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PREPARATION OF SYNTHETIC PITCH BY TWO STAGE PROCESSING OF COAL TAR



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The article proposes a method for producing synthetic pitch based on a two-stage processing of tar obtained from the semicoking of coal from the Shubarkol deposit. The first stage involved hydrogenation of coal tar followed by the separation of a fraction with a boiling point above 270 °C from the resulting hydrogenate. The research results showed that the phenol content in the hydrotreated fraction decreased from 23.73 to 4.67 g/L compared to the initial tar, while the benzene content was 37.6 g/L.

At the second stage, the hydrotreated fraction with a boiling point above 270 °C was subjected to thermal treatment to obtain synthetic pitch. The effect of thermal treatment pressure on the physicochemical properties of the resulting synthetic pitches was investigated using EPR spectroscopy. According to the EPR spectroscopy results, an increase in the concentration of free-radical states in the synthetic pitch is associated with an increased asphaltene content.

In addition, increasing the thermal treatment pressure from 2.5 to 3.5 MPa led to a decrease in the penetration value of the synthetic pitch from 91 to 31 mm, indicating an increased proportion of high-molecular-weight aromatic hydrocarbons in its composition. The resulting material, enriched with aromatic hydrocarbons, is promising for use in the production of carbon materials, electrode masses, and binders.

KEYWORDS: coal tar, hydrogenation, asphalt, coal, thermal treatment.

ПОЛУЧЕНИЕ СИНТЕТИЧЕСКОГО ПЕКА МЕТОДОМ ДВУХСТАДИЙНОЙ ПЕРЕРАБОТКИ КАМЕННОУГОЛЬНОЙ СМОЛЫ

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КАЗАХСКИЙ НАЦИОНАЛЬНЫЙ УНИВЕРСИТЕТ ИМЕНИ АЛЬ-ФАРАБИ
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В статье предложен способ получения синтетического пека на основе двухстадийной переработки смолы, полученной при полукоксовании угля месторождения Шубаркөл. Первая стадия процесса заключалась в гидрогенизации каменноугольной смолы и выделении из образовавшегося гидрогенизата фракции с температурой кипения выше 270 °С. По результатам исследований установлено, что содержание фенолов в составе гидрообработанной фракции снизилось с 23,73 до 4,67 г/л по сравнению с исходной смолой, тогда как содержание бензола составило 37,6 г/л.

На второй стадии гидрообработанная фракция с температурой кипения выше 270 °С подвергалась термической обработке с получением синтетического пека. Влияние давления процесса термической обработки на физико-химические показатели полученных синтетических пеков исследовано методом ЭПР-спектроскопии. Согласно результатам ЭПР-спектроскопии, повышение концентрации свободнорадикальных состояний в синтетическом пеке связано с увеличением содержания асфальтенов.

Кроме того, при повышении давления процесса термической обработки с 2,5 до 3,5 МПа значение пенетрации синтетического пека снизилось с 91 до 31 мм, что свидетельствует об увеличении доли высокомолекулярных ароматических углеводородов в его составе. Полученный материал, обогащённый ароматическими углеводородами, перспективен для применения в производстве углеродных материалов, электродных масс и связующих.

КЛЮЧЕВЫЕ СЛОВА: каменноугольная смола, гидрирование, асфальтены, уголь, термическая обработка

ТАСКӨМІР ШАЙЫРЫН ЕКІ САТЫЛЫ ӨҢДЕУ АРҚЫЛЫ СИНТЕТИКАЛЫҚ ПЕК АЛУ

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Мақалада Шубаркөл кен орны көмірін жартылай кокстеу арқылы алынған шайырды екі сатылы өңдеу негізінде синтетикалық пек алу тәсілі ұсынылды. Бірінші сатыда тас көмір шайыры алдымен гидрогенизацияланды, ал түзілген гидрогенизаттан қайнау температурасы 270 °С-тан жоғары гидроөңделген фракция бөлініп алынды. Зерттеу нәтижелері бойынша гидроөңделген фракция құрамындағы фенолдардың мөлшері бастапқы

шайырмен салыстырғанда 23,73 г/л-ден 4,67 г/л-ге дейін төмендеді, ал бензолдың мөлшері 37,6 г/л құрады.

