton

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401-C (a copolymer of hexafluoropropylene and vinylidene fluoride and curing chemicals), and Viton GFLT (polymerization product of fluorinated vinyl ether monomer in the presence of peroxide at low temperatures) remained almost unchanged. Using blended fuel with 10-20% FAE additive does not reduce the stability of rubber based on nitrile butadiene rubber.[19] Linhares *et al.*[20] showed that increasing acrylonitrile content increases the resistance of elastomers based on nitrile butadiene rubber to FAE.

The situation is complicated by the fact that during the study of the FAE effect on polymer materials, it is necessary to take into account that different processes can occur in parallel: swelling, dissolution, extraction of soluble components from the elastomer structure, such as plasticizers, simultaneous structural changes, volume change with mass change, change tensile strength, elongation at break, hardness. Regardless of such factors, the stability of various polymer materials in FAE has a certain role.

Veza *et al.*[21] presented the study results of the blended fuel effect on the characteristics of an elastomeric material based on nitrile butadiene rubber. It was concluded that the diffusion of FAE with an increase in its concentration leads to a decrease in the mechanical properties of polymer materials and, as a result, to the hardening of the fuel hose, its sudden cracking, and spillage of the petroleum product.

At the moment, predicting the suitability of various types of polymer materials for use in engines running on blended fuel is complicated by the insufficient study of the main processes that occur during the interaction of FAE with structural materials, particularly the swelling kinetics of the latter.

The work aims to determine the features of the swelling kinetic patterns of polymer materials in contact with blended fuel for the effective selection of FAE-resistant matrices.

The study object is the behavior of polymer materials under conditions of long-term contact with blended fuel at different temperatures.

2. Experimental

For diesel engines, hoses are usually made from rubber-modified polyolefins, while gaskets are made from an asbestos-free elastomeric material, ethylene-propylene-diene rubber, nitrile butadiene rubber, fluoro rubber, polychloroprene, and silicone. The usual materials for sealants are polyurethane, materials based on nitrile butadiene and silicone rubbers, primary polytetrafluoroethylene, *etc.*[19] Most of these materials used in a diesel engine are not resistant to FAME.

Considering this, polymer materials based on various matrices were used for the research: filled oleo-benzene-resistant rubber based on nitrile butadiene rubber with an average content of acrylonitrile (BNK-26) and polychloroprene from New Technologies LLC, Dnipro, cast hard polyurethane of the Neuthane 128S brand from Notedome LTD (Great Britain), obtained by the interaction of PTMEG prepolymer with a content of 2.8 wt. % of isocyanate groups with CA6 hardener at a mass ratio of components of 100:6.8 and curing at 100 °C×16 h. and hard polyurea of the EPAPROOF FPCS07 brand from EPAFLEX POLYURETNANES (Italy) (two-component system: component A – aromatic diisocyanates + component B – di- and polyamines based on aliphatic oligomeric polyesters with a viscosity at 25 °C of 680 mPa·s), obtained mixing the components by volume 1:1 in the Reactor A-XP1 installation from GRACO company (USA) at 70 °C with curing at 20 °C x 24 h.

The characteristics of the studied polymer material samples are given in Table 1, their chemical formulas are shown in Fig. 1.

Table 1. Characteristics of polymer materials

Sample	Polyurethane	Polyurea	BNK-26	Polychloroprene
Density, kg/m^3	1120	1070	1400	1400
Tensile strength, MPa	35.6	26.3	12.9	13.8
Elongation at break, %	520	310	445	290
Shore hardness A, conditional units	80	75	70	67

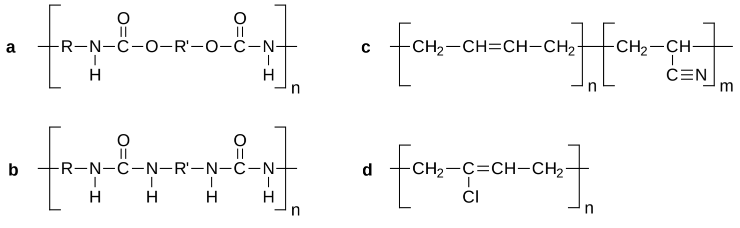
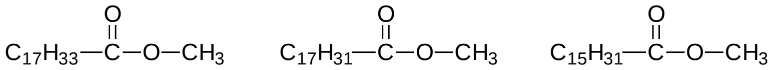


Fig. 1. Chemical formulas of polymer materials: a – polyurethane; b – polyurea; c – BNK-26; d – polychloroprene



$C_{17}H_{33}-\overset{\overset{O}{\|}}{C}-O-CH_3 \quad C_{17}H_{31}-\overset{\overset{O}{\|}}{C}-O-CH_3 \quad C_{15}H_{31}-\overset{\overset{O}{\|}}{C}-O-CH_3$

Fig. 2. Chemical formula of fatty acid methyl esters from sunflower oil

Table 2. Physicochemical characteristics of diesel fuel, FAME, and the studied blended diesel fuel

Properties	Value		
	DF	FAME	Mixed diesel fuel 70%DF+30% FAME
Density, at 20°C, kg/m^3	827	899	849
Kinematic viscosity at 20°C, mm^2/s	2.9	5.4	3.3
Cetane number	51	53	52
Flash point in a closed cup, °C	62	154	121
Filtration temperature limit, °C	-8	-1	-5
Sulfur content, wt. %	0.20	0	0.14

Blended diesel fuel consisting of 70% mineral diesel fuel (DF) and 30% FAME from sunflower oil (the chemical formula of FAME is shown in Fig. 2) was used. This blended diesel fuel was chosen due to the results of previous studies,[22,23] which showed that using this composition leads to the smallest loss of strength and the smallest increase in mass swelling of polymer materials. The characteristics of the proposed blended diesel fuel are given in Table 2.

