

Temperature Capability of Polymer-Wax Composite Materials

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Abstract – Temperatures of phase transformations of polymer-paraffin compositions were investigated. Graphical dependences of temperature of the beginning of crystallization, temperature of the end of crystallization and phase hexagonal-rhombic H→R transformations in the solid state of oil food paraffin Π-1 on the content of polymer modifiers (low-pressure polyethylene, polyethylene waxes, atactic polypropylene and poly ethylene-covinil acetate) are constructed. It is shown that Π-1 crystallizes first in H and then in R-phase.

Keywords – food paraffin; polymers; composite materials; phase transition temperature.

I. INTRODUCTION

Diferent chemistry and physics researches of high-molecular compounds allowed to mark the 21st century as the century of polymeric materials [1-3]. Today it is difficult to overestimate the importance of polymers in our lives. The main reason for the rapid development of polymer chemistry is the modern technology and national economy need for new inexpensive and promising materials [4-8]. Thus, oil ceresines (microcrystalline wax) and waxes [9-10], polymers [11-13] and their copolymers with olefins [14-18], as well as their combination [19] are used as additives modifying the dispersed structure of solid oil paraffins.

The paraffin-wax composite materials given in the literature are used for a number of sectors of the national economy (shipbuilding, machine-tool, poultry, meat and dairy, etc.). These are important practical works, however, of a private nature. Taking into account the wide demand in various industries and the unique temperature and structural and mechanical properties of hard paraffin alloys with higher molecular weight products, we have studied polymer-paraffin

compositions in this paper. The effect of the introduction of additives of polymeric substances on the operational temperature parameters of the quality of paraffins is shown. Studied the thermal properties of the food compositions of the wax-polymer substances for the targeted selection of the component composition with desired thermal properties. Systematic generalization in this direction is carried out by the authors for the first time and is a scientific basis for the creation of various polymer-paraffin compositions with specified temperature properties.

II. METHODS AND MATERIALS

The formation of dispersed structures in polymer-paraffin melts at lower temperatures is a characteristic feature of the hardening system and is possible only at a certain concentration of the crystallized dispersed phase of polymers, sufficient to form a spatial grid of crystals, as well as in the absence of destructive mixing. The structure formation of paraffins at lower temperatures is accompanied by polymorphic transformations [19, 20]. The process of structure formation of paraffin-polymer alloys was considered in stages.

The first stage is the formation of crystal nuclei, which was determined by the temperature of the beginning of crystallization T_b . The second is the accumulation of the solid phase with simultaneous structure formation. The maximum temperature at which the formation of a spatial dispersion structure immobilizing the liquid dispersion medium occurs corresponds to the solidifying point (T_z). Solidifying point T_z or fusing point T_s was assessed according to loss of fluidity (in a simplified form on the ball of the thermometer). The third stage is the continuation of the structure formation from T_3 to the end of the crystallization of T_{cr} , when the entire liquid phase

passes into a solid state. The fourth stage is the modification transition of paraffin crystals from hexagonal H-crystal system (α -phase) to rhombic R ($T_{H \rightarrow R}$) or β -phase. This transition is more typical for pure n-alkanes and solid oil paraffins and often does not manifest itself in more complex mixtures. The fifth stage is the cooling of the crystalline rhombic R or β -phase to a predetermined cooling temperature (T_0). For comparison of polymer-paraffin alloys, their temperature capability were evaluated by a single characteristic –fusing point T_s ($T_s, K = t_s, C + 273$) (GOST 4255-75) or solidifying point T_z, K ($T_z, K = t_z, C + 273$) (GOST 2287-74).

