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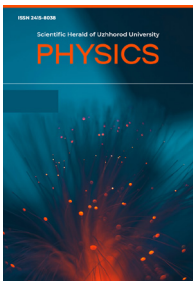

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Effect of phenol-formaldehyde resin on mechanical durability and structure of low-density polyethylene

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Abstract

Relevance. The development of technology for producing new polymer modifications with specific properties, which remain stable even when exposed to external factors, is a key area of focus for researchers in the field of high-molecular compounds.

Purpose. The purpose of this study was to create new composite materials based on low-density polyethylene.

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Methodology. The extrusion blowing method on an industrial URP 1500 unit was used for processing low-density polyethylene (LDPE) and its modified films. Electron microscopic images of the surface were captured using a S-570 scanning microscope (Japan) at X1000 magnifications.

Results. The composition and quantity of a low-molecular organic additive that alters the electrical characteristics of low-density polyethylene grade 10803-020 was ascertained. The modified low-density polyethylene is noteworthy for its minimal usage of additives and their compatibility with technology. Based on experimental data, it was found that the inclusion of 0.05 wt% phenol-formaldehyde resin in low-density polyethylene increases its mechanical strength to the highest level when compared to both the unaltered low-density polyethylene and low-density polyethylene with other additives. Furthermore, the study found that the addition of phenol-formaldehyde resin in small proportions (0.05 wt%) substantially enhances mechanical strength at varying temperatures.

Conclusions. The electrophysical characteristics of low-density polyethylene and its modified versions were comprehensively investigated. The adequately altered low-density polyethylene exhibits considerably improved mechanical durability. It was indicated that the additives used, at optimal levels, influence the physical framework of low-density polyethylene, highlighting their technological compatibility differences.

Keywords: degree of crystallinity; activation energy; lifetime, supramolecular structure; polymer; lamellas

Introduction

One of the primary obstacles confronting researchers in the field of high-molecular compounds pertains to enhancing the stability of their electrophysical and mechanical attributes. The need to cultivate new polymer adaptations with the desired features and sustained against different extraneous factors triggers the surge in polymeric materials' incorporation across multiple sectors of the national economy, instrument making and electronic industry. This is mainly because using polymers on their own lacks the necessary improvements in their electrophysical characteristics, processing techniques, stability to prolonged exposure to static and dynamic loads, as well as different radiation and climatic factors, including excessive temperatures. E.M. Godzhaev *et al.* [1] and C. Wu *et al.* [2] noted that deterioration in the mechanical properties of polymer materials occurs mainly due to ionization processes developing in air inclusions and pores inside the insulation. According to M.A. Rajab *et al.* [3], a significant breakthrough is occurring due to the new capabilities of nanotechnology, namely the ability to regulate the supramolecular structure of polymer composites.

S. Mohandesnezhad *et al.* [4] found that the most accessible methods for increasing the electrical and mechanical properties of polymers is to modify them with micro and nano additives of organic and inorganic products. The current technology used for producing polymer modifications enables the creation of mechanical and dielectric materials with diverse properties through the combination of polymers and additives. The role of micro and nano additives as crystal-forming agents, which form the fine-crystalline structure of the polymer, has also been rigorously described and studied. Sh. Zeynalov *et al.* [5] has demonstrated that the claim stating that a finely crystalline supramolecular region in polyolefins represents the most advantageous form of polymer structure, associated with a range of valuable physical and mechanical attributes, is widely held.

Extensive research has investigated changes in the mechanical strength of polymers, specifically LDPE (low-density polyethylene), with various additives. However, information regarding the influence of additives on the mechanical and electrical properties of LDPE is significantly limited. Previous studies [6] have observed changes in the mechanical strength of polymers through the introduction of fillers and additives. Thus, the strength of low-density polyethylene is enhanced to a small extent upon the addition of phthalic anhydride and orthophenylinediamine.

F. Li *et al.* [7] considered that adding phenol-formaldehyde resin sizing significantly enhances the mechanical properties of injection-molded short carbon fiber-reinforced polyethersulfone composites. This procedure leads to augmented strength, stiffness, and fracture resistance of the material. Consequently, the resulting polymer composites possess the potential for application in diverse high-tech circumstances where mechanical performance is of utmost importance. Nonetheless, this composition is deficient in mechanical strength. The mechanical strength of this composition is within 16-18 MPa [8]. Although this area has received much attention, film aging during operation is still a significant issue. The purpose of this study was to design new polymer composite materials with considerably enhanced electrical properties, as well as to minimise the rate of film aging during their operation, as well as their dependence on the supramolecular structure.

