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REGULARITIES OF OF HYBRID GELS CRYSTALLIZATION ON THE BASIS OF FUNCTIONALIZED LOW DENSITY POLYETHYLENE AND CLINOPTILOLITE

[1] R.V. Kurbanova, [2] N.T. Kakhramanov, [2] Kh.V. Allahverdiyeva, [2]F.A. Mustafayeva, [2]S.R. Abdalova

[1]Azerbaijan State Oil and Industry University,
Azadlg str. 34, Baku, AZ 1000
[2]Institute of Polymer Materials of the National Academy of Sciences of Azerbaijan,
124, S.Vurgun str., Sumgait, AZ 5004, e-mail: najaf1946@rambler.ru

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Abstract: The step dilatometry method was used to study the effect of the concentration of dressed clinoptilolite on the temperature dependence of the specific volume and free specific volume, kinetic regularities, and the crystallization mechanism of nanocomposites based on low-density polyethylene chemically modified with maleic anhydride. The concentration of dressed clinoptilolite in the composition of maleated low density polyethylene varied in the range of 0.5 – 20 wt. %. It was shown that in the process of stepwise cooling, a first-order phase transition for nanocomposites with a filler concentration in the range of 0.5 – 10 wt. % occurs at a temperature of 88°C, and at a concentration of 20 wt. %, this process occurs at 84°C. An interpretation and theoretical justification are given for crystallization processes occurring in the temperature range of 20-170°C. It was found that the dilatometric method allows the graphical method to determine the glass transition temperature of nanocomposites depending on their composition. With an increase in the concentration of dressed clinoptilolite, a regular increase in the glass transition temperature of nanocomposites occurs.

It is established that with the introduction of up to 5.0% of the mass crystallized clinoptilolite into maleinized polyethylene the crystallization process or phase transition of the first kind proceeds with the formation of disk-shaped crystalline structures from sporadic crystallization centers

***Keywords:** nanocomposite, crystallization, clinoptilolite, maleic anhydride, low density polyethylene*
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Introduction

Along with the improvement of engineering and technology for industrial production, an emphasis is laid on the development of new types of polymer structural materials capable of satisfying the increased demands caused by scientific and technological progress. The latter circumstance is achieved due to chemical or mechanochemical modification of the structure and properties of basic industrial polyolefins, by loading mineral fillers, plasticizers, stabilizers, by mixing bipolar polymers, etc. [1-3]. The development of high quality composite materials is one of the simple and innovative directions for polyolefins modification. The loading of fillers in the composition of polyolefin can to some extent improve strength properties of composites. However, the lack of compatibility of the non-polar polyolefin with the polar filler does not allow the full use of the foreign body resource in changing the properties of the polymer composite. In this regard, in recent years, the interest of researchers in using chemically modified polyolefin and dressed filler particles has significantly increased which to larger extent allowed us to get nearer to the solution of the problem of compatibility of mixture components. This is not physical but rather chemical interaction of mixture components [4-6].

But the problem is solved not only by creating a polymer composite of the required quality, but also by the proper selection of the technological mode of their processing, for example, by means of injection molding. When it comes to polymer processing, what we primarily meant is a correct selection of temperature regime of the material cylinder of injection machine and the cooling of a molded product [7, 8]. In the first case, the problem is clarified by conducting rheological studies in a wide range of temperatures and shear stresses. The second problem is solved in the course of selecting an optimal temperature regime for cooling the polymer material in the mold by conducting a complex of dilatometric studies into regularities of their crystallization [9, 10].

Therefore, in this paper, the main attention is paid to the study of mechanism and kinetic regularities of crystallization of a functionalized polyolefin with dessed nanoparticles of the natural mineral filler.

Experimental part

The initial object of study was low-density polyethylene (LDPE) with properties as follows: ultimate tensile strength- 13.1 MPa, bending modulus - 196 MPa, density - 927 kg/m^3, elongation at break-720%, heat resistance-85°C, melting point - 101°C, MFI = 1.3g/10min., crystallinity 60%.

