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Heavy coal tar viscosity from the point of view of quinoline insoluble particle size distribution

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Abstract

Features of the operation of coke chambers in the post-repair period, carried out in order to extend the service life of coke oven batteries, are the main reason for obtaining coal tars with a high degree of pyrolysis for some coking plants. The high content of substances insoluble in quinoline, the increased viscosity of the dispersion medium lead to a growth in the coal tar water content. This causes not only difficulties in the realization of such a product, but also extreme inconvenience in carrying out operations for dehydration, deashing and even simple transportation. A study was made of the features of the particle size distribution of the solid dispersed phase of coal tars of three samples with the approximately same content of substances insoluble in quinoline (30-34%) and water (19-23%). In parallel, the tars rheological characteristics were studied using a Brookfield DV2T rotational viscometer with a thermocell at varying temperature and shear rate.

An increased degree of pyrolysis of coal tar causes a high viscosity, the dependence of which on temperature is described by a power law: $\eta = A/t^n$. The presence of a dispersed phase with different characteristics in the studied coal tars is the reason for the nonlinearity of the dependence $d\eta/dt = f(\eta)$. For tar samples with large aggregates of dispersed substances, the transition of the system to an unstructured state requires a significantly lower amount of viscous flow activation energy than for tar with smaller aggregates.

Comparison of the values of the coal tar dynamic viscosity with the particle size distribution of the dispersed phase showed that the tar sample, which contains the smallest aggregates of substances insoluble in quinoline, had the highest viscosity. At the same time, small aggregates, having a high specific interfacial surface, when the dispersion medium is diluted with a polar solvent, facilitate the coalescence of water droplets and initiate partial dehydration. In addition, mixing tars with a heterogeneous granulometric composition of solid dispersed substances reduces the degree of structurization and association of aggregates, which also leads to the release of water.

Keywords: *High pyrolysis coal tar; Dynamic viscosity; Quinoline insoluble; Particle size distribution.*

1. Introduction

In the Ukrainian consumer market, coal tar is mainly in demand as a raw material for producing pitch, therefore, the following basic requirements are imposed on it: density is not higher than 1190-1200 kg/m^3, the content of insoluble substances in toluene and quinoline, respectively, is not more than 8-12% and 3-6% wt. According to pre-existing regulatory requirements the dynamic viscosity of such a tar, is 30-40 cP (80°C), which ensures its normal pumpability, as well as dehydration and deashing. An increase in the coal tar viscosity significantly complicates the above technological processes, moreover, temperature drop as a result of heaters failure in the tar settling tanks, can cause the threat of stopping operation of spraying the gas collector of the coke oven battery.

Deterioration of the coke oven conditions, a special coal charge regime of the repaired coking chambers in some cases makes it necessary to reduce the completeness of loading to 0.80-0.90 from the design value. Overheating of the primary tar vapors in the underloaded chamber is higher than the temperature of the coke oven free space by tens of degrees Celsius, and it greatly accelerates the reactions of vapor-phase pyrolysis with the production of quinoline insoluble dispersed particles. The resulting tar has a high density (> 1250-1270 kg/m^3) and a high content of substances insoluble in quinoline (> 15-20%). Such viscous product is very difficult to settle from water and the resulting watered tar almost loses fluidity and the ability to be pumped by centrifugal pumps at low temperatures. To select the technological mode of preparing the tar for its processing, it is necessary to know the physicochemical, rheological and structural-mechanical properties of the liquid.

The viscosity of hard pyrolyzed tar is caused by the increase in its molecular weight and the content of dispersed particles in it. The coal tar dispersed phase consists of water droplets, aromatic substances with reduced solubility, supramolecular structures, products of dust particle entrainment from the coke chamber, highly condensed formations (substances insoluble in quinoline). An increase in the degree of pyrolysis of coal tar is accompanied by an increase in the viscosity of the dispersion medium, this leads to a deterioration in the conditions for the separation of dispersed particles (water and solid suspensions). In turn, this causes even greater increase in the viscosity of emulsions and suspensions, respectively. In order to ensure the mobility and subsequent dehydration and deashing of such tars, it is necessary to find out the reasons of increased viscosity and ways to reduce it.

The rheological properties of coal tar are interesting from the point of view of its preparation for processing and the regime of tar fractionation to obtain oils, pitch, or use in road construction. In addition, the study of the viscosity properties of the tar is of great informative value. Coal tar is a complex system both in composition and structure, so the rheology of the tar is a representation of the multicomponent system energy state.

During normal operation of coke oven batteries that have not reached a critical level of accumulated depreciation, the viscosity of coal tar with a low pyrolysis degree usually provides effective dehydration by the gravitational force and, naturally, does not cause difficulties in pumping. Reducing the viscosity of such a tar, or more precisely, the disaggregation of supramolecular structures by introducing additives, was of interest as a way to improve the efficiency of tar distillation [1].