Materials and Methods

The developed modifications of LDPE were processed into films at industrial plastic processing units on the LDPE 10803-020 (Azerbaijan) grades in two stages. The proposed additives were introduced into the feedstock, granulated LDPE 15803-020 with a molecular weight of M-8.45 104 by mechanical mixing. Phenol-formaldehyde resin (PFR), chemical formula [-C6H3(OH)-CH2]n was used as an additive. The thickness of LDPE films and its compositions is 50-60 μm. Additives were introduced into LDPE by mechanical mixing. Before adding the additive to the LDPE, it was dispersed using a sieve analysis on a grain composition machine. The particle size was less than 50 μm.

The installation for sieve analysis consisted of an exhaust drive and a set of sieves. The set of sieves is selected in such a way that the entire range of grain sizes of the bulk material being tested is covered (5-6 sieves). At the very bottom there is a sieve plate to catch the last sifting. The vibration drive imparts vibration and rocking movement to the sieves installed and secured to it. The duration of sifting for coarse-grained material was 10 minutes, for fine-grained material about 20 minutes.

On a vibration drive, the time and strength of vibration were regulated by a timer. Dependency $\tau = f(\sigma)$ i.e., the time elapsed from the moment the sample was loaded until it broke, was determined using a tensile testing machine (Azerbaijan) that ensured constant mechanical stress throughout the experiment [9; 10] (Fig. 1).

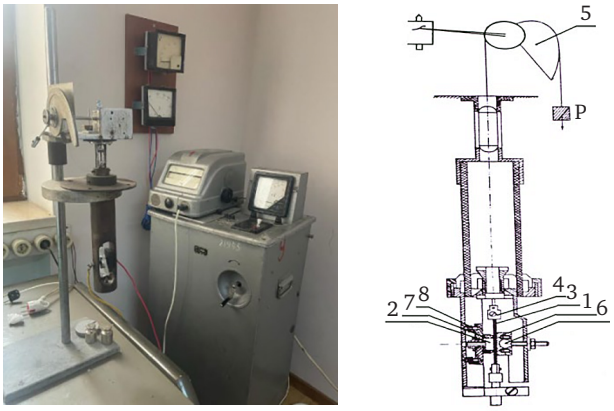

Figure 1. Installation for determining the strength of polymer film dielectrics
Note: 1, 2 – electrodes; 3 – sample; 4 — tips; 5 – lever device; 6 – ball; 7 — security ring; 8 – springs
Source: photographed by the authors of this study

LDPE and its modified variants were processed into films via extrusion blowing using an industrial unit URP-1500-2 (Russia). URP-1500-2 produces film from 30 to 150 μm, sleeve width 1500 mm (without fold), productivity ~70 kg/h, rotation unit on the head. The unit was set technological mode of processing and obtaining a film with a thickness of 20 to 50 ± 4 μm (Table 1).

Table 1. Norms of the technological mode of the unit URP-1500-2 to produce films by blowing (inflating method)

	Parameter name	Unit of measurement	The value of the measured parameter	
			Initial Raw material brand 10803-020	Raw material LDPE + 0.05 wt% PFR
1	2	3	4	5
1.	Temperature by cylinder zones	0°C		
	Zone 1	«»	110	130
	Zone 2	«»	135	150
	Zone 3	«»	140	160
	Zone 4	«»	150	170
2.	Temperature by filter zone	0°C		
	Zone 5	«»	160	170
3.	Temperature by head zones:	0°C		
	Zone 6		150	170
	Zone 7		150	170
	Zone 8		145	165

Table 1. Continued

1	2	3	4	5
4.	Auger rotation speed	rpm	20	20
5.	Load on the main motor	A	28	42
6.	Take-up roll speed	m/min.	13	13
7.	Winding roller speed	m/min.	13,5	13,5
8.	Geometric dimensions of the film:			
	• sleeve width	mm	600	600
	• film thickness	μm	20 ÷ 40 ± 4	20 ÷ 40 ± 4
9.	Performance	kg/h	25	25