Maleinized LDPE - (PEMA) was used as chemically modified LDPE. The concentration of maleic anhydride (MA) in the composition of PEMA was 5.6 wt. %. PEMA was obtained in the process of mechanochemical synthesis or reaction extrusion. Grafting of maleic anhydride is usually carried out mainly in the most vulnerable areas of the LDPE macrochain: end, α-methylene and transvinylylene double bonds, as well as at the place of breaking the macrochains via -C-C- bonds during the thermomechanical effect of reaction extrusion.

Clinoptilolite (CTL) of the Agdag deposit of Azerbaijan with typical oxide formula being $(Na_2K_2)OAl_2O_3 \cdot 10SiO_2 \cdot 8H_2O$. The finely dispersed CTL was presented by the Institute of Geology and Geophysics of the Azerbaijan National Academy of Sciences.

CTL nanoparticles were obtained in an A-11 analytical mill at a maximum speed of 30000 rpm.

The size of the nanoparticles was determined by means of the STA PT1600 Linseiz model (Germany) with 20-110 nm.

Before the CTL nanoparticles had been loaded into the PEMA, the filler was subjected to sizing for 8 h at a temperature of 90°C in acidified HCl (pH = 3.5) distilled water in the presence of the organosilicon compound AGM-9 (γ-aminopropyltriethoxysilane) - $H_2NC_3H_6-Si(OC_2H_5)_3$. As a result of the alkoxide method of sol-gel synthesis between hydroxyl groups of AGM-9 and CTL molecules, a covalent bond was formed on the surface of nanoparticles which, ultimately in the form of a "web" - a thin monolayer of a cross-linked structure of the organosilicon compound, formed "hybrid gels".

Polymeric nanocomposites based on PEMA and finished CTL (DCTL) were obtained by mixing on rollers at a temperature of 170°C. After the polymer matrix being melted on rollers, DCTL was loaded in portions for 8 minutes.

Dilatometric studies were performed on an IIRT-1 device converted into a dilatometer. Also, step dilatometry was carried out at a load of 5.3 kg and in the temperature range from 170 ° C to a room temperature.

Results and discussion

The study into dependence of the specific volume on temperature allows to obtain reliable information about mechanism and regularities of the crystallization of polymer composite materials. It should be noted that the research literature provides insignificant data on the issue [11,12]. According to the theory of the crystallization of polymers from the melt, it consists of two main stages: nucleation and growth of crystalline formations. The nucleation process itself proceeds at homogeneous and heterogeneous centers. Homogeneous nucleation centers are formed and disintegrated until thermodynamic equilibrium is established at a given temperature. As a result of random fluctuation interaction in the melt of oriented macrosegments of polymer macromolecules, primary microcrystalline regions arise.

Heterogeneous nucleation centers are induced as a result of interaction with the surface of the filler nanoparticles [12-14]. The chemical modification of LDPE with maleic anhydride allows us to solve one of the important problematic issues related to improving the compatibility of the polymer matrix with nanoparticles. Along with this, we used dressed clinoptilolite (DCTL) as filler. In this case, the process of interaction of the modified polymer matrix occurs with the AGM-9 dressing available on the surface of the nanoparticles.

Taking into account that in the literature these issues were not given sufficient attention, it seemed interesting to study in more detail the effect of dressed nanoparticles on the kinetic regularities of crystallization of composites.

Fig. 1 shows the regularities of changes in dilatometric curves of nanocomposites based on PEMA and DCTL. Analysis of the curves in this Fig. makes it possible to establish that as the concentration of dressed DCTL nanoparticles increased, a regular decrease in the specific volume was observed.