Samples of polymer materials as squares with a side length of 20 mm and a thickness of 2±0.2 mm were prepared for testing, and an identification label was placed on each sample. The prepared samples were placed in hermetic glasses (to prevent air ingress), which were filled with blended fuel and kept for 30 days at temperatures of 25 and 120±2°C.

After every 12-24 hours, samples of polymer materials were removed from the fuel, excess fuel was quickly removed from the surface using an ashless paper filter, immediately weighed on an ANG-200C analytical balance with an accuracy of 0.0002 g, and returned to sealed beakers for further study.

The degree of swelling was calculated using Eq. (1):[24]

$$\alpha = \frac{m_2 - m_1}{m_1} \cdot 100\% \tag{1}$$

where α is a swelling degree, wt. %; m_1 and m_2 are the sample initial mass and the sample mass after swelling, respectively, g.

The research was carried out at temperatures of 25 ℃ and 120 ℃, which were stabilized by a thermostat. The study's high temperature and duration are due to the need to determine the behavior of polymer materials in contact with blended fuel under conditions close to working conditions.

The swelling process[25] of polymer materials corresponding to the first-order reaction from the standpoint of formal kinetics is well described by the following equation:

$$\frac{dx}{d\tau} = k \cdot (\alpha_{max} - \alpha_\tau) \tag{2}$$

where $\frac{dx}{d\tau}$ is a swelling rate, h^{-1}; k is the process rate constant, h^{-1}; α_{max} and α_τ are swelling degree of the polymer material at the end of the experiment and in the given time, respectively.

Based on Eq. (2), the rate constant was calculated according to the following equation:

$$k = \frac{1}{\tau} \ln\left(\frac{\alpha_{max}}{\alpha_{max} - \alpha_\tau}\right) \tag{3}$$

where τ is a process time, hours.

To characterize the first-order reaction rate, in addition to the rate constant, the value of the half-life was also used. It corresponds to the ratio $\frac{\alpha_{max}}{\alpha_{max} - \alpha_\tau} = 2$, therefore, according to Eq. (3) there is:

$$\tau_{1/2} = \frac{ln\,2}{k} \tag{4}$$

Eq. (4) is allowed to determine the time during which the polymer material swells by 50% of the maximum possible degree.

A kinetic model based on the Arrhenius equation[26] was used to predict the swelling rate of polymer materials at any temperature:

$$k = A \cdot e^{\frac{-E}{R \cdot T}} \tag{5}$$

where *A* is the pre-exponential factor; *E* is an activation energy of the process, J/mol; *R* is the universal gas constant, J/(mol·K); *T* is the temperature, K.

The activation energy *E* was determined by the results of two series of experiments at different temperatures using Eq. (6):[27]

$$E = \frac{-R \cdot (\ln k_2 - \ln k_1)}{T_1^{-1} - T_2^{-1}}, \tag{6}$$

where k_2 and k_1 are swelling process rate constants, h^{-1}, at temperatures T_2=120 ℃ and T_1=25 ℃, respectively.

3. Results and Discussion

The first stage of the study was to determine the swelling kinetic parameters of polymer materials at a temperature of 25℃, which corresponds to the mode of the non-running engine. The results of determining the swelling degree are shown in Fig. 3.