The method of complex differential-thermal and dilatometric study. Temperature limit of state changes– the temperature of the beginning of crystallization (T_b), temperature of the end of crystallization (T_{cr}) and phase hexagonal-rhombic H \rightarrow R transformations in the solid state ($T_{H \rightarrow R}$) for the melts of paraffins and their mixtures were determined by thermographic and dilatometric methods on specially created laboratory facilities that provide programmed cooling of the system under study [20]. Providing a slow cooling rate of $0.01 \div 0.05$ deg/sec. allowed to bring the crystallization conditions to equilibrium [20]. The device for thermal (TA) and more advanced differential thermal analysis (DTA) included a block of programmed cooling of the sample and a unit for measuring its temperature; this allowed to automatically record the curves "temperature-time $T - \tau$ " and DTA. The curves $T - \tau$ (curve 1) and DTA (curve 2), as shown in Fig.1, determined the temperature limits of the phase transformations of T_b, T_{cr} и $T_{H \rightarrow R}$ of the investigated polymer-paraffin systems/.

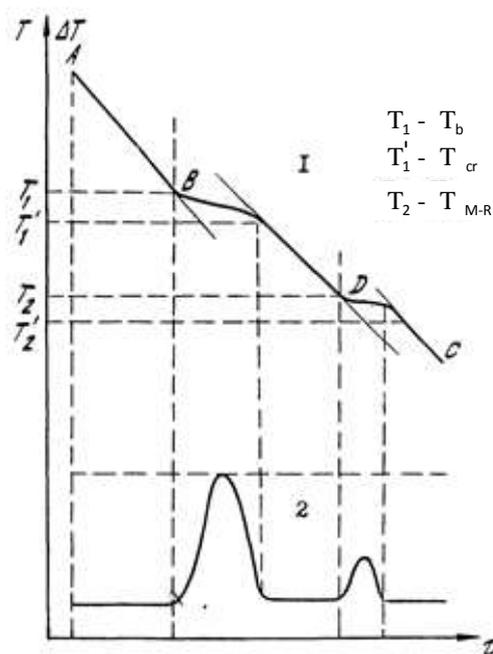


Fig. 1. Thermograms of thermal (1) and differential thermal (2) analysis

These methods favorably differ in higher accuracy and simplicity from ultrasonic and electric methods, providing mechanical and electromagnetic impact on the analyzed product and, thereby, affecting the crystallization process. The

results were refined by dilatometric and thermogravimetric methods. The dilatometric method is based on the specific property of paraffins to change the volume sharply at the temperatures of state changes. To develop this method, a study was conducted on the relationship between the temperatures of state changes with the contraction (volume shrinkage) and the thermal effects accompanying these transitions. The study of the process of phase transformations of paraffins during the cooling of their melts allowed us to establish that the temperature at which there is a sharp change in the value of the contraction almost completely coincides with the temperature of the beginning of crystallization, and in the modification phase transition – with the temperature of this transition. These conclusions were confirmed by differential thermal analysis. The discovered regularity was used for analytical control of phase transformation temperatures.

Combined on one diagram of Figure 2, the recording of thermograms ($T - \tau$) (curve 2) and contractionograms ($V - \tau$) (curve 1) allow a graphical way to determine the temperature intervals of phase transformations. On the curve ($V - \tau$), two areas of structure formation are clearly distinguished: the first $V1 - V1'$ – from the beginning of the formation of a new phase (temperature $T_1 = T_b$) to the formation of a spatial dispersed structure of crystals of hexagonal crystal system (temperature $T_1' = T_{kr}$) and the second $V2 - V2'$ – phase transition of the crystal hexagonal H-phase in the rhombic modification R (temperature limits $T_{H \rightarrow R}$ corresponding to T_2 and T_2'). To determine the temperature of the beginning of crystallization from the point of the dilatometric curve P corresponding to the moment of a sharp change in the volume, it is necessary to conduct a straight line parallel to the ordinate axis, before crossing the thermographic curve at the point B . Point B will characterize the temperature of the beginning of crystallization T_b , indicated on the curve T_1 . The temperatures $T_{cr} = T_1'$ and $T_{H \rightarrow R}$ are found in a similar way (Fig.2). Programmed cooling of the systems studied with the velocity of $0.01 \div 0.05$ deg/sec and automatic recording of curves "temperature-time $T - \tau$ ", differential thermal analysis (DTA), and contractiogram ($V - \tau$) allowed a graphical way to determine the temperature of phase transformation with the reproducibility of results of measurements not less ± 0.2 deg.

