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Study of Reological and Tribological Properties of Terpolymers of Allyl Esters of C6, C8, and C10 Carbonic Acids in Polysintetic Oils

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Abstract

The main objective of this work is to evaluate the rheological and tribological properties of ternary copolymers based on the allyl esters of caproic (C_6), caprylic (C_8), and capric (C_{10}) acids, synthesized via radical polymerization with styrene and butyl methacrylate (BMA). Depending on the synthesis conditions, these terpolymers exhibit excellent solubility in both non-polar mineral oils and polar synthetic ester oils. To ensure complete chemical compatibility, a semi-synthetic reference base oil consisting of SN-600 petroleum oil and a synthetic pentaerythritol ester (PEE, esterified with monobasic carboxylic acids) at a 1:1 weight ratio was investigated. The comprehensive study characterizes the viscosity-temperature (rheological), pour point depressant, chemical degradation, mechanical shear destruction, and boundary tribological properties of the synthesized terpolymers within this semi-synthetic matrix. The results demonstrate that these macromolecules exhibit a multi-functional mechanism, significantly improving the anti-wear and friction-reducing tribological performance of semi-synthetic lubricants while maintaining excellent rheological stability.

1. İntodaction

It is known that one of the main performance properties characterizing lubricating oils is their lubricity; at the same time, oils must have high surface activity to form a strong film on the surfaces of rubbing parts that can prevent or reduce wear under operating loads and speeds (Kuliyev, 1985).

Improved friction performance is an important objective for equipment manufacturers to meet the demands of improved energy efficiency. The addition of friction-reducing additives, or friction modifiers, to lubricants is a key part of the strategy. The performance of these additives is related to their surface activity and their ability to form adsorbed layers on the metal surface (Jaishankar et al., 2019).

Although viscosity modifiers are used in lubricant formulations primarily to improve the solution's high-temperature viscosity, they have also been shown to provide additional benefits as friction modifiers, pour point depressants, and dispersants. Viscosity modifier polymers that perform one or more of these additional functions are known as multifunctional viscosity modifiers (Martini *et al.*,2018; Frank et al., 2021). One commonly studied secondary role of viscosity modifiers is their ability to reduce friction and/or wear, i.e., to act as friction modifiers (Spikes, 2015). The beneficial effect of viscosity modifiers in boundary lubrication was observed in early studies, with larger-than-expected decreases in friction and wear in viscosity-modifier solutions not associated with increased viscosity (Okrent, 1961). Since then, this behavior has been attributed to the formation of boundary films via the physical adsorption of polymers on polar surfaces (Müller *et al.*, 2006). The films consist of dense, viscous layers of polymers whose thickness can be correlated to the polymer's characteristic coil size (Cann and Spikes, 1994). The viscous polymer layer at the contact inlet leads to a thicker film in boundary lubrication and, in turn, reduced friction (Smeeth et al., 1996).

Improved tribological behavior in boundary lubrication has been observed for multiple viscosity modifier chemistries, including polyisobutylenes (PİB) and polyalkylmethacrylates (PAMA). Still, many studies

have focused on PAMA because its versatile architecture enables the investigation of the effects of specific features. Such studies have shown that structure, functionalization, and molecular weight are key factors in determining the effectiveness of a viscosity modifier's boundary layer (Fan et al., 2007). The researchers (Müller *et al.*, 2006) show that medium- to high-molecular-weight polymers will form the thickest boundary layers. However, low-molecular-weight polymers have also been shown to be beneficial for boundary lubrication (Cosimbescu *et al.*, 2017; Robinson et al., 2016). It has also been shown that viscosity modifiers can influence the thickness of a boundary.

The research of authors (Cosimbescu *et al.*, 2017; Cosimbescu et al., 2016) focuses on the design of novel viscosity modifiers that can either complement or replace traditional friction modifiers in lubricant formulations.