The relevance of such studies remains high, especially in connection with the need to obtain pitches free from the content of substances insoluble in quinoline. To reduce the viscosity of coal tar, wash oil and mixed solvents are used, however, tars with a content of insoluble in quinoline not higher than 2.5% are used for this [2]. Such systems have a low viscosity and are classified as low-pyrolyzed tars, which does not allow the data obtained to be extended to high-viscosity systems with a high content of the dispersed phase.

Increasing the viscosity of the tar leads to a degradation in the deashing process, since, similarly to oil systems, a high viscosity favors to the stability of the suspension [3]. In this case, the viscosity of the resulting suspension increases due to the concentration of dispersed particles and their possible aggregation; similar processes occur with an increase in the content of asphaltenes and their aggregates in high-viscosity oils [4].

In addition, the tar high viscosity promotes the formation of stable water-in-tar emulsions, which is true for oils with increased viscosity. An elastic layer is formed around the droplets due to intermolecular interaction between resins and asphaltenes [4], and a coarse emulsion has a lower viscosity than a finely distributed one [5].

In turn, the emulsion viscosity growths significantly with an increase in the volume concentration of the dispersed phase [6]. Thus, high-viscosity systems, regardless of their origin, have a complex flow pattern associated with the stability of disperse systems, and the concentration of asphaltenes in oil, starting from 1% wt. is sufficient to change the nature of the flow from a Newtonian fluid to a non-Newtonian one [7]. Different filling of the free volume in the dispersion medium with the dispersed phase determines the rheological behavior of coal tar and can serve as a measure of its structuring.

The above data show that dispersed systems in complex highly viscous liquids based on oil and coal tars have a non-Newtonian flow pattern, and the granulometric composition also affects their viscosity. As for coal tar pitches, it has been established that with an equal content of substances insoluble in quinoline, systems with a higher degree of dispersion were more viscous [8]. The influence of factors on the viscosity of coal tars with a high degree of pyrolysis is less studied, it is of practical and scientific interest to establish the influence of the characteristics of the dispersed phase on the fluidity of the tars and the stabilization of emulsions.

An important task also remains to find ways to reduce viscosity by changing the characteristics of the dispersed phase due, for example, to tar blending. Similar solutions were used to improve the rheological properties of polymers [9].

2. Experimental

Coal tars obtained in the high-temperature coking process for the production of blast-furnace coke were selected and investigated. Samples were taken after intermediate mechanized clarifiers before the second settling stage. Due to the fact that individual ovens of coke batteries were not fully loaded after repairs, the degree of pyrolysis of the tar was very high.

As a result of serial sampling, three tars from three coke oven batteries were obtained, which were comparable in terms of the content of the solid and aqueous dispersed phases. Table 1 shows the measurement results of the main indicators of the tars quality. Indicators were determined by standardized methods for the coal tar analysis in accordance with TU U 19.1-00190443-100: 2016.

Table 1. Characteristics of testing tars

Indexes		R1	R2	R3
Density at 20°C, water-free coal tar, kg/m^3		1279	1300	1354
Mass fraction of water, %		23	21	19
Mass fraction of substances, water-free coal tar, %:	insoluble in toluene (TI)	33.7	32.8	35.3
	insoluble in quinoline (QI)	31.3	30.0	34.0
	ash substances	0.25	0.17	0.18

The type of emulsions obtained and the granulometric composition of QI substances were determined on a "Neofot" microscope under reflected light. Samples for research were prepared from filtered particles when determining the QI index. Granulometric composition of QI was also determined on a high-precision scanning optical microscope Jeol JSM 840. Digital photos of samples were obtained and processed using a ToupTech device with ToupView software, the number of particles for counting was N=600-800 pcs. The rheological characteristics of the studied tars were determined on a Brookfield DV2T rotational viscometer with a thermocell while changing the thermostating temperature and shear rate.

On the basis of extremely high values of substances insoluble in quinoline and toluene that is several times higher than regulatory values, obtained coal tar samples had sure signs of hard coking volatile products pyrolysis. Such a high-water content in the sample is also not at all typical for process tars with a low degree of pyrolysis.

3. Results and discussion

The results of dynamic viscosity measuring of samples at different temperatures are shown in Fig. 1.

The viscosity of the R3 resin sample is significantly higher than the viscosity of the other samples especially at low temperatures, which is important in the practice of pumping under cold ambient condition. In general, viscosity and the susceptibility of one to temperature also depends on the chemical composition of hydrocarbons and the mechanism of intermolecular interaction. The number of carbon atoms in molecules, the branching of the molecule, and the presence of polar functional groups determine the temperature dependence factor of a complex multicomponent system. It is difficult for coal tar sample to measure the native viscosity of a dispersion medium; in the general case, the viscosity of hydrocarbons increases with an increase in molecular weight (or boiling point), for polycyclic aromatic hydrocarbons, the viscosity increases with a growth in the number of cycles in a molecule. Thus, the viscosity of tars with a higher density and degree of pyrolysis increases.