Comparative results of phase transformation temperature measurements using dilatometry and DTA paraffins were obtained for oil paraffins of Ozeksuat oil of the following grades: food P-1 ($T_s = 328.3K; T_{H \rightarrow R} = 309.1K$); B₂ ($T_s = 327.2 K; T_{H \rightarrow R} = 308.7 K$); T ($T_s = 326.1K; T_{H \rightarrow R} = 308.1K$) and synthetic H-C₂₄H₅₀ ($T_s = 323.7K; T_{H \rightarrow R} = 319.2K$). They confirmed the good convergence of measurements by these methods. At the same time, the reproducibility of the results of measuring the temperature of phase transformations by the dilatometric method, confirmed by differential thermal analysis, was not less than ± 0.2 deg. It should be noted that the temperature of the end of crystallization or initial melting point of paraffin, certain thermal and dilatometric methods practically coincide with temperatures of the onset of X-ray amorfy in these systems when they are heated, we have set Thermoradiographically. Thermoradiograph also allowed to confirm the reliability of the used dilatometric and DTA

methods in the temperature measurement $R \rightarrow H$ transitions of paraffins and their mixtures.

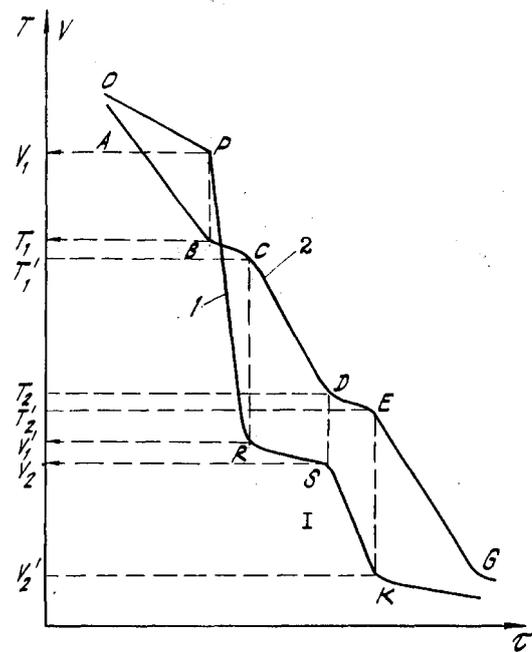


Fig. 2. Combined record dilatometric (1) and thermal (2) curves

Characteristics of the studied polymers. In this paper, the regularities and features of changes in the temperature capability of polymer-paraffin compositions depending on their composition are investigated. In the preparation of polymer-wax compositions were taken as a basis of food paraffin brand P-1 Ozeksuat oil, which floated the polymer components. Joint melting of paraffin P-1 with polymers was carried out at temperatures 20 degrees higher than the dissolution temperature of polymers in paraffin. For the purposeful selection of the component composition of polymer-paraffin compositions with desired properties, we have studied the change in the temperature capability of food paraffin PIP-1 depending on the concentration of polymers (C_n). In the preparation of binary mixtures with paraffin P-1, the following polymers were used: low-pressure polyethylene (HDP), polyethylene waxes (PV),

atactic polypropylene (APP) and ethylene - vinyl acetate copolymers (CMEA). Physical and chemical properties of the polymer systems used by us are presented in tables 1 – 3 with their characteristics.

Polyethylene waxes (PV) were obtained in two ways: by thermal degradation (PV – 25, PV – 100, PV-300, PV-800, PV-1000) and in the production of polyethylene (PE) at different plants (Grozny and Guryev chemical plants and in Germany). Their properties are given in table 1 and 2.