Өңдеудің екінші сатысында қайнау температурасы 270 °С-тан жоғары гидроөңделген фракция термиялық өңделіп синтетикалық пек алынды. Алынған синтетикалық пектердің физика-химиялық көрсеткіштеріне термиялық өңдеу процесі қысымының әсері ЭПР-спектроскопия әдісімен зерттелді. ЭПР-спектроскопия нәтижелері бойынша синтетикалық пектегі бос радикалды күйлер концентрациясының жоғарылауы асфальтендер мөлшерінің артуымен байланысты.

Сондай-ақ термиялық өңдеу процесінің қысымын 2,5 МПа-дан 3,5 МПа-ға арттырғанда синтетикалық пектің пенетрация мәні 91 мм –ден 31 мм-ге төмендеді, бұл пек құрамында жоғары молекулалық ароматты көмірсутектер үлесінің артқанын дәлелдейді. Ароматты көмірсутектерге бай бұл материал көміртекті материалдар, электродтық масса-лар және байланыстырушы материалдар өндірісінде қолдануға тиімді болып табылады.

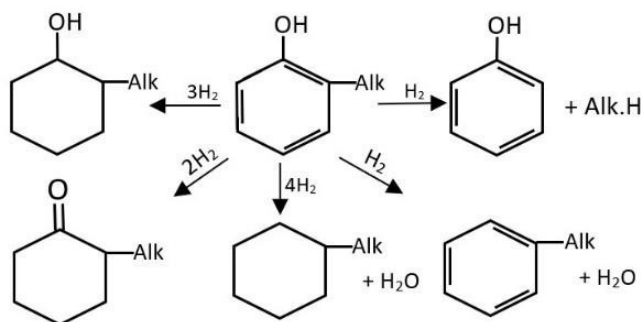
ТҮЙІН СӨЗДЕР: таскөмір шайыры, гидрогендеу, асфальтендер, көмір, термиялық өңдеу.

Introduction. Coal tar primarily consists of a blend of bicyclic and polycyclic aromatic hydrocarbons, alongside polycyclic structures incorporating heteroatoms within their rings. The sum of these substances is about 95 % of tar components. In addition, tar contains phenols, organic bases, a number of quinoline and acridine. Coal tar is a unique, unparalleled source of raw materials that currently meets more than 95% of global demand for condensed aromatic and heterocyclic compounds.

Scientific work was carried out for the purpose of quantitative and qualitative analysis of the component composition of coal tar. A detailed analytical study of its composition and chemical structure will be of great importance for its further processing. Using a combined method of planigraphy – gas chromatography/mass spectroscopy (GC/MS), the composition of coal tar was studied. The results show that the aliphatic compounds in the coal tar mainly included various paraffinic hydrocarbons C6-C28, alkenes and small amounts of alicyclic compounds. Aromatic groups mainly consist of monoaromatic compounds, polycyclic aromatic hydrocarbons (PAH-s) and some heterocycles containing N and S (PAN-H, PASH). The ester groups in the coal tar contain some oxygen compounds and several aromatic compounds with N and S. The main components of the polar groups in the coal tar were various phenols, methyl and dimethylphenols, naphthols and their derivatives. The analysis gives a satisfactory result, offering an acceptable theoretical basis for further processing and disposal of coal tar [1].

The source of formation of resinous asphaltene substances is aromatic hydrocarbons. The main components of the polar groups in the coal tar were various phenols, methyl and dimethylphenols, naphthols and their derivatives. The reduction of phenols into aromatic hydrocarbons has long been of interest from the point of view of the use of phenols of primary tar [2].

It has been shown that the hydrogenation of phenols can be carried out in several ways: 1) hydrogenation of the benzene ring with the preservation of the hydroxyl group; 2) hydrogenation to hydroaromatic ketones; 3) conversion of phenols to aromatic and alicyclic hydrocarbons; 4) hydrodealkylation of higher phenols with the formation of lower phenols:



Therefore, along with the study of ways to rationally use individual phenols, the efforts of researchers are also aimed at finding the possibility of converting individual fractions of primary tar into valuable chemical products without isolating individual components. For such use of phenols, their hydrogenation processing can be of great importance, allowing, depending on the process conditions, to obtain either phenol and cresols or hydrocarbons from higher phenols [1-4].

