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PRODUCTION OF DISTILLED BITUMEN FROM HIGH-VISCOSITY CRUDE OILS OF UKRAINIAN FIELDS

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Abstract. The characteristics of initial crudes of Yablunivske field (Poltava, Ukraine) and the properties of blended crudes have been examined to select the ways of their further processing. The crude oils were found to be heavy high-sulfuric oils without light distillates. The possibility of obtaining paving bitumen from blended crudes under study has been considered. Distilled bitumen obtained through distillation of this blend was found to meet the requirements for commercial paving bitumen 100/150. To improve the durability and resistance to aging of resulting bitumen, a polymeric modifier was added. The modifier amount of 3 wt.% was found to be appropriate to improve the operational characteristics of obtained bitumen to those of BMW 60/90 grade.

Keywords: high-viscosity crude oil, distilled bitumen, modifier, synthetic wax.

1. Introduction

Distilled bitumen is a soft, low-melting product, which is produced by concentrating petroleum residues *via* distilling them in vacuo in the presence of water vapor or inert gas. Production of distilled bitumen is widely used by foreign oil refining to obtain paving bitumen. For example, France produces 85 % of bitumen by vacuum concentration, USA – 50 %.[1-4]

Distilled bitumen is a fine colloidal system. Due to this structure, distilled bitumen is more ductile and has good adhesion characteristics, which contributes to increased hydrophobicity, which affects the water resistance. Increased water resistance in turn increases the durability of the pavement.[5]

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Characteristic features of distilled bitumen in contrast to oxidized ones are relatively high density, high hardness and tensile strength, as well as sensitivity to temperature changes. Moreover, the production of distilled bitumen is one of the most simple and economic technology.[6] One more advantage is environmental safety (as opposed to oxidized bitumen, which produces oxidizing gases that can be pollutants).

However, there are relatively few types of oil from which, as a result of distillation, it is possible to obtain commercial distilled bitumen. The careful selection of raw materials is very important in this technology because the bitumen properties depend upon the properties of the crude oil from which it is manufactured. The grade of bitumen depends on how much volatile material remains in the distilled bitumen –with more volatiles resulting in a less pure, more liquid product.

The crude oil or blends of crude oils can come from several sources, those that would be considered naturally occurring and those created or extracted from oil sands or shale.[7] Of the multitude of crude oils or blends commercially available, only a limited number are considered suitable for producing bitumen of the required quality in commercial quantities. In general, heavy (specific gravity >0.9) crude oils are used to produce bitumen of the required quality. These types of crude oils tend to contain high sulfur contents (>1 wt.%). The production of distilled bitumen is economically justified when the raw material contains a significant amount of asphaltene-resinous substances; the greater the ratio of asphaltene: resin, the better the properties and structure of bitumen. The best oils are naphthenic and naphthene-aromatic ones, *i.e.*, heavy oils with low paraffin content. Mainly, to obtain distilled bitumen high-resin asphalt-based oils with low paraffin content are used.[8]

More than 30 countries have recoverable heavy oil reserves. Four of the world's largest oil fields, the supergiants Al-Burqan in Kuwait, Kirkuk in Iraq, Abu Safah in Saudi Arabia, and the Bolivar Coastal field in Venezuela, contain and have produced very large amounts of heavy oil in addition to conventional oils. Other giant fields producing heavy oil include Zubair in Iraq; Duri in Indonesia; Gudao and Karamai in China; Seria in Brunei; Bacab, Chac, and Ebano-Panuco in Mexico; Belayim Land in Egypt; Maydan Mahzam in Qatar; and Uzen and Zhetybay in Kazakhstan.[9,10]