Poly ethylene-co-vinil acetate (TU N 6-05-1636-97), industrial name ultrathene (CMEA). – high-molecular polyolefins. Compared with polyethylene, they have a high elasticity, adhesion to different materials, heat capacity, solubility etc. Often, in practice, using CMEA with 10÷50% content of vinyl acetate (VA). We used alloys of paraffin P-1 and CMEA with VA(%): 11, 28 and 40-44 (table. 3). As the content of vinyl acetate increases, the dissolution temperatures of copolymers in various hydrocarbons decrease. VA-groups significantly reduce the crystallinity of the CMEA. When the content of VA 9÷14% SAV is similar to LDPE, but has greater elasticity and lower melting point.

Atactic polypropylene (APP) – a by-product of the polymerization of propylene and has stereoregular structure (irregular arrangement of the radical of the R-groups above and below the plane of the main chain). This amorphous substance has low strength and high adhesion properties. Used APP Tomsk production (TU 6-05-194-80) – granular. Softening temperature (TU 6-05-194-80) – 413 K, melting – 423 K, dropping (GOST 6793-74) – 433 K. Density – 850 kg/m³. The melt viscosity of APT at 453 K is 0.5÷5.0 PA·s. It is well combined with paraffin in the molten state.

Low-pressure polyethylene (HDP) is prepared by polymerization of ethylene ($-\text{CH}_2-\text{CH}_2-$)_n with special catalyst systems at a pressure of up to 20 atm. Linear macromolecules of HDP have a minimum of short side chains. The experiment used polyethylene base grade 273 Budennovsky chemical plant. The use of HDP is associated with its performance properties and technical characteristics: good compatibility with paraffin, density – 950 kg/m³, melting point 427 K, molar mass 4250 g/mole.

TABLE I. PHYSICAL AND CHEMICAL CAPABILITY OF POLYETHYLENE WAXES (PV) OBTAINED BY THERMAL DESTRUCTION

Indexes	PV - 25	PV - 100	PV-300	PV-800	PV-1000
Drop ping point, K	369	371	372	373	378
Mole weight, g/ mole	850	1500	2400	3500	4000
Dymanic consistency (413K η , Па·c)	0,028	0,10	0,32	0,75	1,50

TABLE II. PHYSICAL AND CHEMICAL PROPERTIES OF POLYETHYLENE WAXES (PV) OBTAINED DURING THE PRODUCTION OF POLYETHYLENE (PE)

Polyethylene waxes	Drop ping point, T, K	Mole weight, r/mole	Texture ρ^{293} , kg/m ³	Consistency 413K η , Па·c
Deutch PV E – 114	368	1010	880,0	0,060
Grozny chemical plant PV	373	1500	895,0	0,090
Guryev chemical plants PV	378	2600	930,0	0,450

TABLE III. PHYSICAL AND CHEMICAL CAPABILITY OF POLY ETHYLENE-CO-VINYL ACETATE

Indexes	VA content in copolymer, % by weight.			
	5÷7	9÷14	17÷22	24÷30
Texture (293K, kg/m ³)	930	934	944	950
Brittleness temperature, K	173	173	233	
Longitudinal strength, MПа	15–14	14–10	12–8	5–4
Extension coefficient, %	750	700	670	550