As shown in the mentioned works, phenol and its homologues were converted into aromatic hydrocarbons and their derivatives by the hydrogenation process. These aromatic hydrocarbons and their derivatives are the main source in the production of resinous asphaltene substances, which are the main structural component of raw materials.

During hydrogenation without preliminary dehydration and distillation, highly reactive tar compounds are actively stabilized by saturating them with both molecular and atomic hydrogen when transferred from a hydrogen donor, since these reactions occur at a lower temperature than the formation of atomic hydrogen from a molecular hydrogen. Several scientific works [5] have been carried out on the possibility of manufacturing raw materials for the production of various products by changing the chemical composition as a result of the process of hydrogenation of coal tar.

Hydrogenation technology is an important process for the chemical alteration of low-grade oils, such as coal tar. This study proposes a new systematic methodology for determining the characteristics of mixture flows for kinetic modeling of the coal tar hydrogenation process [6-7].

It is not possible to obtain pitch of the required quality by fractionation of coal tar, including pitch with an increased softening point. For these purposes, the use of highly aromatic tars is necessary. Among the available methods, thermal treatment under tar vapor pressure is the most important, as it primarily ensures an increase in the softening point to 85–90 °C and higher, as well as an increase in the content of the α_2 and β fractions. As a result, the aromaticity of the pitch increases. The optimal combination of the α_2 and β fractions is achieved at pitch softening points of 105–110 °C. Processing of the high-boiling anthracene fraction of coal tar into pitch binder appears to be a promising approach [8].

In this regard, based on the work done, it is currently possible to develop the direction of deep processing of coal tar and expand the possibilities of using products derived from it in new industries, increasing the role of coal tar as a unique raw material.

Materials and methods

Based on the results of these works, experiments were carried out on primary tar obtained by partial coking of coal from the Shubarkol deposit (Kazakhstan). The physical and chemical properties of coal tar are listed in *Table 1*. The oil from Kumkol deposit (Kazakhstan) with a boiling point above 270°C was used as H-donor of the distillation residue with the following characteristics: density at 20°C 0.8077 g/cm³; viscosity 9.69 mm²/sec; content, wt. %: paraffins 14.73; asphaltenes 1.52; tar 8.2; elemental composition, wt. %: C 83.85; H 11.27; S 1.81; N 0.80; O 2.27.

Experiment on the hydrogenation process

The process of hydrogenation of coal tar was carried out in a high-pressure reactor Parr Model 4848 (Parr Instrument Company, Moline, Illinois). The stirred reactor was heated to a temperature of 400°C using a digital controller Parr 4848 through a heater connected to the current source. When the effective content of Mo in the catalyst reached 0.05%, and the mixture of coal tar and H-donor in a ratio of 1:1 was brought to a temperature of 420°C, the stirrer was evenly enabled. Before launching, in order to establish the inert medium and the initial pressure (1 MPa), the technical argon collected in the cylinder was introduced into the reactor with pressure control in the gas reducer. The pressure in the system of 4 MPa was controlled by a pressure gauge.

Determination of softening temperature

Determination of the softening temperature of the samples was carried out in an automatic device Lintel ® KiSh-20. The range of automatic determination of sample softening temperatures is 20-200°C. Atmospheric pressure is 680-800 mm Hg. According to this method, the molten sample is poured into a standard copper ring and held until completely solidified. Then a steel ball of a certain size is placed on the surface of bitumen and placed on a special stand in a glass with water. The temperature at which the ball under the action of its own gravity will pass the sample layer in the ring and touch the control disk under the ring is taken as the softening temperature.

Determination of penetration

Determination of asphalt penetration was carried out by an automatic penetrometer for oil products MON-20B. Penetration range is 0 - 630 penetration units. Displacement meter error is ±0.05 mm. Penetration time is from 1 to 3599 sec. Delay time before penetration is from 0 to 3599 sec.

The molten sample is poured into a brass cup. After cooling, the cup with the sample is placed for 1 hour in a water bath, the temperature of which is 25°C. After 1 h, the cup with the sample is placed on the disc-table of the standard device - the penetrometer. The degree of hardness (penetration) is determined by the depth of penetration of the needle into the sample for 5 sec under the load of 50, 100 or 200 g. The penetration unit is the needle penetration depth of 0.1 mm.