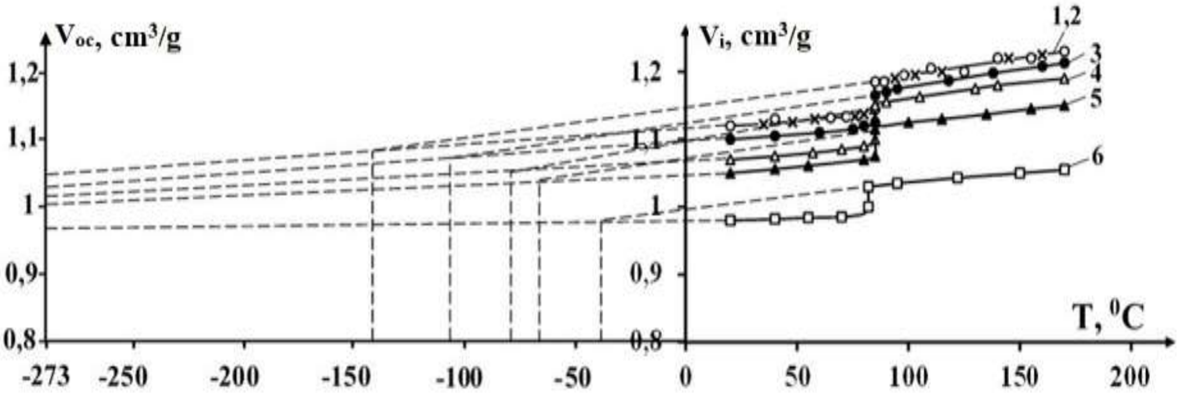

Fig. 1. Effect of the DCTL concentration on the temperature dependence of the specific volume of PEMA-based nanocomposites, wt. %: 1(o) - initial PEMA; 2(x) -0.5; 3(•) -1.0; 4(Δ) - 5.0; 5(▲) - 10; 6(□) - 20.

The decrease in the specific volume of nanocomposites caused by rise in the concentration of DCTL indicates increase in their density. It should be noted that in the process of stepwise cooling of samples, the first-order phase transition or crystallization temperature occurrs at a temperature of 84-88°C. So, for example, the crystallization temperature for the original PEMA and nanocomposites with DCTL content of up to 10 wt. % proceeds at 88° C. A relatively low crystallization temperature is observed for nanocomposite with high DCTL content (20 wt. %). In this case, decrease in the phase transition temperature of a nanocomposite with a 20 wt. % DCTL is due to the fact that with high content of nanoparticles in the volume of the polymer matrix steric difficulties arise in the crystallization and crystal growth zone. As crystalline formations grow from heterogeneous centers on the surface of the dressed nanoparticles and from homogeneous crystallization centers, a part of free nanoparticles is pushed into the amorphous region. Thus, the interphase amorphous region in the process of crystal growth accumulated structural and partially cross-linked structural units with macro-chains [14, 15]. The latter, most likely, create spatial difficulties at the very initial stage of the crystallization process that accordingly affects the decrease in the temperature of the first-order phase transition in a highly filled composite with 20 wt. % DCTL.

Dilatometric curves make it possible to determine the temperature of the second-order phase transition, i.e. glass transition temperature of nanocomposites. The temperature is determined at the intersection of upper and lower branches of the dilatometric curve. As can be seen from Fig. 1, the glass transition temperature of the initial PEMA is -140° C. In nanocomposites with DCTL concentration of 0.5; 1.0; 5.0; 10 and 20 wt. %, the value of this indicator, respectively, increases in the following sequence: -140; -105; -77; -62; -35°C. Such a noticeable increase in the glass transition temperature of nanocomposites starting from 1.0 wt. % DCLT indicated that the emergence of fairly strong Van der Waal bonds at the polymer – dressing size nanoparticle interface. Such an increase in these forces can only be associated with the participation of grafted MA molecules in chemical crosslinking reactions as a result of the sol-gel MA reaction, dressing size and hydroxyl groups present on the surface of DCTL particles [16].

By analyzing the curves in fig. 1, it can be established that if the lower branch of the dilatometric curve is extrapolated to a temperature of absolute zero (-273°C), then the "occupied volume" (V_{oc}) can be determined.

Knowing the specific volume at any temperature, it was possible to determine the free specific volume (V_f) from the difference V_i-V_{oc} [14, 17].

Fig. 2 shows dilatometric curves of the temperature dependence of the free specific volume. Comparison of the curves in Fig. 2 said that as the concentration of DCTL increased, a regular decrease in V_f was observed. This circumstance unequivocally confirms our arguments over the concentration of the most of solid particles in the amorphous space of the interfacial region. It is this region that accounts for most of the free volume.