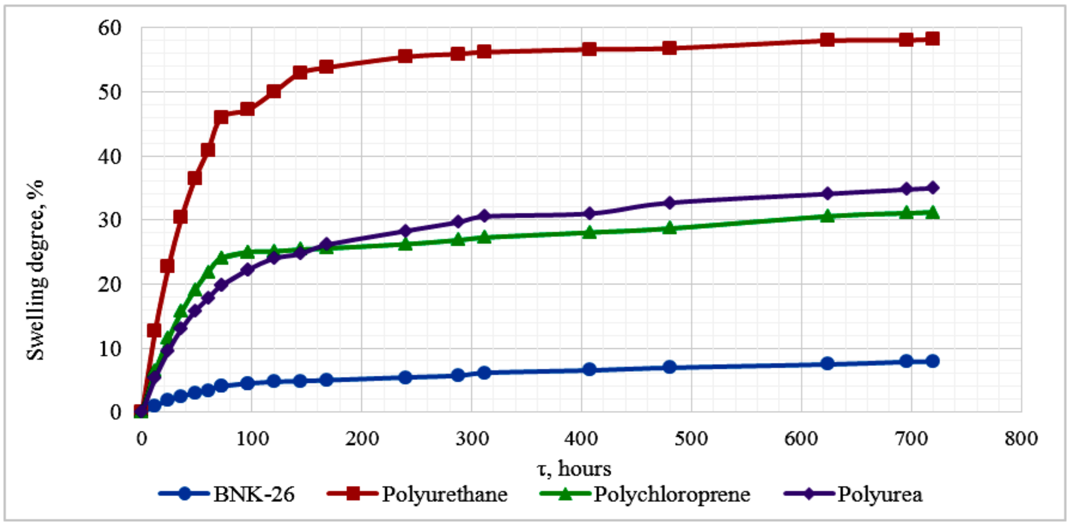


Fig. 3. Swelling kinetic curves of polymer materials in blended fuel at a temperature of 25°C

According to Fig. 3, the swelling degree of the samples increases in the BNK-26-polychloroprene-polyurea-polyurethane series, which completely coincides with the decrease in the tensile strength of the samples.[23] It should also be noted that the greatest change in the swelling degree is observed during the first 72 hours, after which the process slows down significantly. Based on the results obtained, it can be concluded that most of the intermolecular crosslinks present in polymer materials are destroyed during the first 72 hours of contact with blended fuel, which also correlates with a more significant decrease in tensile strength in the initial period of the experiment. The obtained results made it possible to consider the swelling process as two stages consisting of an active (first 72 hours) and a slow stage (72-720 hours).

Eq. (2) was used to determine the rate constants of both swelling stages of polymer materials, after rewriting in logarithmic form:

$$\ln\left(\frac{\alpha_{max}}{\alpha_{max}-\alpha_{\tau}}\right) = k\tau \tag{7}$$

The results of experimental swelling kinetic studies of the polymer materials samples depending on the time contact with the blended fuel were processed according to Eq. (7) and presented graphically (Figs. 4 and 5) to calculate the swelling rate constant.

The graphical dependence of $\ln\left(\frac{\alpha_{max}}{\alpha_{max}-\alpha_{\tau}}\right)$ on τ for Eq. (7) is close to linear, which is the main criterion confirming the first order of swelling kinetics. The calculated values of the swelling rate constant of polymer materials are presented in Table 3. The error in the rate constants are calculated according to the formula:

$$k_{\sigma} = k\sqrt{\frac{R^2-1}{n-2}} \tag{8}$$

where k is the swelling rate constant, h^{-1}; R^2 is the coefficient of pair correlation; n is a number of measurements.

The next step was to determine the swelling kinetic parameters of polymer materials at a temperature of 120ºC, which is close to the working conditions of the internal combustion engine fuel system. The results of determining the swelling degree are shown in Fig. 6.

As in the previous series of experiments, the swelling rate of all samples was the highest in the first hours of the experiment (72 h). It should be noted that later, at a temperature of 25°C, a slow increase in the swelling degree was observed. At a temperature of 120°C, this indicator almost did not change for samples of polychloroprene and BNK-26. At the same time, the rest of the samples were completely destroyed at high temperature for 72 h (polyurethane) or 96 h (polyurea). Such results confirm the hypothesis that during the first 72 h, fuel effectively penetrates the polymer material and the increase in the swelling degree with increasing temperature indicates the endothermic nature of this process.

The swelling rate constants at a temperature of 120°C were calculated according to the data in Figs. 7 and 8 and are presented in Table 4. For polyurethane and polyurea samples, swelling rate constants were calculated only for the first stage.

Based on the results obtained in both series of experiments, the swelling kinetic parameters of polymer materials, namely activation energy and pre-exponential factor, were calculated according to Eqs. (5) and (6). The calculation results for the BNK-26 and polychloroprene samples are shown in Table 5. It is impossible to establish the swelling kinetic parameters for polyurethane and polyurea for the given experimental conditions due to the samples destruction.