Approximately 2 % of the world's heavy oil reserves are concentrated in Ukraine, but due to the complexity of oil extraction, insufficient production of such oils, there are virtually no domestic publications on their study. A number of heavy oil deposits have been found in oil and gas condensate fields of the Dnieper-Donetsk basin. An example is one of the largest – Yablunivske gas condensate field, which is located in Poltava region, in the northwestern part of the axial zone of the Dnieper-Donetsk basin within the southern slope of the Zhdanov depression. Successful experience in the development of this field indicates the feasibility of "connecting" this additional source of hydrocarbons in the production of oil, gas and condensate. At present, the explored oil reserves of the Yablunovske field are about 50 million tons.[11]

According to existing data, the balance of the Ukrainian bitumen market in August 2021 amounted to 190 thousand tons, which is 24 % more than in the same period of the previous year (153.4 thousand tons).[12] The growth of the market was primarily facilitated by an increase in imports, the volume of which amounted to 149.1 thousand tons of road bitumen. Domestic production of bitumen in August decreased by 6.6%, to 41.3 thousand tons. Thus, the Ukrtatnafta plant produced 41 thousand tons, which is 4.7% less than in August last year. Ukrgasvydobuvannya produced 300 tons of the product, which corresponds to last year's August. According to forecasts of A-95 experts,[12] taking into account the announced parameters of financing the construction of 4.5 thousand km of roads with a volume of UAH 98 billion, the consumption of bitumen may grow to 1.5 million tons. From this standpoint and taking into account the existing potential sources, the development of domestic production of distilled bitumen is an urgent problem.

However, the properties of distilled bitumen do not meet the modern requirements for the quality of bitumen. The bituminous binder is expected to have the properties to ensure the durability of the asphalt mixture, the resistance of the asphalt mixture to permanent deformations at high temperatures (elastoplastic behavior, stiffness), and at low temperatures to sufficiently stiffness and relaxation properties to resist low temperature cracking (cohesion and tensile strength of the binder). The efficiency of bitumen production technology determines the need to create a system of regulation of physicochemical properties, which allows to obtain high-quality products of a wide range, regardless of the properties of the original oil mixture.[13]

In order to improve the qualitative properties of bitumen and/or asphalt mixtures, additives and modifiers are applied.[14] The addition of additives to bitumen affects the change in its physicochemical properties: softening point, brittleness, penetration, *etc*. Plasticizing additives affect the dispersion medium of the binder when filling it. The changes are noticeable in terms of increased penetration, decreased softening point and brittleness. When choosing modifying additives, priority is given to factors such as compatibility with bitumen, their acceptable boiling and melting points, as well as manufacturability, toxicological safety, availability, and low cost. The introduction of modifiers, in particular polymeric materials,[15-18] initiates the formation of a more complex colloidal structure of bitumen, in which polymer is highly homogenized. As a result, such rheological characteristics of bitumen as temperature sensitivity, adhesive properties, elastic and strength characteristics change, deformation under load are reduced, such bitumen is prone to restoring elasticity. One of the essential properties of modified bitumen is its stability during storage and operation, *i.e.*, the ability to retain its properties over time.[11,15-18]

So, various modifiers and additives are added to bitumen depending on its specifications.[19] Some of the most common additives are EVA, a polymer, which makes the mix more workable, especially in colder conditions; synthetic wax, that allows the bitumen to be mixed at lower temperatures; and SBS, a thermoplastic rubber which offers tensile strength and strain recovery.[20]

The organic additive (*e.g.*, Synthetic wax and Asphaltan-B), when added to the mixture, melts changing the bitumen temperature-viscosity curve and consequently, the mixing and the compacting can be done at a relatively low temperature. The organic additive should be chosen in a way in which its melting point would be higher than pavement service temperatures since they can alter binder grade, increase high-temperature grade and decrease low-temperature grade.[21] Chemical additives (*e.g.*, Cecabase, Evotherm-ET, DAT, 3G, Rediset), in turn, improve coating, mixture workability, and compaction.

Also, polycondensation resins (phenol-formaldehyde and phenol-cresol-formaldehyde resins),[22-26] low-molecular organic compounds (maleic anhydride)[27] or sulfur/organic copolymers[28] are used as bitumen binder modifiers.