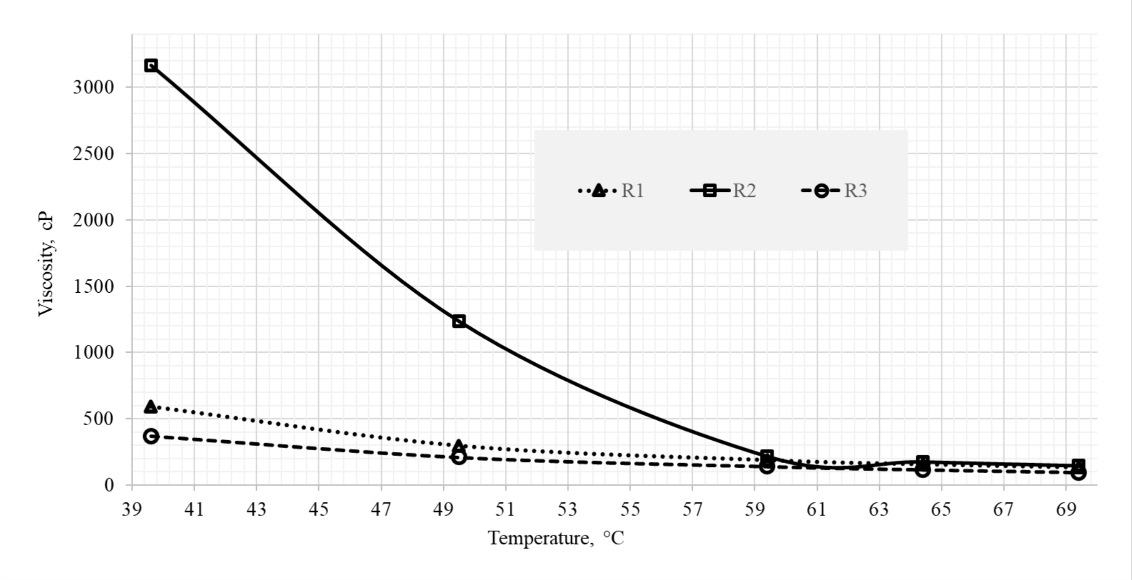

Fig. 1. Dependence of the viscosity of tar samples with approximately the same content of substances insoluble in quinoline and approximately the same water content

As already discussed above, the presence of a dispersed phase, namely water droplets and particles insoluble in quinoline, is also the reason for increase the coal tar viscosity. For comparison, tar viscosity with a QI content of 8.0 % did not exceed 95 cP under comparable conditions [1]. The contribution of the distributed water droplets to the tar viscosity for a temperature of 40 °C can be estimated using the existing exponential dependence [10]:

$\eta_e = \eta_t \times e^{4,58\varphi}$ (1),

η_e - the viscosity of the emulsion (cP) with a volume fraction of water, φ; η_t - the viscosity of water free tar, cP.

According to the given data, in the case under consideration, the watered tar, compared with the water free one, increases its viscosity by 2.3–2.9 times (40°C).

If we consider the studied tars as suspensions according to the Einstein formula [11], the bulk concentration of the suspension filler increases its viscosity (μ_s):

$\mu_s=\mu_l(1+2,5\varphi)$ (2);

μ_l – viscosity of continuous phase.

Equation (2) is right for undiluted systems with filler content (φ) less than 10%. For the concerned case for coal tar with such a high content of substances insoluble in quinoline 30-34%, the formula is unsuitable. There are data for coal tar pitch on the dependence of the viscosity of coal tar pitch on the content of insoluble in toluene [12], however, such a dependence for the coal tar cannot be used due to the expression of the viscosity of the continuous phase of the pitch in terms of the softening temperature.

As follows from Fig. 1, each tar sample obeys its own dependence of dynamic viscosity on temperature, so difference in viscosity could not be explained in concept of concentration of dispersed particles of emulsion (or suspension). All presented dependences can be approximated with sufficient accuracy in the studied temperature range by power-law dependences in the form:

$\eta = A/t^n$ (3),

The processing results are presented in (Table 2).

Table 2. Power dependence of tar samples viscosities on temperature

Indicators	Tar sample		
	R1	R2	R3
Determination coefficient of equation (3), R^2	0.995	0.980	0.999
Constant A	9.0 $\times 10^6$	1.0 $\times 10^{13}$	2.0 $\times 10^6$
Exponent n	2.63	5.95	2.37
at a temperature of 60-70°C			
R^2	0.9998	0.9996	0.9991
A	1.0 $\times 10^6$	6.0×10^6	3.0×10^6
n	2.16	2.50	2.44

A comparison of the presented power dependences shows that the samples have a comparable susceptibility of viscosity to temperature in the field of 60-70°C. As the temperature decreases, sample R2 shows an increase in the exponent *n* at variable *t*, which indicates a significant increase in the temperature sensitivity of the tar viscosity.

The dependence of the derivative dη/dt on the viscosity η obtained as a result of differentiation of equation (3) with respect to temperature is shown graphically in Fig. 2.