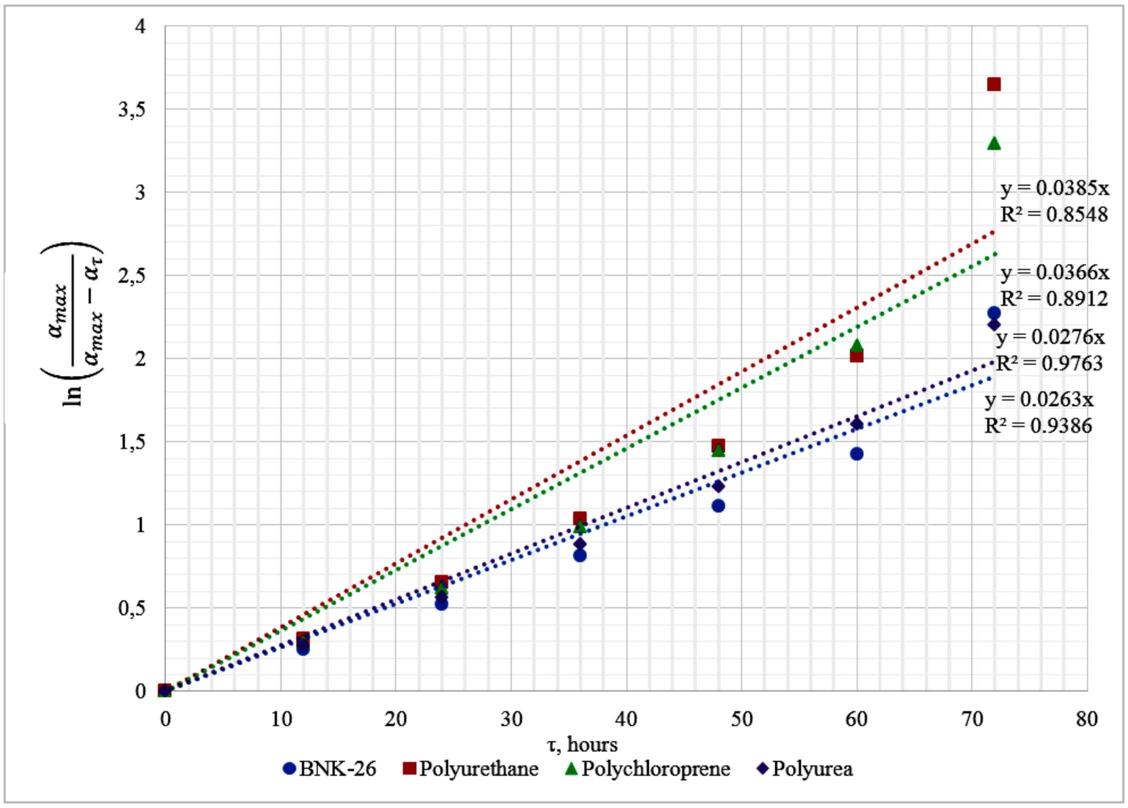


Fig. 4. Determination of the first stage swelling rate constant of polymer materials at a temperature of 25°C

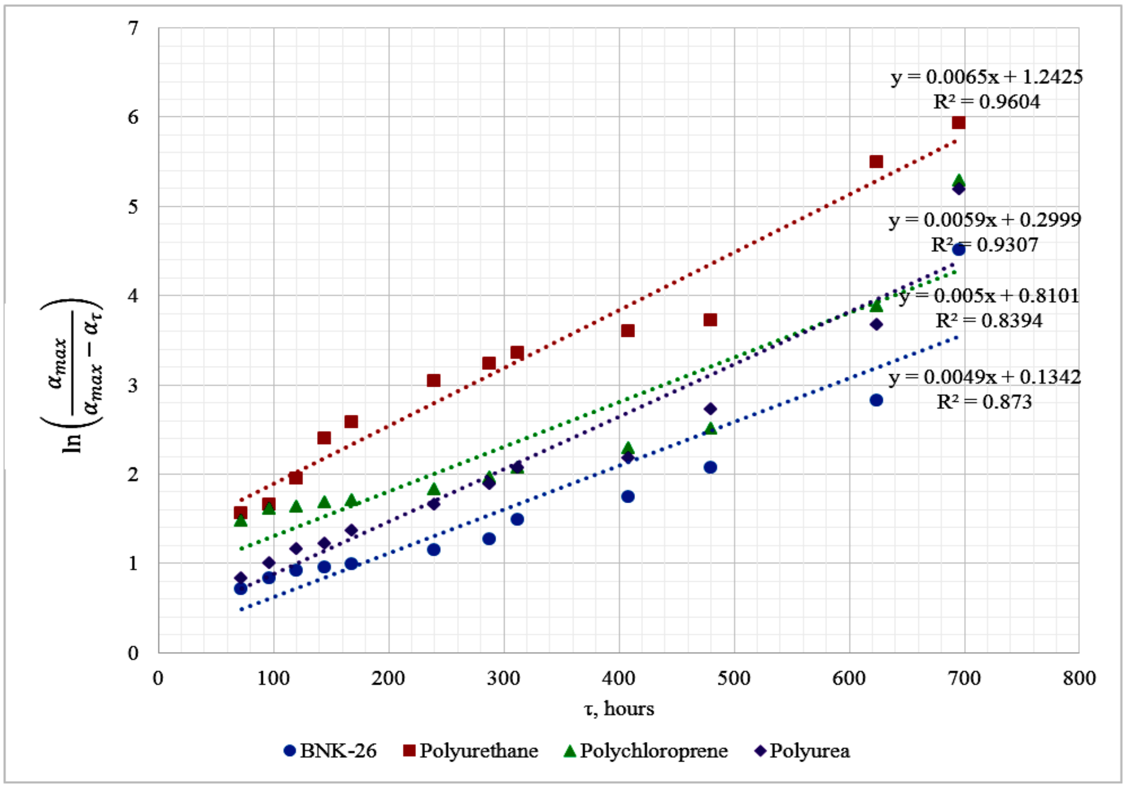


Fig. 5. Determination of the second stage swelling rate constant of polymer materials at a temperature of 25°C

Table 3. Swelling rate constants of polymer materials in blended fuel at a temperature of 25°C