Source: [11]

A qualitative assessment of the change in the structure of the LDPE film, with the introduction of small additives, was the determination of the morphology of supramolecular formations by the method of scanning electron microscopy and X-ray diffraction. Electron microscopic photographs of the surface were taken on an S-570 scanning microscope (Japan) at x1000 magnification. The samples were glued onto object machines, sprayed with a thin layer of Pt-Pd on a JB-3 installation (Japan). The morphology of supramolecular formations using scanning electron microscopy and X-ray diffraction analysis was carried out at the Azerbaijan State University.

Results and Discussion

Figures 2 and 3 illustrate the time dependences of mechanical strength for pure LDPE (3) and with additives of 0.01, 0.05, 0.7 and 0.1 wt% phenol-formaldehyde resin (4, 1, 2, 3, and 5), respectively, in semi-logarithmic coordinates $lg\tau = f(\sigma)$ and dependence $lg\tau$ on mechanical load σ at different temperatures (163, 133, 103K) for the original LDPE and for its optimum modification LDPE + 0.05 wt% PFR (Fig. 3).

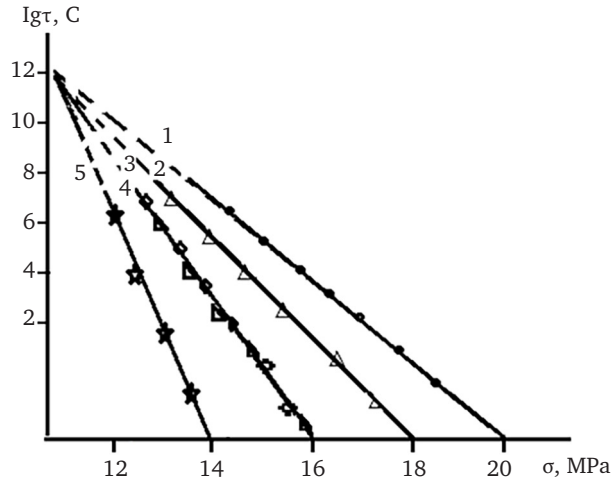

Figure 2. Dependence of the mechanical strength of LDPE (3) and its composition with additives 0.05 wt% (1), 0.07 wt% (2), 0.01 s% (3), 0.1 wt% (PFR)
Source: developed by the authors of this study

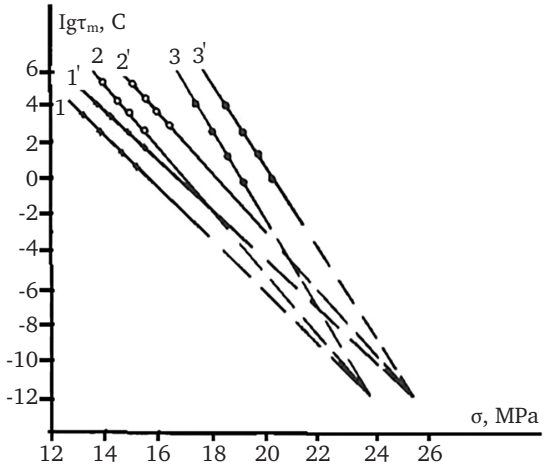

Figure 3. Force dependences of the durability of the LDPE film (1-3) and its optimal modification LDPE + 0.05 wt% PFR (1′-3′) at different temperatures (T, K); 1.1-163; 2.2-133; 3.3-103K
Source: developed by the authors of this study

The functional relationship between durability τ_m, mechanical stress σ and temperature T was obtained for a wide class of materials [12; 13].

$$\tau_\mu = \tau_0 e^{\frac{U_0 - \alpha\sigma}{KT}}, \quad (1)$$

where τ_0, U_0 and α are parameters that determine the strength properties of the material under study.

As the above dependence suggests, the durability of these samples clearly depends on the magnitude of the breaking stress σ; with a decrease in voltage, the durability τ increases greatly, i.e., the well-known equation for durability is fulfilled at $T = const$:

$$\tau = Aexp(-\alpha\sigma), \quad (2)$$

where the parameter A and α are constant coefficients that determine the dependence of durability on stress at a constant temperature at which tests are carried out. The linear dependence $lg\tau$ on σ is also justified at different temperatures (163, 133, 103K) for the original LDPE and for its optimal modification.