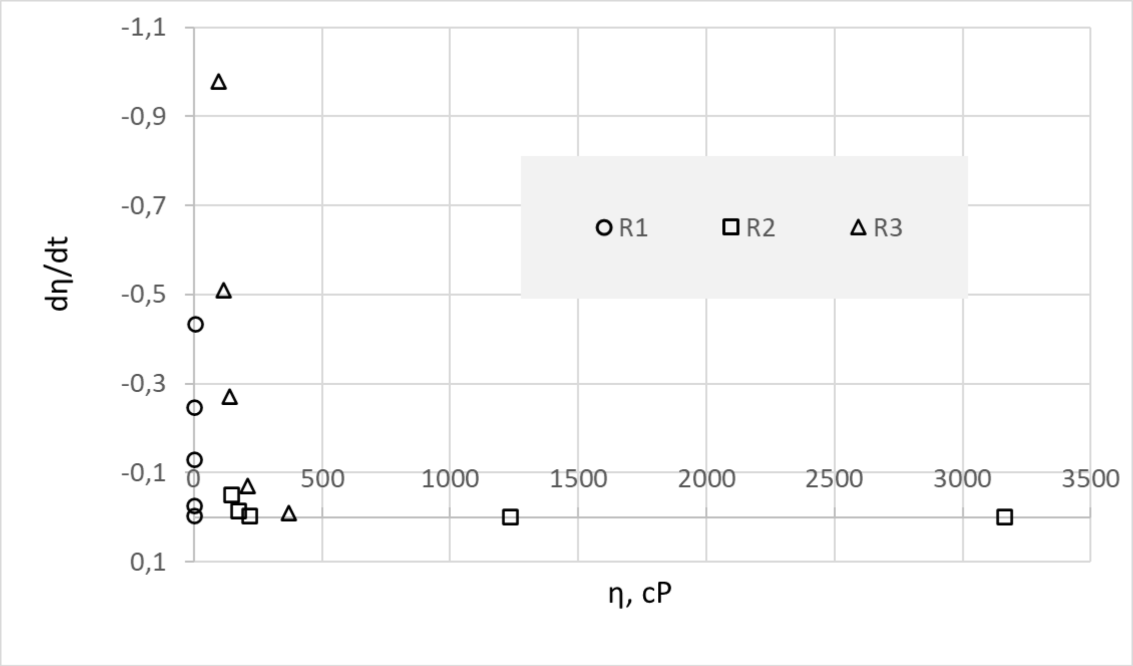

Fig. 2. Dependence of the temperature derivative dη/dt on viscosity

It is believed [13] that the calculated points on the graph should lie on one straight line if the dependence of viscosity on temperature is of a thermal fluctuation nature, that is, the fluid flow is due to the breaking of bonds between flow units with increasing temperature.

Analyzing the dependences obtained, it can be seen that the points for samples R1 and R3 lie on a straight line with a determination coefficient of 0.54 and 0.58, and the points for sample R2 only with a coefficient of 0.25. Therefore, the dependence of viscosity on temperature, especially for sample R2, is of a different nature than the rheology of mineral oils [13]. In contrast to them, the tars under study contain a dispersed phase: water and particles insoluble in toluene and quinoline. But, since the content of water and insoluble particles of the same order, apparently, the presence of a dispersed phase with different characteristics in the studied tars is the reason for the nonlinearity of the dependence dη/dt =f(η).

It is possible to evaluate the nature of the tar flow during their transition point from an unstructured to a structured system by determining the temperature of the break point of the rectilinear dependence lgη=f(10^3/T). Fig. 3 shows the dependence of the tar viscosity on temperature in the corresponding coordinates, and in Table 3 – inflection temperatures of the obtained functions.