Obtaining high-quality distilled (residual) bitumen for road construction is an urgent and important problem for all countries, especially in Ukraine, where mainly oxidized bitumen is produced. The search for new sources of raw materials for the production of distilled bitumen is a particularly important topic today. Production of distilled bitumen is economically justified in the case when the raw material contains a significant amount of asphaltenes and resins substances. However, the properties of distilled bitumen do not meet modern requirements for the quality of bitumen, so to improve its quality properties, modifiers are used.

Thus, the goal of this study was to obtain and modify distilled bitumen from the blend of high-viscosity crude oils of Yablunivske field (Poltava region, Ukraine), and to examine its physico-chemical properties.

2. Experimental

2.1. Materials

For the research crude oils from 4 wells of the Yablunivske field were blended (by 25 % from each well). Physicochemical properties of the initial crudes were determined according to the standard methods described by us previously.[29] The characteristics of the initial crudes are represented in Table 1.

Synthetic wax is a long-chain aliphatic hydrocarbon, which is synthesized *via* Fischer-Tropsch process. The melting point of synthetic wax is in the range of 358–388 K. It is completely soluble in bitumen at the temperatures above 388 K. Physical characteristics of the synthetic was is represented in Table 2.

2.2. Methods of Analysis

The blended crude was distilled to obtain gasoline fraction (313–473 K), diesel fuel (473–633 K) and residue (>633 K). The residue from distillation was analyzed for compliance with the requirements for distilled bitumen of grade 100/150.

Analysis methods used for bitumen characteristics are given in Table 3.

To determine the adhesion to gravel a fraction of 20/40 mm (LLC Novograd-Volyn Stone Crushing Plant, Ukraine) was used.

Table 1. Characteristics of initial crude oils

Properties	Units	Oil from well 1	Oil from well 2	Oil from well 3	Oil from well 4	Standard or Ref.
Density at 293 K	kg/m^3	978	981	973	955	ASTM D1298[30]
Kinematic viscosity at 313 K	mm^2/s	488	2010*	470	28.5**	EN ISO 3104[31]
Coking ability	wt %	9.8	10.2	7.5	6.4	[32]
Water content	vol %	5.1	24.6	5.6	5.4	ASTM D95-13[33]
Sulfur content	wt %	2.8	2.9	3.1	1.68	ASTM D4294[34]
Chlorides content	mg/dm^3	2810	5250	7120	1260	ASTM D3230[35]
Sulfuric resins content	vol %	17.2	23.6	15.1	22.8	ASTM D974[36]
Pour point without solvent	K	305	315	280	278	ASTM D97[37]
Pour point with solvent	K	254	259	260	249	ASTM D97[37]
Fractional composition	wt %					ASTM D2887[38], ASTM D5307[39] and ASTM D6352[40]
Distilled to 473 K		2.9	3.1	4.1	37.0	
Distilled to 633 K		22.0	23.0	21.0	62.0	
Distilled to 773 K		57.0	53.0	56.0	–	

Notes: * viscosity is determined at 323 K; ** indices for oil with diluent.

Table 2. Physical characteristics of synthetic wax

Properties	Units	Values and range	Standard or Ref.
Congealing temperature	K	373	ASTM D938[41]
Penetration at 298 K	0.1 mm	<1	ASTM D1321[42]
Penetration at 338 K	0.1 mm	7	ASTM D1321[42]
Brookfield viscosity at 408 K	cP	12	–
Odor	–	No odor	–
Visual color	–	Greyish-white to yellowish	–
Physical state	–	Pastilles and pills	–

Table 3. Methods of bitumen analysis

Properties	Units	Standard or Ref.
Penetration at 298 K	0.1 mm	EN 1426[43]
Softening point	K	EN 1427[44]
Ductility at 298 K	cm	DSTU 8825:2019[45]
Adhesion to gravel	points	[22]
Fraas breaking point	K	EN 12593[46]
Flash point	K	EN ISO 2592[47]
Resistance to hardening at 436 K (RTFOT method)	–	EN 12607-1[48]

Furthermore, the penetration index was determined according to the formula:

$$\text{Penetration index}=\frac{20\cdot SP+500\cdot \lg P_{25}-1952}{SP-50\cdot \lg P_{25}+120}$$

where SP – softening point (°C); and P_{25} – penetration at 25 °C (0.1 mm).