Analysis of recent studies shows that acrylate- or methacrylate-based polymers have great potential to adjust the viscosity-temperature and depressant properties of lubricants (Yukic *et al.*, 2007; Grosh and Kumar, 2015; Grosh *et al.*, 2017; Cosimbescu *et al.*, 2017; Ahmed et al., 2022). However, to impart tribological properties to these polymers, it is necessary to incorporate monomers with specific functional groups into their composition. In this regard, allyl esters of medium-chain carboxylic acids (C_6, C_8, and C_{10}) are of particular interest due to their lipophilic nature and adhesion to metal surfaces [Hamidova *et al.*, 2020; Hamidova *et al.*, 2020; Hamidova *et al.*, 2020; Hasanova et al., 2020; Hamidova *et al.*, 2021; Hamidova *et al.*, 2023; Hamidova et al., 2024; Farzaliyev et al., 2024; Kazimzade et al. 2022; Kazimzade et al. 2024; Kazimzade et al. 2026; Hamidova *et. al.*, 2026]. The allylic degradative chain-transfer property of allyl monomers during radical copolymerization makes it difficult for them to form high-molecular-mass polymers. In allyl monomers, the C-H bonds at the carbon atom adjacent to the double bond (α-carbon) are very weak. Instead of combining with the monomer's double bond, the growing active radical readily abstracts a hydrogen atom from this weak bond. Thus, in engine oils, high-molecular-mass polymers (MM > 50,000) are easily destroyed under mechanical shear stress, thereby reducing viscosity. Therefore, the synthesis of low-molecular-mass (MM ~ 10,000) terpolymers with high mechanical stability by controlling the chain-transfer process using the benzoyl peroxide (BP) initiator, and the study of their structure-activity relationships, are of great scientific and practical importance.

The main objective of the presented work is to study the rheological and tribological properties of ternary copolymers of allyl esters of caproic (C_6), caprylic (C_8) and capric (C_{10}) acids synthesized by radical polymerization with styrene and butyl methacrylate (BMA) [Hamidova *et al.,2020;*Hamidova et al., 2024; Hamidova et. al., 2024] in semi-synthetic oils.

2. Materials and Methods

2.1. Determination of rheological properties

Rheological properties of the ternary copolymers were determined in accordance with the ASTM D 445 standard – kinematic viscosity of the oil compositions in a capillary viscometer (VPJ-2) at temperatures

of 40°C and 100°C. The viscosity index (VI) was calculated in accordance with ASTM D 22 – 0.

2.2. Determination of depressant properties

The evaluation of oil compositions as pour point depressants was performed in accordance with ASTM D 9793 to determine the pour point (PP).

2.3. Determination of chemical degradation

The degradation of synthesized ternary copolymers in the base oil was investigated using a standard methodology (Papok and Semenido, 1965). This method involves heating the specified solutions to 200°C for 12 hours. At set time intervals, samples are collected to measure the decrease in viscosity. Based on this measurement, conclusions are drawn regarding the stability of the viscosity additive.

2.4. Determination of mechanical destruction

To test the resistance of polymer-added motor oils to mechanical (ultrasonic) destruction, the UZDN-2T apparatus (GOST 6794-75 is equivalent to the Russian standard ASTM D2603 (sonic method)) was used. The working principle of the test is that the apparatus generates cavitation bubbles in the oil medium using high-intensity ultrasonic waves (typically at 22 kHz or 44 kHz). The sudden implosion of these bubbles creates a very strong local hydrodynamic shear stress. This stress mechanically breaks, i.e., destroys, the central covalent bonds of the long-chain polymers (terpolymers) in the motor oil. The advantage of the method is that a very small amount of solution (20 ml) is required, and the test time is 15–60 min. The polymer solution is poured into the device's beaker and subjected to ultrasonic treatment for the required time. Based on the comparison between the solution's initial viscosity and its viscosity after the test, an idea of the polymer's stability under mechanical stress can be formed.

2.5. Determination of tribological properties

Tribological properties were determined in accordance with GOST 9490-75. The calculated indicators were critical load (CL) (*seizing load* - the mode in which the oil film and sliding of rubbing surfaces becomes difficult; in this case, the hydrodynamic friction mode is replaced by a semi-dry friction mode), welding load (WL) (*welding load* – is the force at which the rubbing surfaces are not able to slide relative to each other; moreover, high specific loads cause critical increase in temperature in the direct contact zone, which, in turn, entails welding of the contacting surfaces), the load wear index (LWI) (*load wear index* – an indicator indicating the effectiveness of extreme pressure properties in the interval between the welding load and the seizing load; the higher this indicator the better), wear spot diameter (Wd) (characteristic of anti-wear properties).