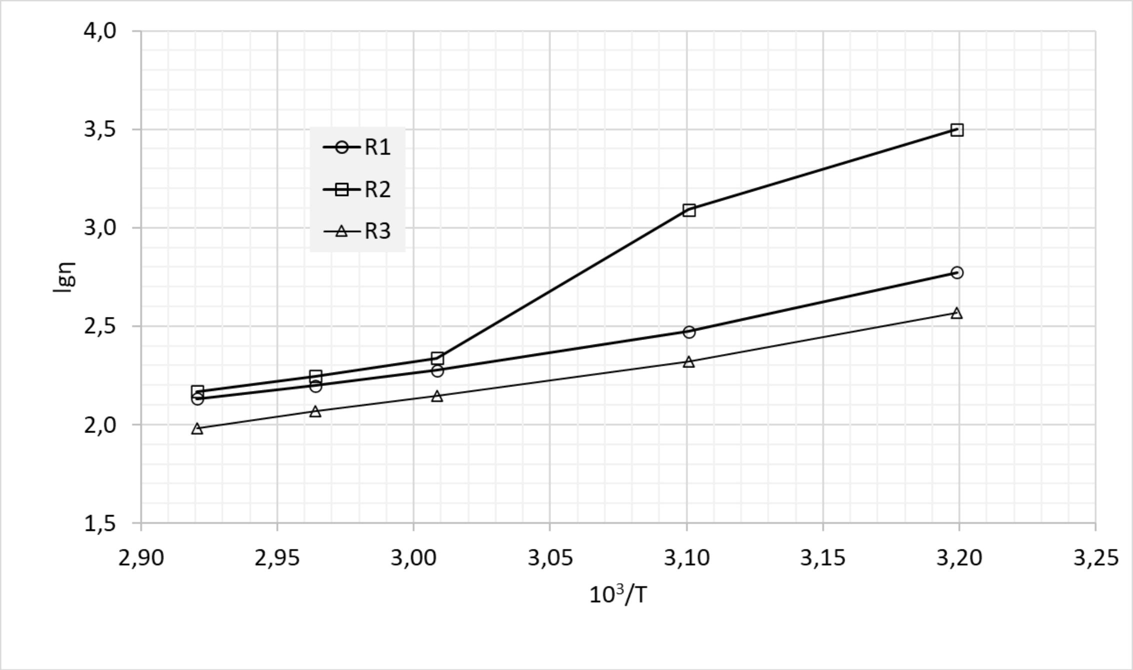

Fig. 3. The dependence of the tar viscosity on temperature in the coordinates of the Arrhenius equation lgη=f(10^3/T) to determine the break point

Table 3. Characteristics of the breakpoints of the function lgη=f(10^3/T)

Tar sample	Transition temperature of a structured system into an unstructured one, °C	
R1	49.5	-
R2	59.4	49.5
R3	49.5	-

The largest amount of break is observed twice for the R2 tar sample, and the R1 and R3 tars have a slight transition at the same temperature. The presence of breaks in the linear dependence lgη=f(10^3/T) is explained by a change in the activation energy of the viscous flow, presumably as a result of structural changes associated with the weakening of bonds between dispersed particles in the tars. At the same time, the energy of such bonds was much higher for the R2 sample, which is confirmed by the results of calculating the activation energy (Table 4), which was determined from the transformed Frenkel-Eyring equation [14]:

$$E=R\times\ln(\eta_1/\eta_2)/(1/T_1 - 1/T_2) \quad (4)$$

Table 4. The value of the activation energy of the viscous flow (kJ/mol) at the corresponding break points

Temperature range, °C	Tar sample		
	R1	R2	R3
45-55		108.0	
55-65	31.4	112.0	28.3

As can be seen from the data presented in Table. 4, the transition of the system to the unstructured state requires significantly less energy for tar samples R1 and R3, although the content of the dispersed phase in the three tar samples is comparable. So, the nature of the dispersed phase had a decisive influence on the viscosity of the studied tars, as was previously established for coal tar pitches [12].

Characteristics of the particle size distribution of solid dispersed particles insoluble in quinoline, obtained on an optical microscope "Neofot" at a magnification 1×250 times, are presented in Table 5, and in Fig. 4.

Table 5. The results of counting of particles insoluble in quinoline at a magnification of 250 times

Indicators	Tar sample		
	R1	R2	R3
Minimum diameter, μm	1	1	2
Maximum diameter, μm	166	100	470
Number average diameter, D_N, μm, $D_N=\Sigma(N\times d)/\Sigma N$	12.2	4.1	15.8
Standard deviation	20.7	6.02	27.7
Average volume diameter, μm, $D_V = \Sigma(N\times d^4)/\Sigma(N\times d^3)$	135.15	82.05	449.02
Polydispersity index: $P_D = D_V/D_N$	11.1	20.07	28.44
Specific interfacial surface, μm^{-1}, $Ss = 6/D_N$	0.5	1.5	0.4