Gas chromatography-mass spectral gas-liquid chromatography

Group hydrocarbon tar samples were determined on the "Crystal 50000.2" chromatograph. Flame ionization detector used for many organic compounds, Programmable evaporator (on-column). Capillary column, carbon dioxide cooling option for column oven (for ASTM D7169 method)

Result and discussion

Composed of elements boiling in the 65-500°C range, coal semi-coking tar is a viable feedstock for synthetic pitch manufacturing.

The properties of synthetic pitch are influenced by the nature of the raw materials. By appropriate selection of raw materials, materials of various properties can be obtained. The characteristics of the primary tar obtained by semi-coking of coal from the Shubarkol deposit are shown in *Table 1*.

Table 1 – Physico-chemical parameters of primary coal tar

Indicator	Parameter value
Density at 20°C, kg/m ³	1071
Elemental composition, wt. %:	
C	91.11
H	5.50
S	0.35
N	1.46
O (different)	1.58

As can be seen from *Table 1*, the sulfur content of the raw materials is 0.35 wt. %. During the hydrogenation of raw materials, sulfur in the tar composition acts as a sulfidizing agent.

Prior to the study, a gas chromatography-mass spectral analysis was performed to quantify the chemical composition of the primary tar. The quantitative content of the chemical composition of the primary tar is presented in *Table 2*.

Table 2 – Quantitative content of coal tar

Name of identified substances	Concentration of substances, g/l	Output time, min
Phenol	23.73	7.65
2-methylphenol	14.31	8.65
3-methylphenol	36.91	8.94
2,4-dimethylphenol	17.19	9.74
3-ethylphenol	23.77	9.95
4-methyl-1,2-dihydroxybenzene	5.30	10.81

As shown in the table, the proportion of phenol and its homologues in the chemical composition of coal tar is relatively high. The proportion of phenol is 23.73 g/l. The process of hydrogenation of coal tar was carried out in order to convert the phenols contained in the tar and their homologues into aromatic hydrocarbons and their homologues.

To achieve a lower sulfur content in the final pitch or coke, the most effective approach is to reduce the sulfur concentration in the feedstock used for coking. Although desulfurization of already finished products is an acceptable solution for materials with high sulfur content (4–7%), it should be taken into account that this process leads to a decrease in their density and a deterioration of mechanical strength [9]. Heavy hydrogenated coal tar is a valuable product with low density, characterized by a high hydrogen-to-carbon (H/C) ratio, minimal heteroatom content, and a negligible amount of metallic impurities. These properties determine its excellent processability and technological performance [10]. A moderate increase in temperature during hydrogenation is an effective method for removing heteroatoms, particularly sulfur. At a hydrogenation temperature of 390 °C, the aromatic compounds present in the feedstock become polarized due to the cleavage of molecular chains and subsequent polycondensation. The composition of the raw material has a decisive influence on the formation of the needle coke structure: in particular, a higher content of tricyclic and tetracyclic aromatic compounds in the refined feedstock directly contributes to achieving a higher degree of graphitization in the final needle coke product [11].

Hydrogenation of coal tar was carried out at a temperature of 420°C and a pressure of 4 MPa, with a catalyst of 0.05 wt.% MoS₂ and a hydrogenate was obtained. Hold up time – 1 hours. Since the largest amount of asphaltenes is concentrated in the fraction boiling above 270°C, and with prolonged exposure of these compounds, the decomposition of asphaltenes occurs with the formation of oils, as well as tars, carbenes and carboids, the conversion of phenols into aromatic and alicyclic hydrocarbons.

As can be expected, under these conditions, both destruction and structuring processes occur. During the hydrogenation process, the main changes occur with alkyl and naphthenic fragments of asphaltene structures. Hydrogenation of coal tar is carried out at 420°C and a pressure of 4 MPa, with 0.05 wt.% of catalyst MoS₂. The results of hydrogenation of coal tar are presented in *Table 3*.

Table 3 – Results of coal tar hydrogenation

Temperature, °C	>180 °C	180-250 °C	250-270 °C	Fraction above >270°C, %
Without catalyst, 420	2,6	11,0	18,4	50.0
420	2,8	26,0	21,0	68.0

The mass of the fraction obtained with the participation of the catalyst is greater than the mass of the fraction above >270°C obtained as a result of the coal tar hydrogenation process. That is, compared to the mass obtained without the participation of the catalyst, it is increased from 50.0 wt.% to 68.0 wt.%. The influence of the hydrogenation process on the chemical composition of the obtained fraction with a boiling point of over 270°C was studied.