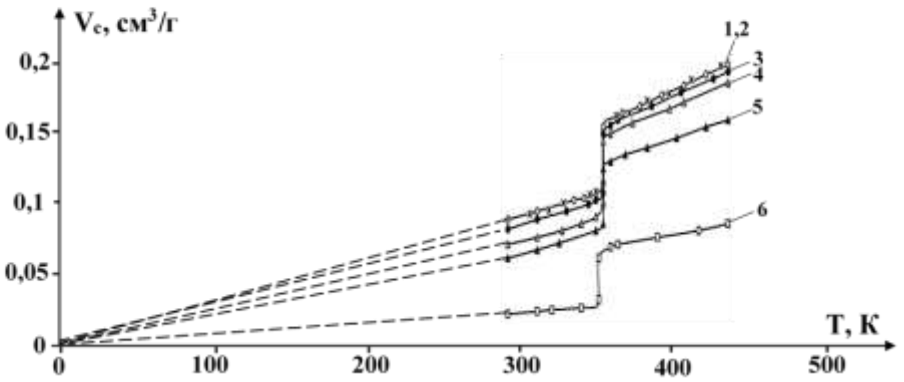

Fig. 2. Effect of the DCLT concentration on the temperature dependence of the free specific volume (V_f) of PEMA-based nanocomposites, wt. % : 1(o) - initial PEMA; 2(x) -0.5; 3(•) -1.0; 4(Δ) - 5.0; 5(▲) - 10; 6(□) - 20.

A decrease in the free specific volume with an increase in the concentration of the dressed DCTL to 20 wt. % indicates that the nanoparticles do not lead to loosening of the crystal structures of the nanocomposites. Otherwise, as large concentrations of nanoparticles were loaded, the reverse process would be observed, i.e. an increase in the free specific volume. Consequently, the dressed DCTL contributes to the compaction of the nanocomposite structure and the strengthening of its strength characteristics [11, 13, 15]. It is characteristic that in this case a sharp jump in the free specific volume is observed in the region of the first-order phase transition. Such a change in the value of this indicator is interpreted as follows: in the process of stepwise cooling, a decrease in specific volume occurrs due to a decrease in the volume fraction of the free specific volume.

It is known that kinetic measurements of the crystallization process in the region of a first-order phase transition are interpreted through the use of the Avrami model characteristic of phase transformations in metals. Studies in this area showed that they were also applicable to the study of kinetic processes of crystallization of polymeric materials. According to this theory, the crystallization process is described using the expression:

$$\varphi = e^{-K\tau^n} \qquad (1)$$

where φ - s the part of the polymer that has not yet undergone transformation into a crystalline phase; K - is the generalized constant of nucleation and crystal growth; n – is a constant ranging from 1-4; τ- is the crystallization time, (sec). Its value depends on the nature of the nucleation and growth process [14].

If V_o is the initial specific volume of the polymer at T_{cr}, V_τ is the specific volume at the time τ, V_∞ is the final specific volume of the polymer at a given crystallization temperature in the phase transition region, then the ratio $(V_o - V_\tau)/(V_o - V_\infty)$ is a part of the polymer that underwent transformation into a crystalline state at time τ. In this case, φ in equation (1) is:

$$\varphi = 1 - (V_o - V_\tau)/(V_o - V_\infty) \qquad (2)$$

The double logarithm of the Avrami equation gives:

$$\lg(-\ln\varphi) = \lg K + n\lg\tau \qquad (3)$$

The above equation is a straight line in the coordinates $\lg(-\ln\varphi)$ of $\lg\tau$. As a result of experimental studies, the applicability of the Avrami theory to the study of the crystallization process of the nanocomposites under study was established.

Fig. 3 shows kinetic regularities of crystallization of nanocomposites based on PEMA and dressed DCTL which make it possible to determine the growth mechanism of crystalline formations in a polymer matrix. Based on the results of the studies, n values were determined to characterize the growth mechanism of crystalline formations in the polymer matrix; according to the obtained kinetic curves, it found that with an increase in the concentration of DCTL in the composition of PEMA within 0.5; 1.0; 5.0; 10; 20 wt. % the value of n changes accordingly in the following sequence: 2.6; 2.6; 2.1; 1.7; 1.6. The initial PEMA had a n value of 2.3.