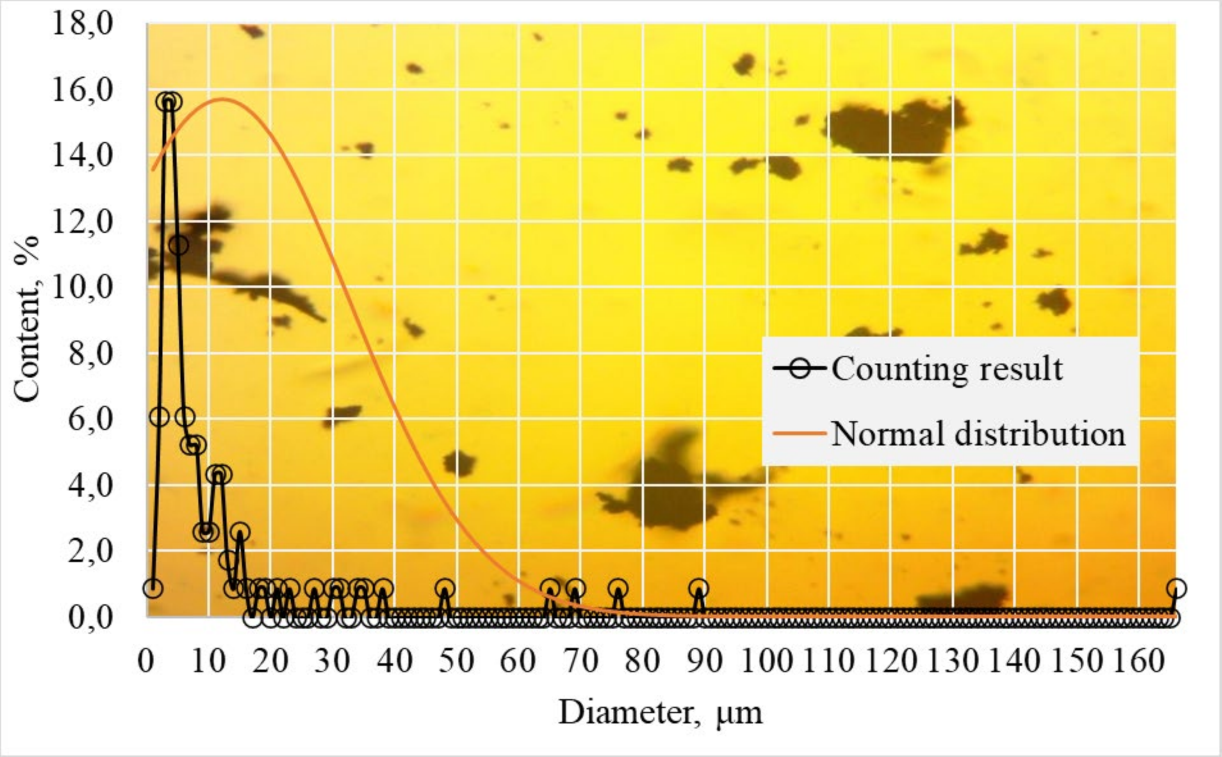

Fig. 4. Photos of quinoline insoluble particles and size distribution curves, sample R1-R3, magnification 1×250

As follows from the above images, the data obtained relate to the aggregates of dispersed particles. The granulometric composition of the single particles, insoluble in quinoline, was determined from the photographs (Fig. 5). Table 6 shows the results of processing the counting of particle sizes, and in Fig. 5 - characteristic images of samples.

The results show that the investigated tars with 30-34% QI content have single particles of approximately the same average diameter (0.44-0.57 μm). However, these dispersed particles form aggregates and it's dimension correlate with the nature of the flow of the studied tar samples. The aggregation of particles, in all possibility, occurs with an increase in their concentration due to an intensification of the pyrolysis processes of tar vapors in the under-roof space of the coke oven chamber. The formed aggregates, which belong mainly to fine (up to 20 μm) systems, affect the rheological behavior of the tar. By analogy with coal tar pitches [8], the granulometric composition of the sample of dispersed solid particles of tar R2 is 80% represented by classes of 1-5 μm, which differs significantly from samples R1 and R3 (2-13 and 3-17 μm, respectively).

Table 6. The results of counting of particles insoluble in quinoline, using an electron microscope with magnification 1×5000

	Tar sample		
	R1	R2	R3
Minimum diameter, μm	0.2	0.2	0.2
Maximum diameter, μm	1.3	2.3	1.5
Number average diameter, D_N, μm, $D_N=\Sigma(N\times d)/\Sigma N$	0.44	0.57	0.57
Standard deviation	0.18	0.21	0.19
Average volume diameter, μm, $D_V = \Sigma(N\times d^4)/\Sigma(N\times d^3)$	0.71	0.99	0.78
Polydispersity index: $P_D = D_V/D_N$	1.62	1.75	1.36
Specific interfacial surface, μm^{-1}, $Ss = 6/D_N$	13.6	10.6	10.5