3. Results and Their Discussion

Depending on the synthesis conditions, ternary copolymers are soluble in both mineral oils and synthetic essential oils. To ensure complete solubility, the authors chose a semi-synthetic oil containing synthetic

PEE (penitaterite ether with monobasic carboxylic acids) as the oil base (SN-600:PPE (1:1) to study the indicated properties.

3.1. Analysis of rheological and depressant properties

The rheological and depressant properties of ternary copolymers blended into base oil are given in Table 1.

Table 1
Rheological and depressant properties of ternary copolymers

Ternary copolymers	**Concentration of samples in oil, %**	**Kinematic viscosity, 100°C, mm^2/s**	**Viscosity Index (VI)**	**Freezing temperature, T_{fr} °C**	**Depressor effect ΔT_{fr}, °C**
Base oil (SN-600:PEE (1:1)	0	7,70	70	minus 15	-
Allylcapronate + Styrene + BMAK	1	7,89	96	minus 24	9
	2	8,55	100	minus 30	15
	3	8,89	110	minus 32	17
Allylcaprylate + Styrene + BMAK	1	7,95	96	minus 26	11
	2	8,88	108	minus 32	27
	3	9,22	114	minus 34	19
Allylcaprinate + Styrene + BMAK	1	7,99	100	minus 28	13
	2	8,98	110	minus 34	19
	3	9,68	116	minus 36	21

Table 1 shows that when the concentration of terpolymers in oil increases from 1% to 4%, the positive effect is observed at 3%. As the alkyl chain length increases from C_6 to C_{10}, the solubility of the terpolymer in oil and the expansion (increase in volume) of the macromolecular mass at 100°C increase. This occurs because of increased thermal motion and greater flexibility of the polymer chain. The highest viscosity index (VI) (116) and the maximum depressant effect (ΔT_{fr}= 21°C; T_{fr} = minus 36°C) were recorded at a concentration of 3.0% for the terpolymer with an alkyl chain length of C_{10}.

Analysis of chemical degradation

There are several methods for assessing the thermal degradation of polymers. Since viscosity additives are used in oil solutions, it is more practical to conduct degradation tests in solution. A special method has been developed for this purpose. This method involves heating 5% solutions of viscosity additives

(terpolymers) in oil at 200°C for 12 hours. At specific time intervals, samples are taken, and their kinematic viscosities at 100°C are measured. By comparing these measurements with the initial viscosity, the viscosity decrease (VD) can be calculated. This allows for an evaluation of the thermal stability of the polymer compound.

Calculations are made according to the following form

$$VD = \frac{V_i - V_a}{V_i} \cdot 100\%$$

where - V_i initial viscosity of the oil at 100°C, mm^2/s,

V_a - viscosity after testing, mm^2/s.

The results of thermal destruction of ternary copolymers with a molecular mass of 10000 in the studied oil are given in Fig. 1 in comparison with the industrially produced polyalkylmethacrylate viscosity additive “B-2” in turbine oil “L” with the same molecular mass.

The structural evaluation of the ternary copolymers reveals a prominent structure-property relationship regarding both thickening efficiency and thermal-oxidative stability. As illustrated in Fig. 1, a direct linear correlation exists between the length of the alkyl chain in the ester fragment and the initial viscosity of the formulated oils. Specifically, increasing the carbon number from C_6 to C_{10} results in a steady rise in the initial kinematic viscosity from 10.97 mm²/s to 12.77 mm²/s. This enhancement is primarily attributed to the larger hydrodynamic volume and higher macromolecular expansion of the longer alkyl derivatives in the hydrocarbon medium.

The kinetics of thermal degradation over a 12-hour exposure period demonstrate that all ternary copolymers exhibit remarkable shear and thermal resilience compared to the commercial standard. At the intermediate 4-hour interval, the viscosity loss remains low, ranging from merely 3.6% (for the C_6 copolymer) to 3.9% (for the C_{10} copolymer). By the end of the 12-hour thermal stress test, the viscosity drop marginally increases to 4.5% (C_6), 4.6% (C_8), and 4.8% (C_{10}). The slightly higher degradation rate observed for the longer alkyl chains (C_{10}) can be attributed to their greater vulnerability to thermal cleavage under prolonged stress. A finding of this study is the comparative performance against the industrial reference, polyalkyl methacrylate B-2. Under identical testing conditions, the oil thickened with B-2 shows a 11.2% drop in viscosity at 12 hours.