Sample of polymer materials	First stage swelling rate constant k_1, h^{-1}	$\tau_{1/2}$, h	Second stage swelling rate constant k_2, h^{-1}	$\tau_{1/2}$, h
Polychloroprene	0.0366±0.0054	18.9	0.0050±0.0006	138.6
Polyurethane	0.0385±0.0066	18.0	0.0065±0.0004	106.6
Polyurea	0.0276±0.0019	25.1	0.0059±0.0005	117.5
BNK-26	0.0263±0.0029	26.3	0.0049±0.0006	141.5

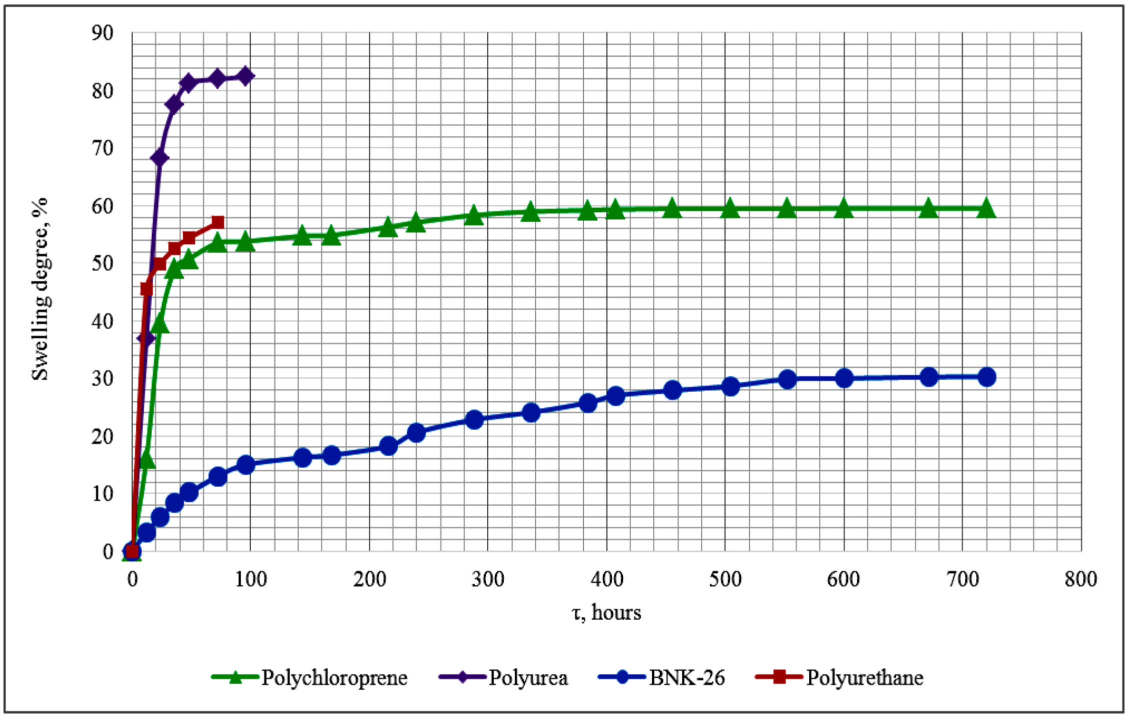


Fig. 6. Swelling kinetic curves of polymer materials in blended fuel at a temperature of 120°C

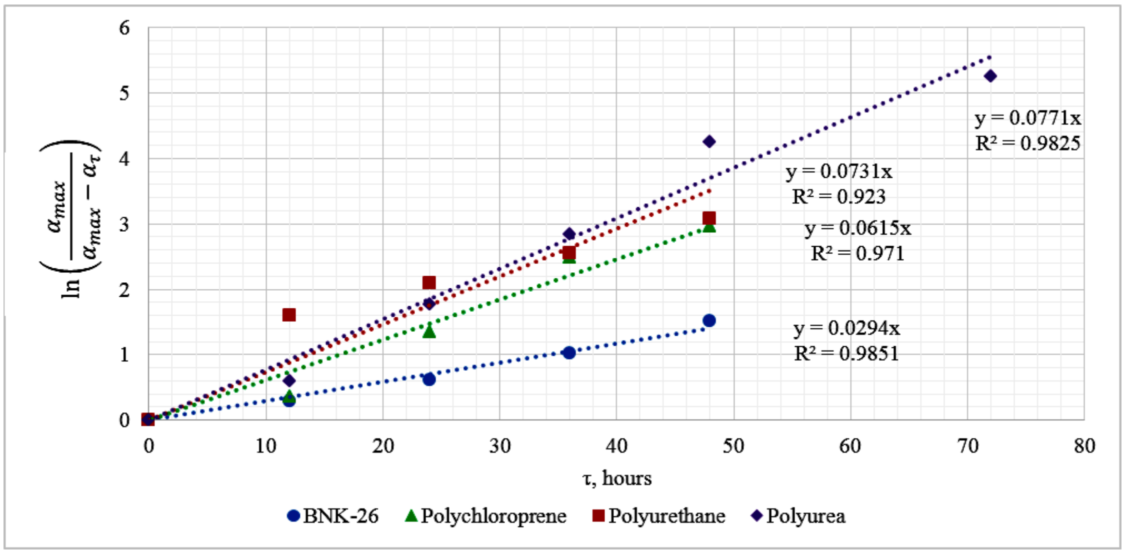


Fig. 7. Determination of the first stage swelling rate constant of polymer materials at a temperature of 120°C