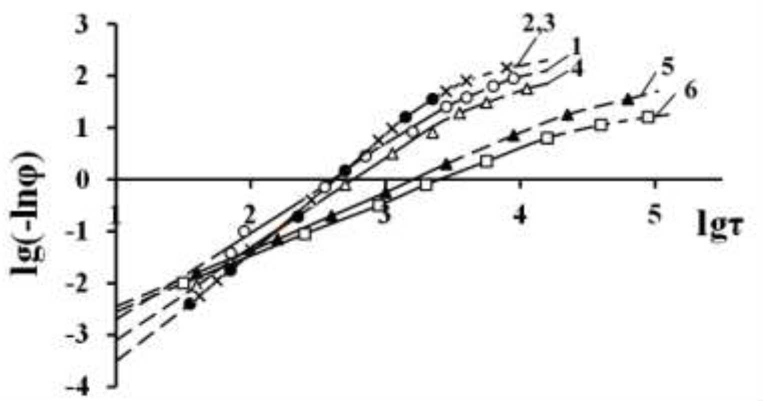

Fig. 3. Effect of the DCLT concentration on kinetic regularities of crystallization of nanocomposites based on PEMA, wt. %: 1(o) - initial PEMA; 2(x) - 0.5; 3(●) - 1.0; 4(Δ) -5.0; 5(▲) - 10; 6(□) - 20.

From a comparative analysis, it can be established that n has fractional values to indicate that crystal growth occurs from two heterogeneous nucleation centers: homogeneous and heterogeneous. Values of the nucleation constant (K) for nanocomposites changes in the following sequence are as follows: -3.5; -3.5; -3.3; -2.5; -2.4.

For nanocomposites with 0.5–5.0 wt. % DCTL content, the n value varies within 2.1–2.6 which corresponds to the growth of disk-like crystalline formations from sporadic crystallization centers. Nanocomposites with a 10–20 wt. % DCTL content and with n equal to 1.6–1.7 are characterized by the growth of rod-shaped crystalline formations from sporadic nucleation centers. The obtained results prove that at relatively high concentrations of DCTL, nanoparticles can essentially affect the growth mechanism of crystalline formations.

Based on the obtained experimental data, the Avrami equation (3) determined empirical dependences allowing one to establish, in a given value of the crystallization time (τ), in the region of a first-order phase transition the amount that underwent into the crystalline phase of the polymer matrix in nanocomposites:

(0.5 and 1.0 wt. % DCTL) $\lg(-\ln\varphi) = \lg(-3.5) + 2.6\lg\tau$ (4)

(5.0 wt. % DCTL) $\lg(-\ln\varphi) = \lg(-3.3) + 2.1\lg\tau$ (5)

(10 wt. % DCTL) $\lg(-\ln\varphi) = \lg(-2.5) + 1.7\lg\tau$ (6)

(20 wt. % DCTL) $\lg(-\ln\varphi) = \lg(-2.4) + 1.6\lg\tau$ (7)

Given the crystallization time, it is possible to determine from the equations (4-7) that part of the nanocomposite that has already undergone transformation into the crystalline phase.

Thus, on the basis of the foregoing, it can be concluded that the loading of dressed nanoparticles into the PEMA promotes the formation of supramolecular structure where the crystalline phase depends entirely on the DCTL concentration. With an increase in DCLT concentration to 5.0 wt. %, the crystallization mechanism in nanocomposites changes from disk-like to rod-shaped crystalline formations formed from sporadic nucleation centers.

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ЗАКОНОМЕРНОСТИ КРИСТАЛЛИЗАЦИИ ГИБРИДНЫХ ГЕЛЕЙ НА ОСНОВЕ ФУНКЦИОНАЛИЗИРОВАННОГО ПОЛИЭТИЛЕНА НИЗКОЙ ПЛОТНОСТИ И КЛИНОПТИЛОЛИТА

[1] Р.В. Курбанова, [2] Н.Т. Кахраманов, [2] Х.В. Аллахвердиева, [2]Ф.А. Мустафаева, [2]С.Р. Абдалова

[1]Азербайджанский государственный университет нефти и промышленности,
Azadlg str. 34, Baku, AZ 1000
[2]Институт полимерных материалов Национальной АН Азербайджана,
AZ 5004, Сумгаит, ул. С.Вургуна, 124, e-mail: najaf1946@rambler.ru