As Figures 2 and 3 suggest, the introduction of 0.05 wt% phenol-formaldehyde resin into LDPE leads to a significant increase in the mechanical strength of the polymer composition by more than 20%. The calculated values of the parameters of the LDPE film and its optimum modification are presented in Table 2.

Table 2. Values σ_0, U_0, α for LDPE and its optimal modification

Material	σ_0, C	$U_0 \frac{\text{kcal}}{mole}$	$\alpha, \frac{\text{kcal}}{mole} \mu P\alpha$	σ, $\mu P\alpha$ 163K
LDPE (no additive)	10^{-12}	21	0,073	16,2
LDPE + 0.05 wt.% phenol-formaldehyde resin	10^{-12}	21	0,061	20

Source: developed by the authors of this study

From the data of the tables obtained as a result of testing the LDPE film and its optimal modification at a constant temperature (133K) and a constant rupture time ($\tau = 1c$), it can be seen that the coefficients U_0 and τ_0 are constant for samples from the LDPE film and its optimal modification. The proposed additive leads to a decrease in the α coefficient and a significant increase in mechanical strength (from 16.2 for the original LDPE to 20 MPa for the modified one). The constancy of the values and activation energy U_0 of the original LDPE and its improved modification is mainly due to the same chemical bonds U_0 [14; 15]. The mechanical strength of the LDPE film noticeably increases when 0.05 wt% PFR is included in its composition. This can be attributed to the supramolecular fine spherulite structure that forms within the LDPE. The investigation into LDPE with the use of scanning electron microscopy allowed for a greater comprehension of the supramolecular arrangement of the examined specimens. Figures 4 and 5 display micrographs of the primary PE (polyethylene) and PE with the inclusion of phenol-formaldehyde resin.

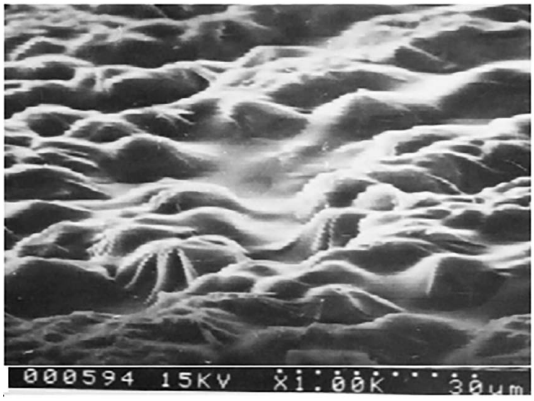

Figure 4. Micrographs of LDPE film
Source: developed by the authors of this study

Figure 5. Micrographs of LDPE film with the content of the modifying additive (PFR) 0.05 wt%
Source: developed by the authors of this study

Observations reveal that the introduction of additives into LDPE at a concentration of 0.05 wt% PFR leads to the growth of larger structural formations, resulting in an enhanced continuity or packing organization. The resulting structural elements are in the nature of lamellas intertwined within a certain polymer microvolume and at the same time passing in the form of an intrusion into neighbouring structural elements similar in structure. H. Hamadache *et al.* [16] noted that to acquire a three-dimensional image of the surface, a scanning electron microscope is utilised by employing electrons that are either scattered or emitted by the sample's surface. In this case, the sample is fixed, dried, and coated with a thin film of heavy metal, and then scanned with a narrow beam of electrons. R. Hsissou *et al.* [17] discussed the different types of polymer composites, including polymer-matrix composites (PMCs), metal-matrix composites (MMCs), and ceramic-matrix composites (CMCs). Each type had unique properties and applications. It explored the various reinforcement materials used in polymer composites, such as carbon fibres, glass fibres, and nanoparticles. Additionally, the choice of polymer matrix, such as epoxy, polyester, or thermoplastics, was discussed in detail. This review covered common manufacturing processes for polymer composites, including methods like hand layup, filament winding, and resin transfer moulding (RTM). Each process is explained, along with its advantages and limitations. V. Biziks *et al.* [18] focused on evaluating the changes in the mechanical properties of LDPE when it is combined with phenol-formaldehyde resin. Mechanical

properties include attributes like tensile strength, impact resistance, and elasticity. The study aimed to determine whether the addition of phenol-formaldehyde (PF) resin enhances or alters these properties. To mechanical testing, the study conducted a structural analysis of LDPE after the introduction of phenol-formaldehyde resin. This analysis involved techniques such as microscopy or spectroscopy to examine changes in the molecular and crystalline structure of the material. Understanding how PF resin affected LDPE's mechanical properties and structure is relevant to industries that utilize LDPE in composite materials.