III. THE RESULTS OF AN EXPERIMENTAL STUDY OF STATE CHANGES TEMPERATURES OF POLYMER-WAX COMPOSITE MATERIALS

For a comparative assessment of the temperature parameters of the quality of polymer-paraffin alloys investigated of temperature of the beginning of crystallization (T_b), temperature of the end of crystallization (T_{cr}) and phase hexagonal-rhombic $H \rightarrow R$ transformations in the solid state ($T_{H \rightarrow R}$). The value of the solidifying point T_z almost coincided with the Zhukov's melting temperature T_s ($T_s = T_z$). The temperature dependences of state changes of paraffin Π -1 alloys with polyethylene waxes (PV-1000, P-800, PV-25) on their composition in the range from 0 to 100% were studied. Fig.3(a,b) show the dependence of the temperatures of crystallization (T_b , K) and solidification (T_z , K) of paraffin alloys P-1 with polymers HDP, PV-300, SAV (11% VA), SAV (28% VA), APP on the content of the latter (C_p). Fig. 4. as an example, are given the graphic dependence of the temperature of phase transformations (T, K) of polymer-paraffin alloys (a – PV-300; b – SAV (11% VA); b – APP) on the concentration of polymers (C_p). At the same time, real experimental points record phase transitions for paraffins in a mixture with polymers according to the data of complex Thermoradiographic and dilatometric analysis. To clarify polymorphic phase transformations of paraffin and polymers in the solid state, we used thermoradiographic phase analysis. The work was carried out at the Department of crystallography, Leningrad State University on the diffractometer DRON-1, DRON-2 and DRON-3, equipped with high temperature attachment KRV-1100. The results obtained (Fig. 3 and 4) showed that the investigated polymer modifiers dispersed structure of the paraffin N-1 are arranged in the following series in order of increasing solubility in the paraffin, HDP, polyethylene waxes, thermal degradation (MF-1000, F-800, DF-25), polyethylene waxes, polymer processing GHZ PV, PV-300), SAV from 11% VA, SAV with 28% VA, AMS, SAV with a 40% VA.

At the same time, modifiers that exceed the melting temperature of solid petroleum food paraffin P-1, increase, as expected, the melting temperature of the alloys. The greatest increase in temperatures T_k and T_3 was observed at 10% additives of the most high-melting polyethylene HDP (curves 1 in Fig. 3a, b). With increasing concentration of vinyl acetate groups (VA) in CMEA from 11 to 40 wt.% improved solubility of this copolymer in paraffin Π -1. This conclusion was confirmed by us in the study of its alloys with synthetic paraffin $H-C_{24}H_{50}$. Study of temperatures of phase transitions were carried out with alloys of synthetic tetracosane $H-C_{24}H_{50}$ with the Pv-300, SAV (11% VA), of the CMEA (28% VA) and CMEA (40-44% VA) and 25% wt. of these polymer additives. Studies have shown that fractional crystallization of the n-alkanes and polymers. The temperature phase transformations

in the solid state $T_{\alpha \rightarrow \beta}$ almost does not change if the structure melts tetracosane with polymers regardless of their content.

The difference between T_{kk} и T_k temperatures for PV-25, PV-800 and PV-1000 was 5.0, 2.0 and 1.0 deg. respectively. This indicates a spontaneous mass crystallization of higher molecular weight PV, accompanied by spatial dispersed structure formation of these systems. The most significant (2, 7 deg /%; 2, 8 deg /%; 3.9 deg /%) increase in T_k alloys of paraffin Π -1 with PV-25, PV-800 and PV-1000, respectively, occurs with the addition of PV to paraffin to 10% by weight. Starting with 30% alloys, they are the slowest (by 0.10 deg /% and proportionally almost the same for all PV increase T_k . The difference between T_{kk} и T_k PV 10% of their alloys with Π -1 was for PV-25, PV-800 and PV-1000 respectively 8, 5 and 4 deg. For alloys with 30% of all investigated PV and above to 100% difference between T_{kk} и T_k is 2 deg. Paraffin alloys with a 10% content of polymer modifiers are characterized by a greater value of the temperature range from the beginning of crystallization to solidification (6-10 deg) Fig. 3 (a-b).