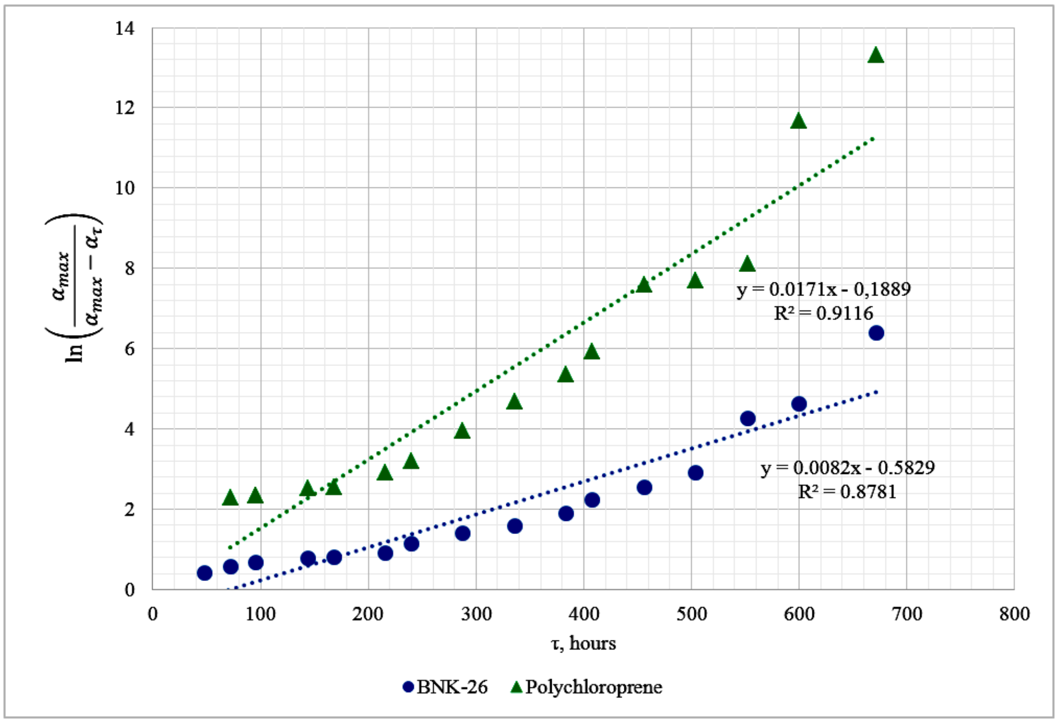


Fig. 8. Determination of the second stage swelling rate constant of polymer materials at a temperature of 120°C

Table 4. Swelling rate constants of polymer materials in blended fuel at a temperature of 120 °C

Sample of polymer materials	First stage swelling rate constant k_1, h^{-1}	$\tau_{1/2}$, h	Second stage swelling rate constant k_2, h^{-1}	$\tau_{1/2}$, h
Polychloroprene	0.0615±0.0060	11.3	0.0171±0.0014	40.5
Polyurethane	0.0731±0.0117	9.5	-	-
Polyurea	0.0771±0.0051	9.0	-	-
BNK-26	0.0294±0.0021	23.6	0.0082±0.0008	841.5

Table 5. Swelling kinetic parameters of polymeric materials

<table>
<tr><td colspan="7">The first (active) stage of the swelling process</td></tr>
<tr><td>Sample</td><td>T_1, K</td><td>T_2, K</td><td>k_1, h^{-1}</td><td>k_2, h^{-1}</td><td>Ea, J/mol</td><td>A, h^{-1}</td></tr>
<tr><td>BNK-26</td><td>298</td><td>393</td><td>0.0263</td><td>0.0294</td><td>1142.04</td><td>0.0417</td></tr>
<tr><td>Polychloroprene</td><td>298</td><td>393</td><td>0.0366</td><td>0.0615</td><td>5319.29</td><td>0.3133</td></tr>
<tr><td>Polyurethane</td><td>298</td><td>393</td><td>0.0385</td><td>0.0731</td><td>6571.56</td><td>0.5463</td></tr>
<tr><td>Polyurea</td><td>298</td><td>393</td><td>0.0276</td><td>0.0276</td><td>10529.00</td><td>1.9314</td></tr>
<tr><td colspan="7">The second (slow) stage of the swelling process</td></tr>
<tr><td>BNK-26</td><td>298</td><td>393</td><td>0.0049</td><td>0.0082</td><td>5277.37</td><td>0.0412</td></tr>
<tr><td>Polychloroprene</td><td>298</td><td>393</td><td>0.0050</td><td>0.0171</td><td>7532.68</td><td>0.1715</td></tr>
</table>