In the unaltered polyethylene, the nature of structure concentrators is similar to those of the modified samples, but with the difference that they often represent separate, unconnected islands. Based on microphotography data and a comparative analysis, we can assert that PFR microadditives can accelerate the process of structure formation on the supramolecular surface. These microadditives penetrate mainly into the amorphous phase of the polymer, creating centres for structure formation. The elements of these centres then intertwine with polymer crystallites, forming a single, ordered structure with a high degree of packing. It is evident that PFR microadditives have a significant impact on structure formation. The polymer's structure does not take the form of spherulitic formations, as many researchers believe. Instead, it has a lamellar structure. The added additive accelerates the polymer's crystallization and creates larger structural elements composed of lamellas bound in various directions, irrespective of the film's orientation. E.U. Van Velzen *et al.* [19] researched that Recycled polyethylene (PE) is widely used in various applications, including packaging, construction, and automotive industries. During recycling processes, impurities such as contaminants, additives, or residues from previous use may be introduced into the recycled material. The mechanical properties of PE, including tensile strength, elongation, impact resistance, and stiffness, are critical for determining its suitability for specific applications. Understanding how impurities influence these properties is essential for ensuring the quality and performance of recycled PE products. Impurities in recycled PE can be diverse and include foreign particles, residues of other polymers, pigments, additives, and degradation products. Each type of impurity may have a different effect on the mechanical properties of the material. Y. Hiejima *et al.* [20] and N. Darie-Niţă *et al.* [21] noted that LDPE is a widely used polymer in various applications, including packaging materials. However, when exposed to environmental factors such as UV radiation, LDPE can undergo photodegradation, leading to changes in its chemical and structural properties. The study delves into the subtle structural modifications that occur at a microscopic level within LDPE when subjected to photodegradation. These changes may not be visible to the naked eye but can significantly affect the material's properties [22].

An improved organisation of the polymer surface should lead to an increase in the degree of crystallinity. Nevertheless, according to the experiment, the degree of crystallinity barely changes. This is also supported by the X-ray diffraction experimental results outlined in Table 3.

Table 3. Size and degree of crystallinity of LDPE and its optimum modifications

Material	Crystal size	Degree of crystallinity %
LDPE 10803-020 (original)	126	41
LDPE 10803-020 + 0.05 wt% PFR	130	40

Source: developed by the authors of this study

The obtained data enable a scientific approach to explaining experimental results, establishing the correlation between polymer product structure and properties, and conducting a focused search for the optimal alloying additives that can enhance the physical, mechanical, and technological characteristics of the polymer [23]. Consequently, through comprehensive analysis of modified low-density polyethylene samples with the inclusion of phenol-formaldehyde resin, we found the connection between the supramolecular structure type and the physicochemical, strength, and technological attributes of polymer products. This study investigated the temperature-time dependence of the mechanical strength of LDPE (low-density polyethylene) polymer films and its modifications under the simultaneous action of a mechanical load on them. The mechanical load value was selected to prevent any stretching or rupture of the test specimens caused by orientation.

Conclusions

Polymer composite materials based on low-density polyethylene grade 10803-020 containing a significantly small amount of the organic additive phenol-formaldehyde resin were obtained. It was revealed that compositions with additives of 0.05 wt% phenol-formaldehyde resin significantly improve the physical, mechanical, and technological properties of the polymer, and the mechanical strength of the material increases significantly. In addition, it was found that phenol-formaldehyde resin in a small amount (0.05 wt%) significantly increases mechanical strength at various temperatures.