The ternary copolymers exhibit significantly reduced viscosity loss, measuring less than half that observed in the commercial reference. This represents an approximate 2.5-fold increase in thermal-oxidative stability. The superior performance of these copolymers confirms that the incorporation of styrene and butyl methacrylate (BMAK) into the allyl ester backbone results in a highly rigid and robust copolymeric structure. As a result, these innovative ternary systems effectively balance high thickening capability with excellent thermal endurance, making them highly promising candidates for advanced lubricating oil formulations in modern materials applications.

3.2. Analysis of mechanical destruction

One important operating indicator for lubricants is their resistance to mechanical degradation. Mechanical destruction occurs when the carbon-carbon (C–C) bonds in the main chain are broken. The susceptibility of C–C bonds to breaking is influenced by the uneven distribution of mechanical force applied to individual bonds.

The rate of destruction increases as the degree of polymerization rises. In the case of macromolecules, the breakage of C–C bonds typically occurs near the middle of the chain. Consequently, mechanical destruction narrows the molecular mass distribution of the polymers.

The mechanical destruction of terpolymers is carried out in a base oil (SN-600: PEE (1:1) solution, and the comparative results of the mechanical destruction of synthesized copolymers with a molecular mass of 10000 and the industrial additive polyalkyl methacrylate B-2 in transformer oil are given in Table 2.

Table 2
Mechanical destruction of solutions of ternary copolymers in base oil

Ternary Copolymer			**Solution of ternary copolymer oil**			
sample	mol. mass, g/mol.	concentration in base oil, %	initial viscosity, mm^2/s	viscosity reduction during the specified time (min.), %		
				15	30	60
Allylcapronate+ Styrene+BMAK	10000	5,0	9,70	10,4	14,1	18,1
Allylcaprylate+ Styrene+BMAK	10000	5,0	9,75	10,6	14,2	18,3
Allylcaprinate+ Styrene+BMAK	10000	5,0	9,79	10,5	14,3	18,5
Polyalkyl methacrylate B-2	10000	6	9,78	18,4	21,4	26,9

As can be seen from Table 2, a comparative study of the results with the industrial additive polyalkylmethacrylates shows that the ternary copolymers have an advantage in terms of the studied properties – the viscosity reduction is 18.1%, 18.3%, 18,5% and 27.2% after 1 hour, respectively.

3.3. Analysis of tribological properties

The structure of terpolymers forms a multilayer protective film on the metal surface, which reduces friction (Scheme).

Scheme

Tribological surface mechanism

The allyl ether component in ternary copolymers contains polar groups, specifically a complex ether group. These polar groups adhere strongly to the metal surface, creating a stable adsorption-solvate layer. The medium-length C_6-C_{10} chains within the copolymers promote a "fluid friction" effect between the rubbing surfaces, which helps to reduce the friction coefficient. The copolymers' anti-wear properties can be attributed to the presence of the vinyl monomer styrene in the polymer chain, which imparts high mechanical strength to the material.

Tribological characteristics of ternary copolymers in oil are illustrated in Table 3.

Table 3
Tribological characteristics of ternary copolymer oil compositions

Samples of ternary copolymers	**Concentration of samples in oil, %**	**Tribological characteristics GOST 9490-75**		
		Wear spot diameter W_d, mm	**Wear reduction effect, %**	**Welding load P_c, N**
Base oil (SN-600:PEE (1:1)	00	1,20	0,0	1210
Allylcapronate + Styrene + BMAK	3,0	0,85	29,2	2420
Allylcaprylate + Styrene + BMAK	3,0	0,80	33,3	2580
Allylcaprinate + Styrene + BMAK	3,0	0,75	37,5	2710

As shown in Table 3, the ternary copolymer additives significantly improve the tribological properties of the base oil, with longer alkyl chains (C_6 to C_8 and C_{10}) demonstrating a synergistic increase in antiwear and extreme properties, achieving a maximum wear reduction of 37.5% (C_{10}). The modification increases the welding load from 1210 N to 2710 N ($C_{10)}$ by forming a durable, highly resistant protective tribofilm on metal surfaces under high loads.

The dependence of the wear diameter on the concentration of ternary copolymers is given in the figure below.