The plasticity interval was determined as the algebraic sum of the softening point and bitumen breaking point:

$$\text{Plasticity interval}=SP-F$$

where SP – softening point (K); and F – Fraas breaking point (K).

The group chemical composition of the obtained bitumen was determined by fractionation using chromatographic adsorption. It is a non-standardized method using silica gel of ASK grade as an adsorbent. The grain size was 0.2–0.6 mm.

800 g of previously dried (473 K, 6 h) silica gel were loaded into a column with a length of 2500 mm and a diameter of 35 mm. The bitumen : silica gel ratio was 1:8 (w/w). Bitumen sample was previously twice diluted with isooctane. The hydrocarbon fractions were sequentially washed with isooctane, isooctane-benzene mixture (4:1, 1:1, 1:9), and pure benzene. Resinous substances were desorbed from silica gel with the alcohol-benzene mixture (1:1). The separation process was carried out at elevated temperatures. After distilling off the solvent in a stream of nitrogen, the obtained eluents were mixed to obtain paraffin-naphthenic and aromatic groups of hydrocarbons. We adopted a scheme of hydrocarbons grouping, according to which bitumen was divided into paraffin-naphthenic, aromatic fractions and resinous substances. Paraffin-naphthenic fraction includes hydrocarbons with n^D_{20} < 1.49. The aromatic fractions were divided into groups: I group with n^d_{20} = 1.49–1.53; II group with n^d_{20} = 1.53–1.55; III group with n^d_{20} = 1.55–1.59 and IV group with n^d_{20} > 1.59.

3. Results and Discussion

The main physicochemical properties and operational characteristics of the sample received by the blending crudes of Yablunivske field are represented in Table 4.

Table 4. Physicochemical properties of the blended crude of Yablunivske field

Properties	Units	Value
Color	–	black
Coking ability	wt %	6.9
Density	kg/m^3	965
Water content	vol %	4.2
Sulfur content	wt %	2.4
Chlorides content	mg/dm^3	9600
Sulfuric resins content	wt %	16.9
Asphaltenes content	wt %	4.2
Pour point without solvent	K	291
Pour point with solvent	K	261
Kinematic viscosity at 313 K	mm^2/s	254.2
Fractional composition	wt %	
Distilled to 473 K		14.6
Distilled to 633 K		42.3

The experimental results show that this mixture of crudes belongs to the extra-heavy type of oils; its relative density is 965 kg/m^3. The heavier the oil, the heavier fractions in it, and the higher its viscosity. The kinematic viscosity of the mixture at 313 K is 254.2 mm^2/s. The content of asphaltenes and resins is 20.1 wt %. Thus, high-viscosity crude contains a large enough amount of bitumen-resin hydrocarbons, which makes the processing of this oil more difficult. The pour point of the solvent-free oil mixture is high, which indicates a high content of asphaltene-resinous hydrocarbons. Difficulties will arise in transporting such oils, especially during low temperatures, and additional measures will need to be taken to reduce the pour point. The sulfur content in the mixture is high (2.4 wt.%). So, the refining of blended crudes will require deep hydrogenation processes. In addition, the presence of sulfur can cause corrosion of equipment.[49] The water content in the oil mixture of the Yablunivske field is 4.2 vol %, which requires careful dehydration of crude oil with the use of special reagents – demulsifiers.[50]

A characteristic feature of the blended crudes is the low content of light fractions distilled to 633 K. The yield of the residue is about 57 wt %. Therefore, the entire economic component of this mixture refining will ultimately depend on the efficiency of the residue processing.