The study showed that a change in the properties of LDPE when modified by the addition of phenol-formaldehyde resin (0.05 wt%) is reflected in changes in the structure-sensitive coefficient α alone (and an increase in strength properties corresponds to a decrease in α, and their decrease-increase in α). When adding phenol-formaldehyde resin additives to LDPE, a decrease in local stresses occurs (a decrease in the coefficient α), then according to the proposed mechanism, the probability of perturbation of chemical bonds decreases, as a result of which the mechanical strength increases.

The microstructures of low-density polyethylene, which were modified with phenol-formaldehyde resin, underwent analysis through the use of electron scanning microscopy and x-ray diffraction techniques. Based on microphotography data and a comparative analysis of the structure, we can confidently state that microadditives of phenol-formaldehyde resin can accelerate structure formation on a low-molecular-weight surface. By primarily introducing these additives into the amorphous phase of the polymer, they create formation centres. The resulting elements interweave with the polymer's crystallites, culminating in a solitary ordered structure with a superior degree of packaging. The polymer's structure deviates from spherulitic formations, once believed by most researchers, and instead exhibits a lamellar structure. The added additive's capacity to hasten polymer crystallization generates larger structural components consisting of lamellae that are interwoven in different directions, irrespective of the film's orientation.

Thus, as a result of comprehensive studies of samples of modified low-density polyethylene phenol-formaldehyde resin in the optimal amount (0.05 wt%) according to the physicochemical, strength and technological characteristics of film samples, we have established the relationship between the type of supramolecular structure and the properties of polymer products. The ability of the organic additive phenol-formaldehyde resin to act as a structure former has been shown. It has been established that the specified organic additive is an effective lamellar structure former. It has been shown that this type of structure contributes to the production of a new polymer composition with specified physical, mechanical, electrophysical and technological properties.

To determine the most suitable conditions for processing and operating polymers, one must have a comprehensive understanding of their behaviour in the crystalline glassy and viscous-flow states, as well as the corresponding transition patterns between these states. In this respect, it is intriguing to assess the impact of precise minor additives on the speed and procedure of polyethylene crystallization. The study examined dielectric properties, such as specific volumetric electrical resistance, dielectric losses, and the dielectric constant of the polymer composition under review.

Further investigation into the physical and chemical properties of these materials could involve examining their interactions and the impact these interactions have on the mechanical strength of LDPE.

Acknowledgements
None.

Conflict of Interest
The authors of this study declare no conflict of interest.

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Вплив фенолформальдегідної смоли на механічну міцність і структуру поліетилену низької щільності

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Анотація

Актуальність. Одним з основних актуальних завдань, що стоять перед дослідниками, які працюють у галузі високомолекулярних сполук, є розробка технології одержання нових модифікацій полімерів із заданим комплексом властивостей та відносно стабільних по відношенню до впливу зовнішніх чинників.

Мета. Метою науково-дослідної роботи було створення нових композиційних матеріалів на основі поліетилену низької густини.

Методологія. Переробку поліетилену низької щільності та його розроблену модифікацію в плівки здійснювали методом екструзійного роздування на промисловій установці марки URP 1500. Електронно-мікроскопічні фотографії поверхні виконували на скануючому мікроскопі S-570 (Японія) при збільшенні X1000.

Результати. Визначено склад та кількість низькомолекулярної органічної добавки, що модифікує електричні властивості поліетилену низької густини марки 10803-020. Розроблена модифікація поліетилену низької густини відрізняється значно меншою кількістю використаних добавок та їх технологічною сумісністю. На основі експериментальних даних визначено, що за вмісту 0,05 мас. % фенолформальдегідної смоли в поліетилені низької густини його механічна міцність досягає максимального значення порівняно як з вихідним поліетиленом низької густини, так і з поліетилену низької густини з іншим вмістом добавок. Встановлено, що фенолформальдегідна смола в невеликій кількості (0,05 мас. %) суттєво підвищує механічну міцність за різних температур.

Висновки. Проведено систематичне дослідження електрофізичних властивостей ПНГ та його розроблених модифікацій. Оптимально модифікований поліетилен низької густини має суттєво підвищену механічну міцність. Показано, що використані добавки в оптимальному вмісті впливають на фізичну структуру поліетилену низької густини, тобто відрізняються технологічною сумісністю.

Ключові слова: іступінь кристалічності; енергія активації; час життя; надмолекулярна структура; полімер; ламелі