The tribological performance of the semi-synthetic oil compositions was evaluated by measuring the wear scar diameter (WSD) as a function of terpolymer concentration and alkyl chain length (Fig. 2). The additive-free base oil (SN-600: PEE, 1:1) exhibited a high initial wear scar diameter of 1,20 mm, indicating poor anti-wear efficiency under boundary lubrication conditions. Upon the introduction of the claimed terpolymers, a significant reduction in WSD was achieved across all samples. At the optimal concentration of 3 wt.%, the anti-wear efficiency improved progressively with increasing alkyl chain

length, yielding WSD values of 0,85 mm for C_6 (hexyl), 0,80 mm for C_8 (octyl), and a minimum of 0,75 mm for the C_{10} (decyl) terpolymer. This pronounced enhancement is attributed to the formation of a robust, high-load-bearing protective polymer film on the rubbing metal surfaces, facilitated by the optimized hydrophobic-hydrophilic balance of the longer alkyl substituents.

Because the C_6 chains (hexyl) are short, they cannot provide sufficient thickness and steric hindrance between the rubbing surfaces. Under high loads, these short chains are easily crushed, and the direct metal-to-metal contact results in a large WSD.

C_8 (octyl chains) is the transition and balance point. This chain is located between the short C_6 and the long C_{10}. Compared to the C_6 chain, the Van der Waals forces between the C_8 chains are stronger. They form a more ordered and dense molecular layer on the metal surface than the C_6 chain. For this reason, WSD is lower. Compared to the C_{10} chain, this chain does not have the best density of layer thickness of the C_{10} chain. Due to the shorter chain length, the steric repulsion force is weaker than in the C_{10} chain under extreme loads, resulting in WSD being slightly larger than that of C_{10}.

In laboratory tests, the following trend is often observed in C_8: at a concentration of 1%, C_8 is still unable to cover the surface and gives results close to C_6, but when moving to concentrations of 2% and 3%, the effectiveness of C_8 increases sharply as the number of molecules increases and its curve approaches C_{10}. This shows that the effectiveness of C_8 depends directly on concentration. On the other hand, the C_8 chain has an optimal hydrophilic-lipophilic balance (HLB) in an oily environment. Its solubility in oil is better than that of C_6. However, since it is not as large as C_{10}, the polymer chains do not tend to form associations (micelles) within the oil. This facilitates the migration of the polymer into the friction zone as free molecules.

The C_{10} (decyl) chains are longer. They are more tightly packed with each other through Van der Waals forces (intermolecular packing). As a result, a thicker, hydrophobic, and highly viscoelastic boundary layer forms, which is resistant to shear stress, leading to a decrease in WSD.

On the other hand, the elongation of the alkyl chain increases the hydrocarbon chain (hydrophobic part) of the polymer. This dramatically increases the solubility and stability in the base oil. The well-soluble C_{10} macromolecules unfold more easily in the oil environment (uncoiled conformation) and create a good solvation layer, carrying oil molecules with them to the friction zone.

4. Conclusion

In the course of the research work, new multifunctional additives with high efficiency for internal combustion engines were synthesized, and their performance properties were studied in comparison with the commercial additive used in industry. As a result of the research, the following main scientific and practical results were obtained:

The performance properties of ternary copolymers obtained by radical copolymerization of allyl esters of caproic (C_6), caprylic (C_8) and capric (C_{10}) acids with styrene and butyl methacrylate (BMA) in a semi-synthetic base oil with a composition of SN-600: PEE (1:1) were studied in comparison with the commercial additive used in industry and the following main scientific and practical results were obtained; the use of semi-synthetic oil ensured complete solubility and the optimal concentration of the additive was determined to be 3%; it was found that ternary copolymers significantly lower the freezing point of semi-synthetic engine oil at this concentration and demonstrate a high depressant effect in the range of 17–21°C; ternary copolymers showed high destruction resistance compared to existing industrial analogues in accordance with the high-temperature and heavy mechanical loading conditions of internal combustion engines; thermal decomposition during the 12-hour test period was only 4.5–4.8%, which is approximately 2.5 times more effective than the indicator of the polyalkylmethacrylate "PMA B-2" commercial additive widely used in industry (11.6%); the degree of mechanical destruction varied within 8.1–8.5%, which is approximately 3 times lower than the analogous indicator of the commercial additive (26.9%); it was proven that ternary copolymers, in addition to rheological and depressive properties, also have effective functionality against friction and wear, and the use of these additives resulted in a wear reduction effect in the rubbing parts of internal combustion engines of 29.2–37.5%.