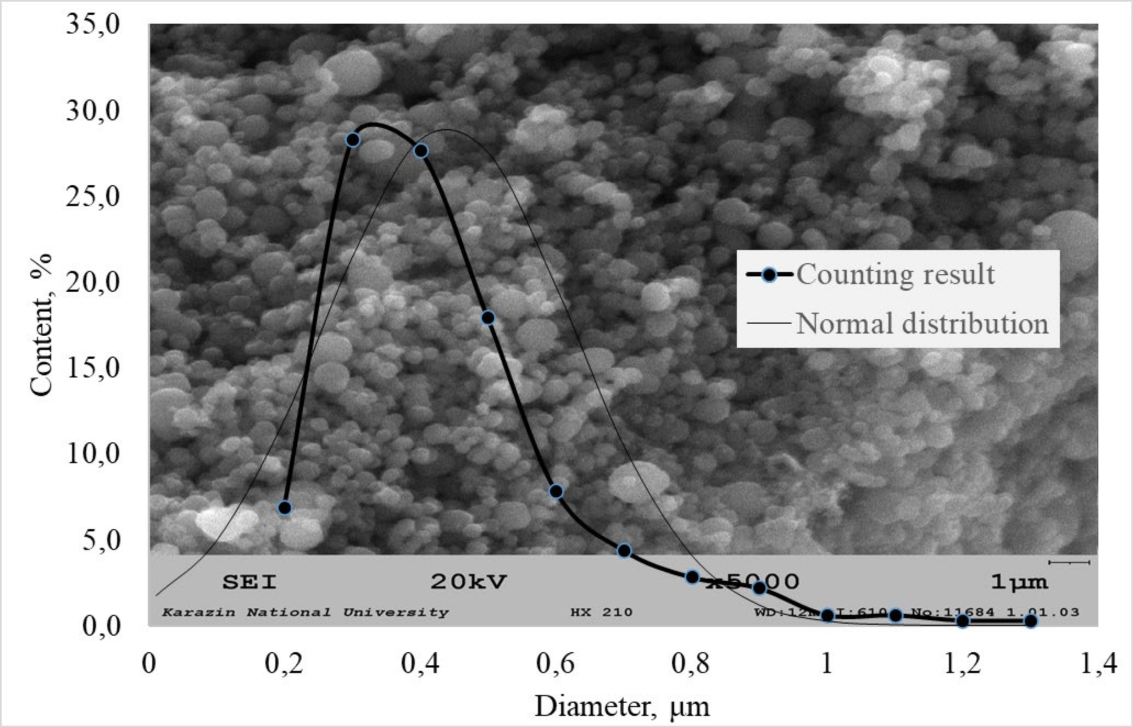

Fig. 5. Photos of quinoline insoluble particles and size distribution curves, sample R1-R3, magnification 1×5000

Despite the same concentration of particles insoluble in quinoline, their aggregate diameters in the studied samples differ significantly: tar R2 has the smallest aggregates and the highest viscosity. R3, in contrast, has the largest aggregates and the lowest viscosity. This statement is consistent with theoretical concepts of the viscosity of suspensions. Reducing the size of solid particles in suspension increases their number, while growing the degree of interaction between the particles. The resulting interaction complicates the flow of the liquid and its viscosity increases.

The measure of dispersity is the specific interfacial surface (Ss), that is, the ratio of the total surface of droplets to their total volume:

$$Ss = (\pi D_N^2)/(\pi D_N^3/6) = 6/D_N \qquad (5)$$

The specific interfacial surface of the aggregates in the high viscosity tar is greatest, which is a consequence of the increase in the degree of interaction between the aggregates with a high specific surface.

The polydispersity index (P_D) characterizes the width of the particle distribution function and is the ratio of the average particle diameter to the average particle diameter in suspension. For structured systems, this value is less than 2 units [15].

The polydispersity index is maximum for a non-viscous tar, which is consistent with the available data that the viscosity decreases with an increase in the width of the particle size distribution curve and is minimal for a bimodal distribution [16].

It is possible to analyze the size of aggregates of solid dispersed particles under a microscope directly in a tar sample in a mixture with such strong solvent as quinoline. However, the aggregates are breaking up to the smaller units, and unexpectedly, water was released in the quinoline solution of R2 tar (Fig. 6).

