

ПАРАМЕТРЫ ПРОЦЕССОВ КРИСТАЛЛИЗАЦИИ И РАСТВОРЕНИЯ ТВЕРДЫХ УГЛЕВОДОРОДОВ НЕФТИ

Ю.П. Гуров, Е.О. Землянский, А.Г. Мозырев, С.Г. Агаев

Юрий Петрович Гуров*, Евгений Олегович Землянский, Андрей Геннадьевич Мозырев,
Славик Гамид оглы Агаев

Кафедра переработки нефти и газа, Тюменский индустриальный университет, ул. Володарского, 38,
Тюмень, Российская Федерация, 625000

E-mail: gurovjp@tyuiu.ru*, zemljanskije@tyuiu.ru, mozyrevag@tyuiu.ru, agaevsg@tyuiu.ru

В предлагаемой работе сопоставлены экспериментальные данные по процессам кристаллизации, а также рекристаллизации парафиновых углеводородов различной природы в углеводородных растворителях. Использовались технический парафин Т-1 (ГОСТ 23683-89) с температурой плавления 54 °C и церезин-80 (ГОСТ 2488-79) с температурой каплепадения 80 °C. В качестве углеводородных растворителей применялись керосин марки РТ-1 (ГОСТ 10227-86) и депарафинированное масло фракции 420-490 °C. Приводятся экспериментальные данные по процессам кристаллизации и рекристаллизации твердого парафина с температурой плавления 54 °C и церезина с температурой каплепадения 80 °C в керосине и депарафинированном масле. Для исследуемых систем определялись температуры начала кристаллизации (помутнения) t_n (ГОСТ 5066) и растворения t_p . Основное влияние на процессы кристаллизации и рекристаллизации оказывает химическое строение твердых углеводородов нефти. Обнаружено превышение температур растворения t_p твердых углеводородов над температурами начала их кристаллизации t_n , что объясняется гистерезисными процессами. Разница температур $\Delta t = t_p - t_n$ зависит от природы твердых углеводородов и их содержания в растворителях. Растворители на углеводородной основе оказывают существенное влияние на температуры начала кристаллизации. Растворы парафина в керосине имеют более высокие значения Δt относительно растворов церезина, что объясняется различием в химическом строении твердых углеводородов. С увеличением содержания твердых углеводородов в растворителях из-за различий в скорости диффузии твердых углеводородов Δt уменьшается. Обнаруженные закономерности по кристаллизации и рекристаллизации твердых углеводородов должны учитываться в процессах добычи парафинистых нефтей, их транспортировки и переработки.

Ключевые слова: парафины, твердые углеводороды нефти, процессы кристаллизации и растворения, гистерезис

PARAMETERS CRYSTALLIZATION PROCESSES AND SOLID PETROLEUM HYDROCARBONS DISSOLUTION

U.P. Gurov, E.O. Zemlianskii, A.G. Mozyrv, S.G. Agaev

Yuri P. Gurov*, Evgeny O. Zemlianskii, Andrey G. Mozyrev, Slavik G. Agaev

Department of Oil and Gas Refining, Industrial University of Tyumen, Volodarsky st., 38, Tyumen, 625000, Russia

E-mail: gurovjp@tyuiu.ru*, zemljanskije@tyuiu.ru, mozyrevag@tyuiu.ru, agaevsg@tyuiu.ru

In the proposed work, the experimental data on the processes of crystallization and different nature waxy hydrocarbons recrystallization in hydrocarbon solvents have been compared. Technical paraffin (GOST 23683-89) with the melting point of 54 °C and ceresin-80 (GOST 2488-79) with the dropping temperature of 80 °C have been used. PT-1 kerosene (GOST 10227-86) and de-

waxed oil of fraction 420-490 °C have been used as hydrocarbon solvents. The experimental data on crystallization and recrystallization processes of paraffin wax with a melting temperature of 54 °C and ceresin with a dropping temperature of 80 °C in kerosene and dewaxed oil are presented in this paper. It is shown that chemical structure has the main influence on the processes of crystallization and recrystallization of solid petroleum hydrocarbons. An exceedance of solid hydrocarbons solution temperatures t_p above their cloud points t_n has been observed which is explained by hysteretic processes. The temperature difference $\Delta t = t_p - t_n$ depends on the solid hydrocarbons nature and their content in solvents. Wax solutions in kerosene have higher values Δt relative to ceresin solutions in kerosene, which can be explained by the difference in chemical structure of solid hydrocarbons. With the increase in solid hydrocarbons content in their solvents due to the differences in solid hydrocarbons diffusion rate, Δt decreases. The discovered regularities of solid hydrocarbons crystallization and recrystallization should be taken into account in the processes of paraffin oil production, transportation and processing.