Thus, multifunctional ternary copolymers can be recommended as promising additives in semi-synthetic oil compositions for modern internal combustion engines, since they demonstrate high rheological, thermal, and mechanical stability, as well as tribological indicators.

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Figures

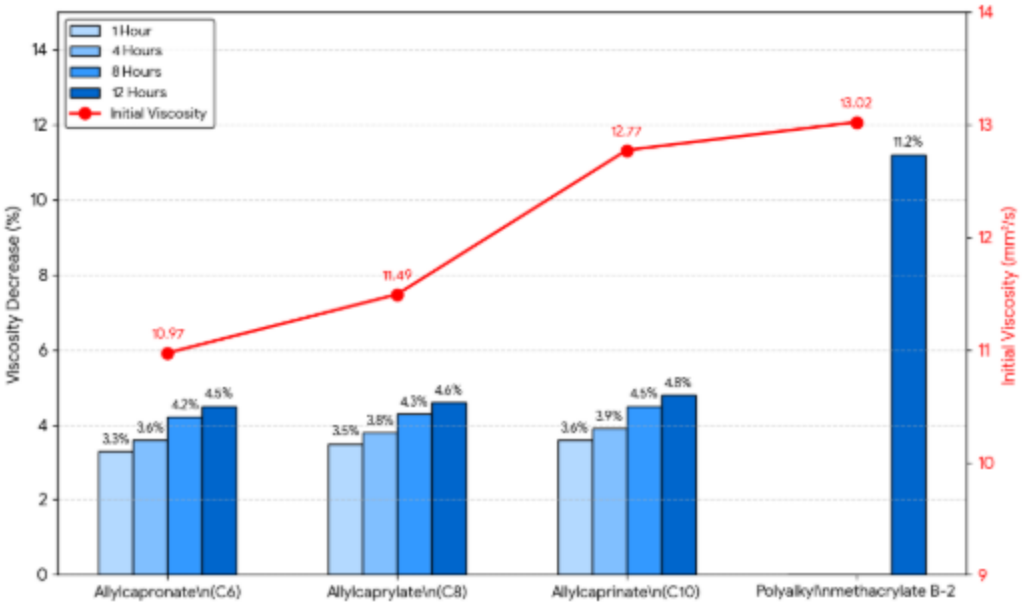


Figure 1

Comparative diagram of initial oil viscosity and thermal degradation kinetics (viscosity decrease, %) of polysynthetic base oil (SN-600: PEE 1:1) thickened by ternary copolymers as a function of alkyl chain length and exposure time (B-2 is the industrial reference)

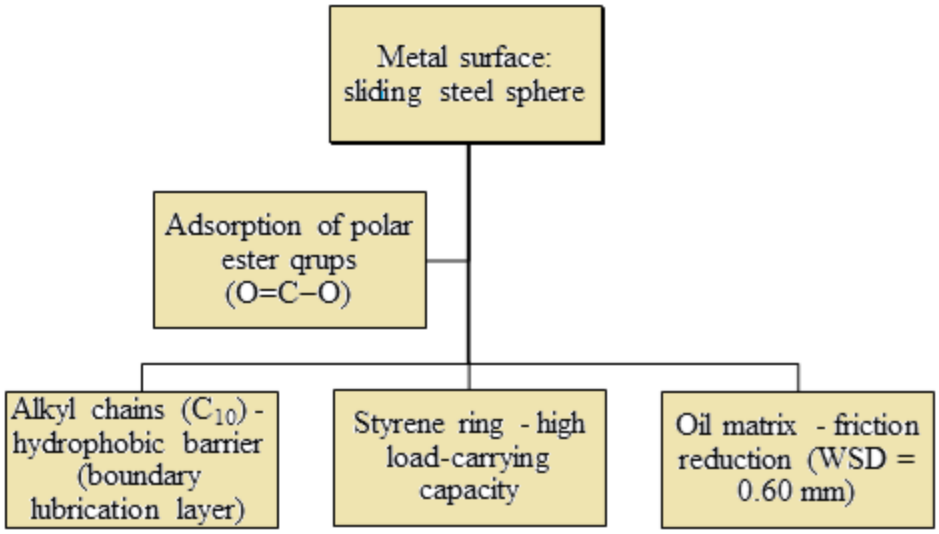


Figure 2

The dependence of the wear diameter on the concentration of ternary copolymers