Методом ступенчатой дилатометрии изучено влияние концентрации аппретированного клиноптилолита на температурную зависимость удельного объема и свободного удельного объема, кинетические закономерности и механизм кристаллизации нанокомпозитов на основе химически модифицированного малеиновым ангидридом полиэтилена низкой плотности. Концентрацию аппретированного клиноптилолита в составе малеинизированного полиэтилена низкой плотности варьировали в интервале 0.5 – 20%масс. Показано, что в процессе ступенчатого охлаждения фазовый переход первого рода для нанокомпозитов с концентрацией наполнителя в пределах 0.5 – 10%масс. происходит при температуре 88°С, а при концентрации 20%масс. этот процесс протекает при 84°С. Дается интерпретация и теоретическое обоснование процессам кристаллизации, протекающим в температурном интервале 20-170°С. Найдено, что метод дилатометрических исследований позволяет графическим методом определить температуру стеклования нанокомпозитов в зависимости от их состава. С увеличением концентрации аппретированного клиноптилолита происходит закономерное повышение температуры стеклования нанокомпозитов. Установлено, что при введении до 5.0%масс. аппретированного клиноптилолита в состав малеинизированного полиэтилена процесс кристаллизации или фазовый переход первого рода протекает с образованием дисковидных кристаллических структурных образований из спорадических центров кристаллизации. При концентрации аппретированного клиноптилолита 10-20%масс. образуются стержневидные кристаллические структуры из спорадических центров кристаллизации.

Ключевые слова: *нанокомпозит, кристаллизация, клиноптилолит, малеиновый ангидрид, полиэтилен низкой плотности*

FUNKSİONALLAŞDIRILMIŞ AŞAĞI SIXLIQLI POLİETİLEN VƏ KLİNOPTİLOLİT ƏSASINDA HİBRİD GELLƏRİN KPİSTALLAŞMA QANUNAUYĞUNLUQLARI

[1]R.V. Qurbanova, [2]N.T. Qəhrəmanov, [2]X.V. Allahverdiyeva, [2]F.Ə. Mustafayeva, [2]S.R. Abdalova

[1]Azərbaycan Dövlət Neft və Sənaye Universiteti
[2]Azərbaycan Milli Elmlər Akademiyası Polimer Materialları İnstitutu
e-mail:najaf1946@rambler.ru

Pilləli dilatometriya üsulu ilə appretləşdirilmiş klinoptilolitin miqdarının malein anhidridi ilə kimyəvi modifikasiya olunmuş aşağı sıxlıqlı polietilen əsasında olan nanokompozitlərin xüsusi həcminin və sərbəst həcminin temperatur asılılığına, kristallaşmanın kinetik qanunauyğunluqlarına və mexanizminə təsiri öyrənilmişdir. Maleinləşdirilmiş aşağı sıxlıqlı polietilen tərkibində appretləşdirilmiş klinoptilolitin miqdarı 0.5-20 kütlə % miqdar intervalında dəyişdirilmişdir. Doldurucunun miqdarı 0.5 – 10 kütlə % olan nanokompozitlər üçün pilləli soyutma prosesində birinci növ faza keçidinin 88^{o}C, 20 kütlə% miqdarda isə bu prosesin 84^{o}C temperaturda getdiyi göstərilmişdir. 20-170^{o}C temperatur intervalında gedən kristallaşma prosesinin izahı və nəzəri əsaslandırılması verilmişdir. Dilatometrik tədqiqat üsulunun, qrafiki üsulla nanokompozitlərin tərkibindən asılı olaraq onların şüşələşmə temperaturunun müəyyən olunmasına imkan verdiyi tapılmışdır. Appretləşdirilmiş klinoptilolitin miqdarı artdıqca nanokompozitlərin şüşələşmə temperaturu qanunauyğun olaraq yüksəlmişdir. 5.0 kütlə % qədər appretləşdrilmiş klinoptilolitin maleinləşdirilmiş polietilenin tərkibinə daxil edilməsində kristallaşma və ya birinci növ faza keçidinin spor şəkilli kristallaşma mərkəzlərindən disk şəkilli kristallik quruluşların əmələ gəlməsi ilə getdiyi müəyyən olunmuşdur. 10-20 kütlə % appretləşdirilmiş klinoptilolit miqdarında sporşəkilli kristallaşma mərkəzlərindən mil şəkilli kristallik quruluşların əmələ gəlmişdir.

Açar sözlər: *nanokompozit, kristallaşma, klinoptilolit, malein anhidridi, aşağı sıxlıqlı polietilen*