Key words: paraffin wax, solid petroleum hydrocarbons, dissolution and crystallization processes, hysteresis

Для цитирования:

Гуров Ю.П., Землянский Е.О., Мозырев А.Г., Агаев С.Г. Параметры процессов кристаллизации и растворения твердых углеводородов нефти. *Изв. вузов. Химия и хим. технология*. 2020. Т. 63. Вып. 6. С. 90–94

For citation:

Gurov U.P., Zemlianskii E.O., Mozyrv A.G., Agaev S.G. Parameters crystallization processes and solid petroleum hydrocarbons dissolution. *Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.* [Russ. J. Chem. & Chem. Tech.]. 2020. V. 63. N. 6. P. 90–94

INTRODUCTION

Solid hydrocarbons crystallization processes and their structure formation complicate waxy oils production, gathering and transportation. Waxing tubing pipes leads to a reduction in the flow rate of wells. Waxing field pipes and pumping equipment leads to their breakdown and mechanical destruction. Pumping of waxy oils is accompanied by waxing trunk pipelines, which leads to an increase in hydraulic resistance in them. For such pipelines, the problems of re-start after their temporary stop arise [1-5].

The processes of low-temperature dewaxing and de-oiling of petroleum products based on waxy hydrocarbons crystallization of wax are also accompanied by many problems, including waxing regenerative crystallizers and difficulties in filtering solid hydrocarbon suspensions on vacuum filters [6, 7]. Solid waxy hydrocarbons determine the performance properties of oils and diesel fuels. The occurrence of C₁₄₋₂₆ n-paraffin hydrocarbons in diesel fuels complicates their operation at low temperatures: the cloud point (the beginning of crystallization), the cold filter plugging point and chilling point increase [8, 9]. The problems of oil production, gathering, transportation and processing and the use of petroleum products determined by the occurrence of solid hydrocarbons in them should be solved taking into account their chemical composition, as well as the processes of their crystallization [4, 10].

EXPERIMENTAL PART

In the proposed work, the experimental data on the processes of crystallization and different nature waxy hydrocarbons recrystallization from hydrocarbon solvents have been compared. T-1 technical paraffin (GOST 23683-89) with the melting point of 54 °C and ceresin-80 (GOST 2488-79) with the dropping temperature of 80 °C have been used. PT-1 kerosene (GOST 10227-86) and de-waxed oil of fraction 420-490 °C have been used as hydrocarbon solvents. The choice of kerosene and de-waxed oil was defined by the fact that they do not contain any n-alkane hydrocarbons affecting the crystallization processes of relatively high-melting waxes. The used de-waxed oil and kerosene differ mainly in viscosity: the viscosity of kerosene at 20 °C is 1.25 mm²/s (own data), the viscosity of the de-waxed oil is 190.1 mm²/s [11].

The study of the processes of solid hydrocarbons crystallization and recrystallization was carried out on their model systems in kerosene. The solid hydrocarbon content (C_{SH}) in kerosene was 5, 10 and 20%. For the systems under study, the cloud point t_n (GOST 5066) and the solution temperature t_p were defined. There is no commonly accepted method for determining the temperatures of solid hydrocarbons solution from oil dispersed systems.

The method of determining the cloud point was adopted as a basis for the method of determining the solution temperature. Conventional thermostatic

test tubes provided with plastic plugs and mechanical stirrers were used. The stirrers excluded the influence of local overheating and supercooling on the processes of crystallization and dissolution of solid hydrocarbons in kerosene. The solid hydrocarbons crystallization processes depend on the rate of their cooling [12] and the content of solid hydrocarbons in the solvent [13]. And the processes of recrystallization depend on the rate of heating. The rate of temperature variation in the tubes was provided with the help of the Lauda RE-107 thermostat and amounted 40, 60 and 120 °C/h. The thermostat provided temperature control with an accuracy of ±0.1 °C. The temperature in the tubes was recorded with thermometers TH-8.

The cloud points and solution temperatures were determined sequentially in several stages. Initially, within 15 min, the model mixtures of solid hydrocarbons in kerosene were subjected to heat treatment at 90 °C, which provided complete dissolution of solid hydrocarbons. Then, the temperature in the thermostat was decreased to 10 °C with a given cooling rate. At clouding of the studied systems, the cloud point t_n was recorded visually, for more precise definition of which lighting was used. For a formation of stable reproducible spatial colloidal structures, the model systems were kept in tubes at a temperature of 10 °C for 15 min. Further, to determine the solution temperatures, the temperature in the thermostat was risen to 90 °C with heating rates of 40, 60 or 120 °C/h. The temperature of complete solution of the solid t_p was also recorded visually using lighting. The determination of cloud points and solution temperatures was repeated 3-4 times. The average values of t_n and t_p were calculated according to parallel experiments (Table).

The analysis of the table data shows that the cloud points t_n and solution temperatures t_p depend on the nature of solid hydrocarbons, their content in kerosene C_{SH} (% wt.), cooling and heating rates, as well as the direction of the process (crystallization and recrystallization).