To stabilize the highly reactive compounds of the primary tar, we carried out hydrogenation refinement without prior distillation in the presence of a hydrogen donor by saturating it with both molecular and atomic hydrogen when transferring it from a hydrogen donor, since these reactions occur at a lower temperature than the formation of atomic hydrogen from a molecular hydrogen. In the work, the residue of distillation with a boiling point above 270°C of oil from Kumkol deposit was used as a hydrogen donor.

This, in turn, reduces the coke formation process and increases the yield of the liquid product. For example, in the following work the coke formation passed through a minimum at 510°C. Among the diluents such as tetralin, cyclohexane, crude and pure benzene, and synthetic and low-temperature gasolines, decrease of coke yield was observed with tetralin and cyclohexane in the hydrocracking of tar [12].

This, in turn, reduces the viscosity of the resulting hydrogenate with the possibility of its further oxidation. That is, the viscosity of the mass obtained as a result of the oxidation process of the hydrogenate decreases. The results of comparison of the chemical composition of the fraction with a boiling point above 270°C with the chemical composition of primary coal tar are presented in *Table 4*.

Table 4 – Results of comparison of the chemical composition of the fraction with a boiling point above 270°C with the chemical composition of primary coal tar

Name of identified substances	Concentration of substances, g/l	
	coal tar	fraction above >270°C
phenol	23.73	4.67
2-methylphenol	14.31	1.2
3-methylphenol	36.91	3.5
2,4-dimethylphenol	17.19	0.65
3-ethylphenol	23.77	0.73
benzene	-	37.6

As can be seen from the table, the proportion of phenol in the fraction with a boiling point above 270°C decreased from 23.73 g/l to 4.67 g/l compared to primary coal tar, and the proportion of benzene – 37.6 g/l.

The enhanced proportion of aromatic hydrocarbons within coal tar's chemical composition, which are integral to the structural makeup of synthetic pitch, enables its future utilization as a source material for coke manufacturing.

Mesophase pitch is primarily formed through the process of thermal polycondensation, which can be conventionally divided into two stages. First, the raw material undergoes thermal decomposition: aromatic and cycloalkane compounds are dehydrogenated, and long aliphatic chains are cleaved, leading to the formation of numerous reactive radicals and low-molecular-weight species. Then, these reactive intermediates undergo free-radical polymerization, forming large polycyclic aromatic molecules. However, due to the complex composition of the raw material, these stages may occur simultaneously and interdependently [13].

Further, the thermal treatment of the fraction with a boiling point of over 270°C was carried out at a temperature of 500°C, 2,5-3,5 MPa, the physical and mechanical parameters of the pyrolyzed product were determined (*Table 5*).

As can be seen from the table, with increasing oxidation time, the penetration value decreases while the softening temperature increases. The penetration value of the light synthetic pitch is 91 mm, which is the highest value among the studied pitches, indicating

Table 5 – Physical and mechanical properties of synthetic pitch

Type	Temperature °C	Pressure, MPa	Penetration (25°C)	Softening Point	Density kg/m ³
Light synthetic pitch	500	2,5	91	80	2075
Heavy synthetic pitch	500	3,5	31	111	2093

its lower hardness. In contrast, the penetration value of the heavy synthetic pitch is 31 mm, reflecting its higher degree of condensation. The softening temperature of the light synthetic pitch is 80 °C, whereas the heavy synthetic pitch exhibits a higher softening temperature. The density of the heavy synthetic pitch is higher than that of the light synthetic pitch.

Pressure of the thermal treatment process significantly affected the softening temperature and penetration value of light and heavy synthetic pitch.

In continuation of studies on the production and forecasting for synthetic pitch, the effect of the pressure of the thermal treatment on the quality of the final product was studied using EPR spectroscopy (Table 6). The EPR spectral characteristics of the samples under investigation are detailed in Table 6 and Figure 1,2.