In the case of additives PV -300, APP and SAV with 11% VA spatial structure formation (solidification) of these relatively few concentrated (10%) polymer-paraffin compositions occurs only at the crystallization temperature of the paraffin (Fig.4). Temperatures of the end of crystallization T_{kk} of such alloys and their softening T_p coincide with T_{kk} paraffin Π -I and are equal to $\approx 323K$ (Fig.4). Compared to paraffin-eresine mixtures [20], the mixture of paraffin with polymers have the more complex T_s dependence of the composition. Thus, with the increase in the content of a higher molecular weight high-melting component, the value of T_s of the mixture naturally increases, and the degree of increase T_s decreases. The greatest increase in the melting temperature occurs with the addition of high-melting HDP, and more effectively up to 1.0 wt.% (curves 1 in Fig. 3 a-b).

Phase transformations in the solid state. As shown by the dilatometric and DTA studies confirmed by thermoradiogram, alloys of paraffin with polymers when they are cooled characterised by the fractional crystallization of high-melting first polymer, $R(\beta)$, and then the wax in his characteristic at the respective temperatures, $H(\alpha)$, then $R(\beta)$ - crystal structure (Fig. 4). Solid paraffin crystallizes first in hexagonal $H(\alpha)$, and then in rhombic $R(\beta)$ -phase, polymers – only in $R(\beta)$ -crystal structure. The temperature of the modification phase $H \rightarrow R$ - transition of paraffin Π -I in its hard alloys with polymers, regardless of their quantitative ratio, remains almost the same 308 ÷ 310K, characteristic of the Π -I (curves 5 in Fig. 4).

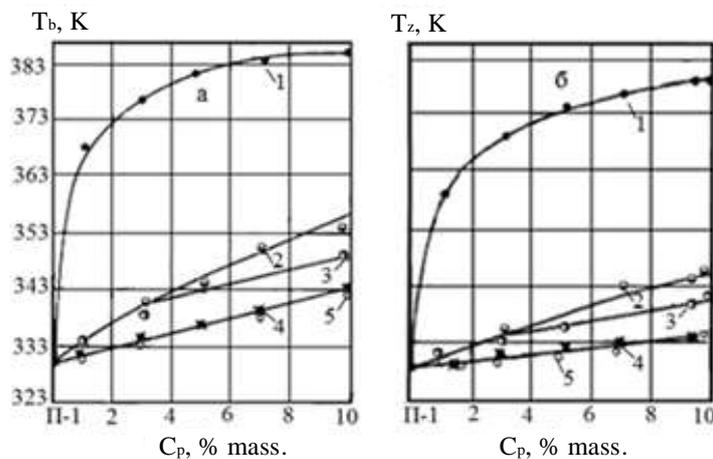


Fig. 3. The dependence of the beginning of crystallization temperature a (T_b , K) and solidification b (T_z , K) alloys of paraffin $\Pi-1$ with polymers (Π) from their content (C_n) 1 – HDP, 2 – PV-300, 3 – SEV (11% VA), 4 SAV (28% VA), 5 – AMS

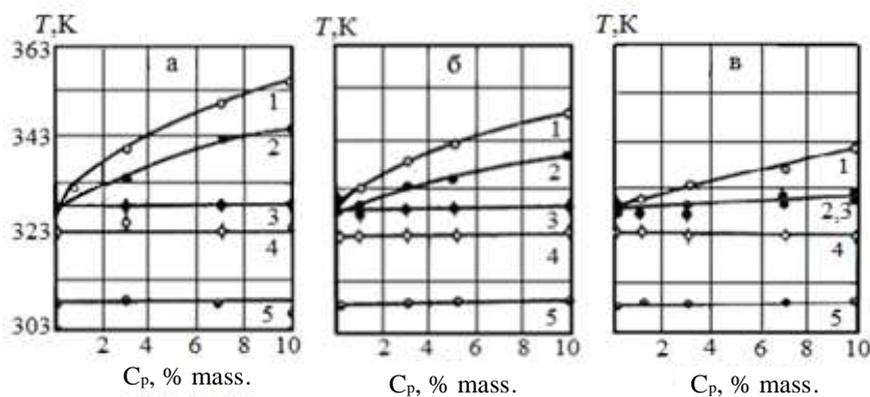


Fig. 4. Temperature dependence of phase transformations (T , K) of polymer-paraffin alloys from the concentration of polymers (C_n): a – PV-300; b – SAV (11 % VA); v – APP 1 – 1 – T the beginning of crystallization of the polymer, 2 – T solidification polymer-paraffin, 3 – T the beginning of crystallization of paraffin P-1, 4 – T the end of crystallization of $\Pi-1$, 5 – T ($\alpha \rightarrow \beta$) $H \rightarrow R$ – transformation P-1