The discrepancy between the temperatures t_n and t_p is conveniently assessed by their difference $\Delta t = t_p - t_n$. In general, the solution temperatures in almost all cases exceed the cloud points: the value of Δt depends on all the factors above. Paraffin T-1 solutions have slightly higher values of Δt relative to ceresin solutions in kerosene. With increasing C_{SH} in kerosene Δt decrease. The maximum values of Δt , ranging from 6.0 to 7.5, are observed in 5% solutions. Δt also decreases, with rare exception (ceresin content 5% by weight., cooling rate 120 °C/h), with the growth of cooling rates and heating of solid hydrocar-

bon models in kerosene. The more pronounced effect of crystallization-recrystallization parameters on paraffin models relative to ceresin models in kerosene (see Table) may be due to the difference in the chemical structure of solid hydrocarbons. T-1 paraffin consists mainly of *n*-alkanes, and ceresin – mainly of paraffin-naphthalene-aromatic hydrocarbons [6]. Pure paraffin hydrocarbons have a higher degree of crystallinity, which is the cause of the observed differences. A higher melting point (dropping point) of ceresin compared to T-1 paraffin is a secondary factor.

Table
Crystallization and recrystallization indicators of solid hydrocarbons in kerosene
Таблица. Показатели кристаллизации и рекристаллизации твердых углеводородов в керосине

Solid	Solid content in kerosene C_{SH} , % wt.	Cloud points (°C) and solution temperatures of model systems	Rate, °C/h		
			40	60	20
Paraffin	5.0	t_n	16.0	16.0	18.0
		t_p	23.5	23.5	24.0
		$t_p - t_n$	7.5	7.5	6.0
	10.0	t_n	23.7	23.6	24.5
		t_p	27.4	27.4	24.5
		$t_p - t_n$	3.7	3.6	0.0
	20.0	t_n	30.0	29.5	29.5
		t_p	32.4	32.4	30.0
		$t_p - t_n$	2.4	2.9	0.5
Ceresin	5.0	t_n	50.6	50.6	50.5
		t_p	57.0	57.0	58.0
		$t_p - t_n$	6.4	6.4	7.5
	10.0	t_n	61.3	61.3	62.3
		t_p	62.0	61.9	62.0
		$t_p - t_n$	0.7	0.6	-0.3
	20.0	t_n	64.8	64.8	64.8
		t_p	65.6	65.7	64.0
		$t_p - t_n$	0.8	0.9	-0.8

Note: t_n – cloud point, t_p – solution temperature

Примечание: t_n – начало кристаллизации, t_p – температура растворения

RESULTS AND DISCUSSIONS

According to the authors, the main reason for the detected regularities in the processes of crystallization and recrystallization of solid oil hydrocarbons is hysteresis processes.

Solid hydrocarbons crystallization processes depend not only on their nature (see table), but also on the nature of the dispersion medium [10]. The effect of the dispersion medium nature has been studied on the example of T-1 paraffin and ceresin in PT-1 kerosene and de-waxed oil (Figure).

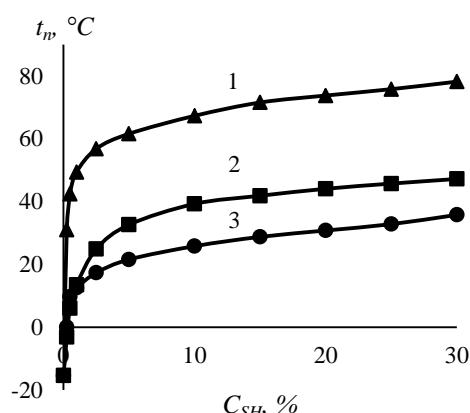


Fig. Relationships of solid hydrocarbons cloud points t_n and their content C_{SH} in de-waxed oil and kerosene: 1 – ceresin in oil, 2 – wax in oil, 3 – wax in kerosene

Рис. Соотношения начала кристаллизации твердых углеводородов t_n и их содержания в депарафинизированном масле и керосине: 1 – церезин в масле, 2 – воск в масле, 3 – воск в керосине

The relationships of solid hydrocarbons cloud points and their content in solvent $t_n = f(C_{SH})$ are presented. The common character of the paraffin and ceresin crystallization curves does not depend on the dispersion medium. The cloud points of the solid hydrocarbons solutions in oil (see curves 1, 2 in the fig-

ure) increase with an increase in melting temperature (dropping) of solid hydrocarbons, i.e. with the transition from paraffin to ceresin.

The cloud points of paraffin solutions (see curves 2, 3 in the figure) increase with an increase in the disperse medium viscosity, i.e. with the transition from paraffin-in-kerosene systems to the paraffin-in-oil systems. In the area of spontaneous (quick) crystallization, (paraffin content in solvent is over 5% of weight) the cloud points of paraffin in oil are 11-13 °C higher than the temperatures of crystallization of paraffin in kerosene.

Thus, it has been shown that the chemical composition and crystal structure of solid oil hydrocarbons have a major effect on the processes of their crystallization and recrystallization. An exceedance of solid hydrocarbons solution temperatures above their cloud points has been observed which is explained by hysteretic processes. The hydrocarbon-based solvents have a significant effect on cloud points. The discovered regularities in solid hydrocarbons crystallization and recrystallization should be taken into account in the processes of paraffin oil production, transportation and processing.

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Поступила в редакцию 12.12.2019
Принята к опубликованию 23.04.2020

Received 12.12.2019

Accepted 23.04.2020