Based on the results presented in Table 5, the kinetic equations of the swelling process for BNK-26 of the first (active) stage (Eq. 8) and the second (slow) stage (Eq. 9), polychloroprene of the first stage (Eq. 10) and the second stage (Eq. 11), polyurea and polyurethane of the first stage (Eq. 12, Eq. 13) are compiled:

$$k = 0.0417e^{\frac{-1142.,04}{8.314T}} \tag{8}$$

$$k = 0.0412e^{\frac{-5277.37}{8.314T}} \tag{9}$$

$$k = 0,3133e^{\frac{-5319.29}{8.314T}} \tag{10}$$

$$k = 0.1715e^{\frac{-7532.68}{8.314T}} \tag{11}$$

$$k = 1.9314e^{\frac{-10529.00}{8.314T}} \tag{12}$$

$$k = 0.5463e^{\frac{-6571.56}{8.314T}} \tag{13}$$

The graphical interpretation of the obtained kinetic equations is shown in Fig. 9.

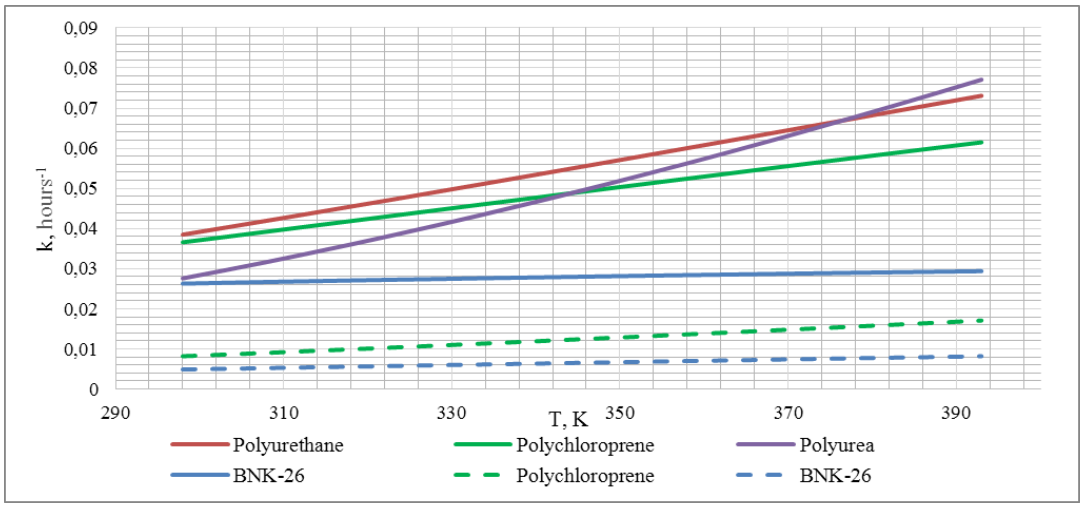


Fig. 9. Dependencies of the swelling rate constants of the first (—) and second (- – -) stages for polymer materials on temperature.

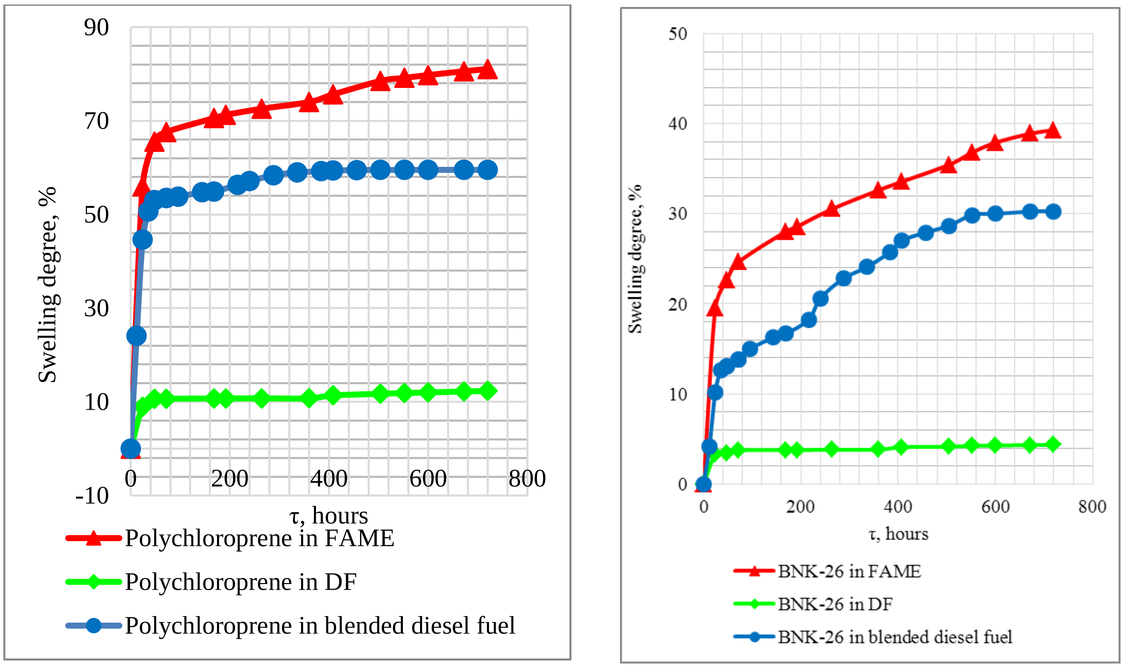


Fig. 10. Swelling curves of polymer materials in diesel fuel (DF), methyl esters of fatty acids from sunflower oil (FAME), and blended fuel at a temperature of 120°C