Table 6 – Characterization of EPR spectral features for the analyzed specimens

No.	Sample Name	Free radicals (spin/g)	EPR line width, erstedes	g-factor
2	Light weight synthetic pitch	$7.8 \cdot 10^{17}$	5.5	2.0031
3	Heavy synthetic pitch	$1.1 \cdot 10^{18}$	5.3	2.0030

It can be seen from the table that the width of the EPR line and the g-factor change little in these samples, the exception is heavy synthetic pitch, for which there is some decrease in the width of the EPR line and the value of the g-factor, clusters are possibly formed from a large number of condensed cyclic rings.

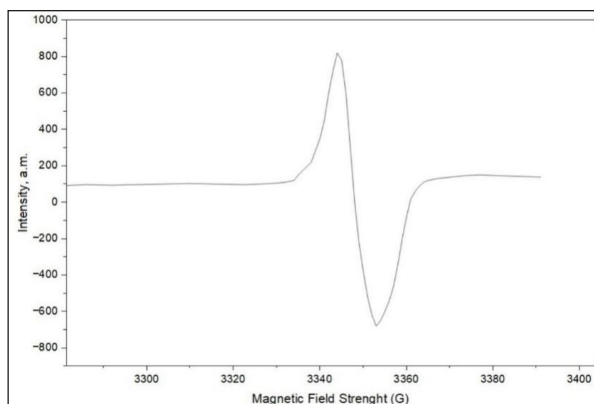


Figure 1 – EPR – spectra of light synthetic pitch (500 oC, 2,5 MPa)

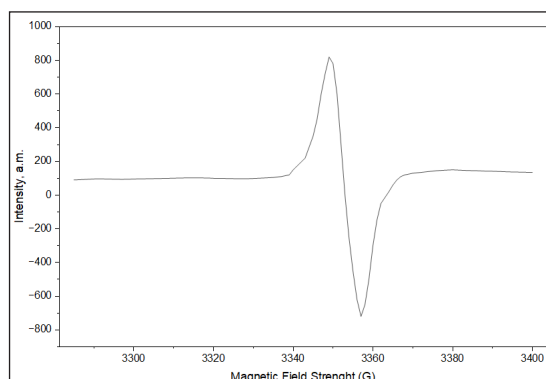


Figure 2 – EPR – spectra of heavy synthetic pitch (500 oC, 3,5 MPa)


The structure of the pitch is characterized by the presence of radicals linked by intermolecular dipole-dipole interactions [14]. The concentration of free radical states in heavy synthetic pitch is greater than in light synthetic pitch. This may be due to the formation of additional asphaltenes in synthetic pitch, which are mainly carriers of paramagnetism. The presence of a large number of paramagnetic centers is a distinctive feature of coal tar pitch [15]. This, in turn, clarifies the increase in softening point value from 80°C to 111°C. The parameters of the EPR spectra of the test samples prove changes in the penetration value of light synthetic pitch (80°C) and heavy synthetic pitch (111°C).

Conclusions

1. Due to the relatively high proportion of phenol and its homologues in the chemical composition of coal tar, a hydrogenation process was carried out in order to reduce the proportion of phenol and its homologues in the composition of coal tar. The reaction was performed at a temperature of 420°C and a pressure of 4 MPa, with a Mo-containing catalyst of 0.05 wt. %, a hydrogenate was obtained.

2. Using gas chromatography-mass spectral analysis, it was shown that an increase in the proportion of aromatic hydrocarbons involved in the formation of structural components of cementitious materials as a result of the hydrogenation process makes it possible to use it as a raw substance for the further production of road materials. As a result of hydrogenation, the proportion of phenol compared to primary coal tar decreased from 23.73 g/l to 4.67 g/l. The proportion of benzene was 37.6 g/l.

3. Light and heavy synthetic pitch was obtained by pyrolyzing the boiling fraction with a boiling point above 270°C at a temperature of 500°C, 2,5-3,5 MPa.

4. Changes in the physical and mechanical parameters of asphalt obtained by pyrolyzing a fraction with a boiling point above 270°C were analyzed by EPR spectroscopy. The softening point value of light synthetic pitch is 80, the softening point value of heavy synthetic pitch is 111. On the example of heavy synthetic pitch, a decrease in the width of the EPR line and the value of the g-factor possible leads to the formation of clusters from a large number of condensed cyclic rings and specifies that the softening point value is 111. 

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