The distillation of the blended crudes results in obtaining a residue, which according to the preliminary estimates may correspond to the distillation (residual) paving bitumen of 100/150 brand. So, the main indices of its quality were determined, which are shown in Tables 5 and 6.

It is obvious that the obtained distillation (residual) bitumen meets all technical requirements. The penetration index, as an indicator of bitumen colloidal ability or the deviation of its state from pure viscosity of the resulting bitumen, is equal to 0, which is typical of distilled bitumen.[52]

In Ukraine, the problem of high-quality bitumen production is of special importance. Bitumen produced at Ukrainian refineries is mostly characterized by insufficient cohesion strength and adhesion, although generally, it meets the standard requirements. In addition, the increase in road traffic leads to a constant increase in the load on the road surface, which under certain conditions (including climate change during the year) leads to the occurrence of irreversible deformations in the road surface.[53]

According to the analysis of hydrocarbon composition (Table 6), the content of paraffin-naphthenic hydrocarbons is 19.8 wt % with a low yield of paraffin (3.6 wt %). The content of group I aromatics is 8.8 wt %, and there is also a relatively high content of heavy aromatic hydrocarbons (groups 3 and 4) and resins – 35.2 wt % and 23.6 wt %, respectively. These groups should further have a positive effect on the low-temperature properties of bitumen – brittleness and ductility – at 273 K. The total content of heavy aromatic hydrocarbons (groups 3 and 4) and resins in bitumen is 58.8 %.

Table 5. Main indices of obtained distilled bitumen for conformity with commercial paving bitumen 100/150

Properties	Units	Distilled bitumen produced from blended crudes of Yablunivske field	Standard requirements[51] for 100/150
Softening point (R&B)	K	315	312–320
Penetration at 298 K	0.1 mm	128	100–150
Ductility at 298 K	cm	157	–
Adhesion to gravel	points	2	–
Fraas breaking point	K	255	≤ 258
Flash point	K	504	≥ 503
Solubility in organic solvent	%	99.5	≥ 99.0
Plasticity interval	K	54	–
Penetration index	–	-0.36	from –1,5 to 0,7

Table 6. Hydrocarbon composition of obtained distilled bitumen 100/150

Hydrocarbons	Content in bitumen, wt %
Paraffin-naphthenic hydrocarbons (n_D^{20} <1.49), including paraffines	19.8 3.6
I group of aromatic hydrocarbons (n_D^{20} from 1.49 to 1.53)	8.8
II group of aromatic hydrocarbons (n_D^{20} from 1.53 to 1.55)	12.6
III group of aromatic hydrocarbons (n_D^{20} from 1.55 to 1.59)	35.2
IV group of aromatic hydrocarbons (n_D^{20} > 1.59)	
Resins	23.6

Various modifiers are added to the bitumen to improve its thermal sensitivity, brittleness and aging property.[54]

Synthetic wax modifier in the amount of 3 % was used to improve the physicochemical characteristics and aging property of bitumen obtained from blended crudes of the Yablunivske field.

Synthetic wax is a synthetic hard wax, which has been used successfully worldwide in asphalt road construction since 1997.[55] The effect of wax in bitumen depends greatly on its content. Synthetic wax content higher than 4% affects negatively the low-temperature properties of bitumen.[56] For this reason, content higher than 3% is not recommended.

The process was carried out at a temperature of 453 K for 3 h under constant stirring. It is under these conditions that synthetic wax is completely soluble in bitumen. Characteristics of modified bitumen 100/150 + 3 wt % synthetic wax are given in Table 7.