As can be seen from Fig. 9, according to the change in the swelling degree, the polyurea sample is the most sensitive to temperature increase, and the BNK-26 sample is the least sensitive. At the same time, it should be noted that the second stage of swelling of BNK-26 and polychloroprene samples is characterized by a decrease in the effect of temperature changes on the swelling degree compared to the first.

Comparison results of determining the swelling degree of polymer materials in blended fuel, diesel fuel, and methyl esters of fatty acids from sunflower oil in their pure form at a temperature of 120°C are shown in Fig. 10. Even at a temperature close to the real operating conditions of a diesel engine, the swelling degree of the samples in contact with diesel fuel was insignificant (4.4% for BNK-26 and 12.3% for polychloroprene) and almost did not change after the first 48 h. At the same time, the swelling degree of samples in contact with FAME constantly increased throughout the study and reached 39.3% for BNK-26 and 81.0% for polychloroprene. It can be concluded that polymer material based on polychloroprene is not recommended for use in pure FAME medium due to its low swelling resistance.

Polymer samples that were in contact with blended diesel fuel during the study reached 30.3% for BNK-26 and 59.5% for polychloroprene, respectively, significantly higher than the swelling degree in diesel fuel. However, in contrast to the results obtained using pure FAME, this swelling degree can be considered acceptable since it almost did not change during the last 168 h (BNK-26) and 384 h (polychloroprene).

4. Conclusions

1. The swelling kinetic patterns of different chemical composition polymer materials in blended diesel fuel were established, and, it was determined that this process proceeds in two stages both at a temperature of 25 °C (close to the not-running engine mode) and at 120 °C (close to the running diesel engine mode): the first (active) stage, and the second (slow) stage of the process.
2. The dependence of the change in the activation energy of the swelling process of polymer materials on the chemical structure of the polymer matrix was clarified.
3. The dependence of the change in the activation energy of the swelling process of polymer materials on the chemical structure of the polymer matrix was clarified. It was shown that the value of the activation energy for all polymer materials samples is leveled by the pre-exponential factor A values, which characterize the total number of collisions between the molecules of polymer materials and the molecules of the solvent depending on the density and structure of the three-dimensional grid. Thus, under the conditions when the engine is running, the samples of polyurea and polyurethane were the least resistant to swelling, and the samples of BNK-26 and polychloroprene were the most resistant.
4. An approach to the selection of elastomeric materials resistant to the influence of fatty acid esters is proposed. It is shown that the swelling resistance of polymer materials in contact with blended diesel fuel decreases in the following series of studied polymer materials: rubber based on nitrile butadiene rubber with an average content of acrylonitrile (26 wt. %), rubber based on polychloroprene, polyurea, and polyurethane.
5. It is recommended to evaluate the stability of polymer materials in contact with blended diesel fuel according to the first active stage of the swelling process, both at 25 °C and at 120 °C.

Abbreviations

FAE – fatty acid esters;
FAME – fatty acid methyl esters;
BNK-26 – filled oleo-benzene-resistant rubber based on nitrile butadiene rubber with an average content of acrylonitrile;
DF – diesel fuel.

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ОСОБЛИВОСТІ КІНЕТИКИ ПРОЦЕСУ НАБРЯКАННЯ ПОЛІМЕРНИХ МАТЕРІАЛІВ У СУМІШЕВОМУ ДИЗЕЛЬНОМУ ПАЛИВІ

Анотація. *Робота присвячена визначенню особливостей кінетичних закономірностей набрякання полімерних матеріалів під час контакту з сумішевим паливом, що містить естери жирних кислот. Отримано кінетичні рівняння для прогнозування поведінки полімерних матеріалів під час контакту з сумішевим паливом за різних температур. Вивчені особливості кінетичних закономірностей набрякання полімерних матеріалів різної хімічної будови в сумішевому дизельному паливі. Встановлено, що для будь-яких температурних умов процес перебігає у дві стадії. Визначено вплив естерів жирних кислот у складі сумішевого дизельного палива на стійкість полімерних матеріалів з урахуванням їхньої будови та температурного фактору. Запропоновано підхід до вибору полімерних матеріалів, стійких до впливу естерів жирних кислот. Визначені закономірності зміни стійкості полімерних матеріалів до набрякання залежно від будови під час контакту з сумішевим дизельним паливом*

Ключові слова: *сумішеве дизельне паливо, естери жирних кислот, полімерні матеріали, ступінь набрякання, кінетичні рівняння, енергія активації.*