Binary mixtures of paraffin (P-1) with polymers (P) in the solid state at room conditions (293K) are heterogeneous systems consisting of two crystalline phases in the R – crystal structure, respectively. In this case, a limited dissolution of the low-molecular part of the n-alkanes of polymers in paraffin is possible, which contributes to a small increase in the parameter C the paraffin crystal lattice modified by polymers. So in the alloys of P-1 + 20% PV (PV-25, PV-800 and PV-1000); P-I + 10% AMS; P-I + 10% SAV (28% VA) the hexagonal phase of modified paraffin is characterized by a small increase in T_b and T_{cr} compared to pure P-I.

It should be noted that 10% additives of PV-300 and CMEA (11 % VA) to paraffin $\Pi-1$ did not affect T_b and T_{cr} of pure $\Pi-1$ (curves 3 and 4 Fig. 4 a, b). During crystallization, $\Pi-1$ first forms an isomorphic hexagonal H -phase, and then with a decrease in temperature to 309 K, the paraffin crystals of $\Pi-1$ undergo a hexagonal-rhombic $H \rightarrow R$ transformation. The value of $T = 309\text{K}$ $H \rightarrow R$ -transformation of $\Pi-1$ remained practically unchanged during phase transformations of the investigated polymer-paraffin alloys with different concentrations of polymers (C_n).

The obtained data confirm the practical absence in the studied cases of the influence of polymers on the process of polymorphic hexagonal-rhombic transformation of paraffin $\Pi-1$. This is due to the fact that with a significant difference in the length of the chains of n-alkanes of binary polymer-paraffin mixtures, the condition of the densest packing of molecules is provided with their separate (fractional) crystallization. In this case, two independent phases of solid paraffin $\Pi-1$ and polymer compounds are formed. The results of the diffractometric study showed the presence of rhombic R-crystal structure of their crystals at room temperatures (293 K). During the heating process, the polymers did not pass into hexagonal modification, keeping rhombic until the beginning of melting.

Thus, the temperature conditions under which the polymer-paraffin compositions are located are the main factor of their polymorphic phase state. The comparative characteristic of temperatures of phase transformations of the investigated polymer-paraffin mixtures at their cooling to 293 K allows us to scientifically prove their purposeful choice for various sectors of the national economy.

IV. CONCLUSION

1. Graphical dependences of the temperatures of the beginning (T_b), end of crystallization (T_{cr}) and phase hexagonal-rhombic $H \rightarrow R$ transformations in the solid state ($T_{H \rightarrow R}$) on the composition of oil paraffin P-1 compositions with polymer modifiers are constructed. Conclusion and conclusions on the work are done.

2. The compounding of the paraffin wax P-1 with other high molecular weight products helps to increase the value of the melting temperature T_s . The degree of increase in T_s P-1 is associated with how much the added polymeric substances have this indicator T_s in magnitude higher than paraffin.

3. It is found that polymer-paraffin alloys are characterized by separate crystallization of first high-melting polymer and then low-melting paraffin component in their characteristic at these temperatures R - or H - crystal structure.

4. Solid paraffin Π -1 crystallizes first in H , and then in the R -phase. The value of $T_{H \rightarrow R}$ of paraffin P-1 309 K practically does not change when 10% of the mass is added to polymer system.

5. At 293 K the binary alloys of paraffin P-1 and polymer products are a highly heterogeneous system consisting of two phases of orthorhombic R -crystal structure.

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