Table 7 shows that the introduction of 3 wt % of synthetic wax leads to the changes in the physicochemical properties of bitumen and now it corresponds to the standards required for bitumen of BMW 60/90 grade.[38] When adding the modifier, the softening point increased by 34.5 K compared to the original bitumen (from 314 to 348.5 K); the penetration decreased by 54 points (from 126 to 72). This indicates an increase in the hardness of the original bitumen. Ductility was reduced by more than 100 points. The penetration index increased to 2.51, which indicates the acquired high elasticity and ductility of bitumen, providing it with gel properties. It can be noted that the change in properties after heating the modified bitumen meets the technical requirements. There is also a significant increase in the adhesion of modified bitumen to the surface of gravel (crushed stone). Fig. shows that when adding 3 wt % of synthetic wax to paving bitumen100/150, its adhesion to gravel increases from 2 to 5 points.

Table 7. The main characteristics of the commercial bitumen and Synthetic wax modified bitumen

Properties	Units	100/150 + 3 wt % synthetic wax	Standard requirements[57] for BMW 60/90
Softening point (R&B)	K	348.5	> 331
Penetration at 298 K	0.1 mm	72	61–90
Ductility at 298 K	cm	57.5	> 24
Adhesion to gravel	points	5	–
Fraas breaking point	K	261	261-263
Flash point	K	505	–
Plasticity interval	K	88.5	–
Penetration index	–	2.51	–
Resistance to hardening at 436 K (RTFOT method):			
– change in mass after RTFOT	wt %	0.27	–
– ΔR&B	K	5.0	–
– residual penetration	%	62	–

Fig. Adhesion to gravel: 1 – 100/150 (2 points), 2 – BMW 60/90 (5 points).

4. Conclusions

From high-sulfur and high-resin blended crude oils of the Yablunivske field, it is possible to obtain distilled bitumen, because the oil mixture has a low content of light fractions boiling up to 633 K – 42.3 %. The yield of the residue is about 57 % by weight. The characteristics of the obtained distilled bitumen meet the requirements for distilled bitumen BD 100/150. The obtained bitumen contains a relatively low amount of naphthenic-paraffinic hydrocarbons (19.8 wt %), low content of paraffin (3.6 wt %), and high content of heavy aromatic hydrocarbons (groups 3 and 4) and resins – 58.8 wt %. This hydrocarbon composition provides sufficient adhesive properties of bitumen, ductility, and temperature resistance.

However, to improve the properties and durability of bitumen, it is advisable to add a modifier of synthetic wax in the amount of 3 wt %. This modifier improves the performance of bitumen, namely the softening temperature increased by 34.5 K, the penetration decreased by 54 · 0.1 mm, which, in turn, led to an increase in the penetration index (2.51). Therefore, we can conclude that the obtained modified bitumen has a greater hardness in comparison with the unmodified sample. It should be noted that this modified bitumen meets the requirements of regulatory documents for bitumen brand BMW 60/90. In addition, this modifier can be used as an adhesive additive.

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ОДЕРЖАННЯ ДИСТИЛЯЦІЙНИХ БІТУМІВ З ВИСОКОВ'ЯЗКИХ НАФТ УКРАЇНСЬКОГО РОДОВИЩА

Анотація. *Визначено характеристики вихідних нафт та суміші нафт Яблунівського родовища Полтавської області України, для вибору подальших шляхів переробки. Встановлено, що нафти є важкими з високим вмістом сірки, не містять світлих дистилятів, мають високу густину та в'язкість. В статті розглядається можливість одержання дорожніх бітумів з високов'язкої нафти. В результаті розгонки суміші нафт одержано дистиляційний бітум, який відповідає вимогам на бітум дорожній 100/150. Щоб покращити довговічність та стійкість до старіння даного бітуму до нього вводили полімерний модифікатор. Дослідження показали, що додавання модифікатора в кількості 3 % є доцільним та підвищує експлуатаційні характеристики бітуму до марки БМВ 60/90.*

Ключові слова: *нафта високов'язка, бітум дистиляційний, модифікатор, синтетичний віск.*