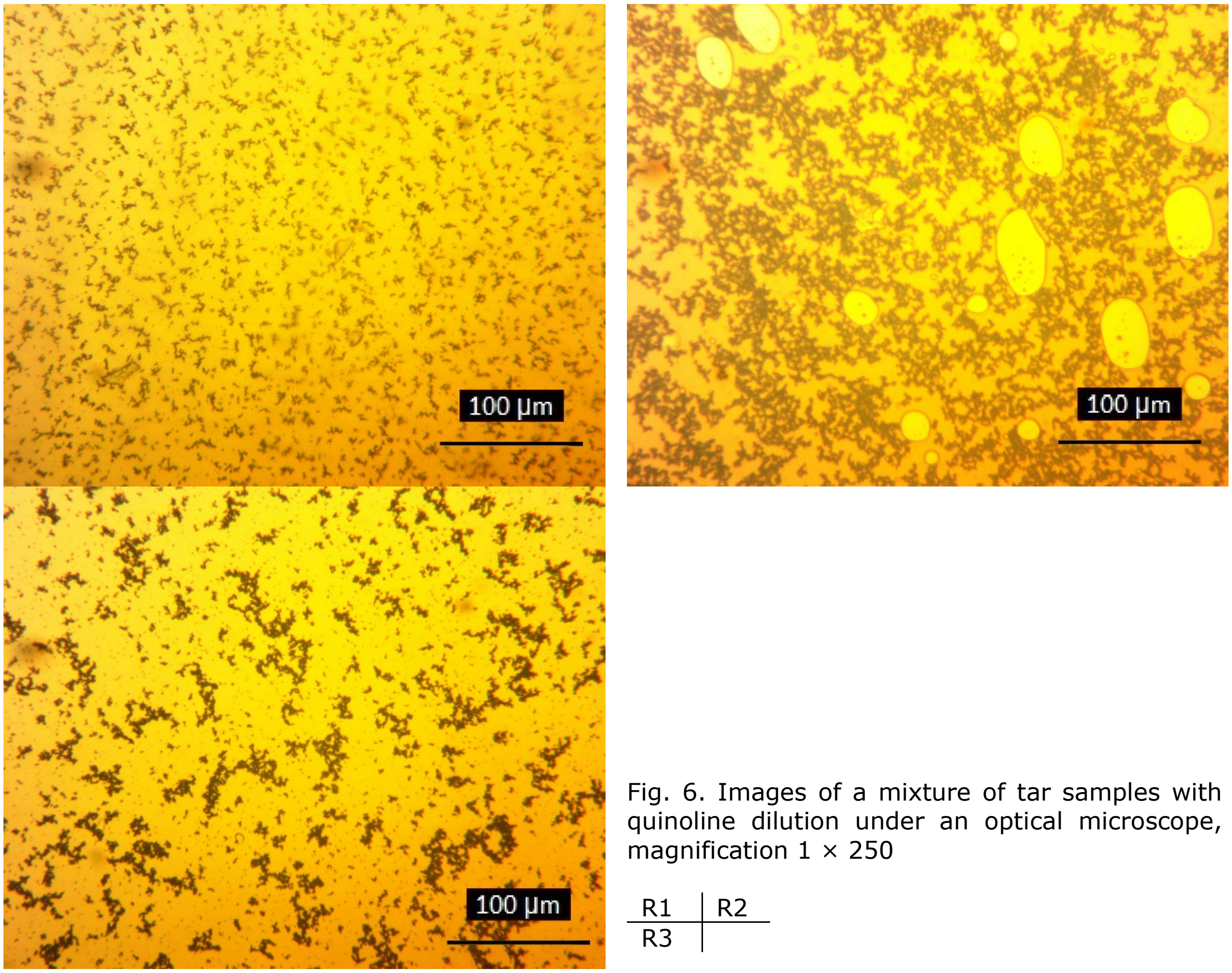

Fig. 6. Images of a mixture of tar samples with quinoline dilution under an optical microscope, magnification 1 × 250

R1	R2
R3	

The reason for this phenomenon may be a higher interfacial surface of solid dispersed particles of sample R2 (1.5 μm^{-1}) versus 0.4-0.5 μm^{-1} for samples R1 and R3. This led to a decrease in the viscosity of the dispersion medium, a weakening of the armoring layer around the droplets, and the transfer of blocking solid dispersed particles into the dispersion medium.

Presumably, there is the existence of strong bonds between large aggregates of QI substances, that counteract with such a polar solvent as quinoline and do not allow weakening the armoring layer and initiating coalescence of droplets. Apparently, larger aggregates form a stronger armor layer around water drops than smaller ones. The phenomenon of self-aggregation of substances insoluble in quinoline arises due to the bonds between the functional groups, which lowered the polarity of the formed aggregates [17].

To determine the behavior of solid dispersed particles with changes in the properties of the blend, tars with the smallest and highest aggregate sizes (R2 and R3) were mixed. Table 7 shows the corresponding measured viscosity values.

Table 7. Comparison of tar viscosity at a shear rate of 186 s^{-1} and at 60°C

Experiment No.	Content of tar samples in the mixture, %		Viscosity, cP
	R2	R3	
1	100	0	217
2	0	100	140
3	80	20	219
4	50	50	225

The addition of a high viscous tar (R2) to a less viscous one (R3) did not lead to a significant change in the measured values. It should be noted that at a lower shear rate, the difference in the viscosity of the mixtures is more significant, according to [18], the effect of the particle size distribution of the dispersed phase on the viscosity of the mixture at a high shear rate is minimal.

However, in experiment No. 3, when measuring viscosity on a rotational viscometer, water was released, which most likely occurred due to a change in the particle size distribution of the solid dispersed phase. When blending tars with the same concentration but heterogeneous solid dispersed substances, an increase in the polydispersity index of the mixture occurs, which is an indicator of a decrease in the structuredness of the system. The weakening of the structuring of the mixture contributes to the merging of water droplets and further partial dehydration. Apparently, when tars with different granulometric composition of QI substances are mixed in a certain ratio, the degree of structuredness and association of aggregates is violated.

A possible reason for the difficult settling of highly pyrolyzed tars is not only the high concentration of the solid dispersed phase and the high viscosity of the continuous phase, but also an increase in the degree of aggregation of QI substances. The latter form a more durable armoring layer around water droplets, with less wettability than small aggregates.

By analogy with the processes of electrode pitches production in air blowing reactors, it can be assumed, that during the pyrolysis of tar vapors in the under-roof space of the coke chamber, large aggregates are formed under thermal heating with lack of atmospheric oxygen. The formation of small aggregates, on the contrary, does not require long-term thermal exposure, but occurs under more thermodynamically favorable conditions of oxidative pyrolysis.

4. Conclusion

A significant effect of the granulometric composition of aggregates of QI substances on the viscosity of tars with the same content of solid dispersed substances and emulsified water has been established. Similar to the known disperse systems, the increase in the viscosity of coal tars with a high degree of pyrolysis is due to the presence of smaller aggregates with the same sizes of single QI particles.

For tar samples with large aggregates of dispersed substances, the transition of the system to an unstructured state requires a significantly lower amount of activation energy of the viscous flow, although the content of the dispersed phase in three tar samples is comparable. The size of the dispersed phase has a decisive influence on the viscosity of the studied samples, as it was previously established for coal tar pitches.

Large aggregates form a stronger armor layer around water drops than small ones. Most likely, stronger bonds arise between large aggregates of substances insoluble in quinoline, which counteract such a polar solvent as quinoline.

When blending tars with the same concentration but heterogeneous solid dispersed substances, an increase in the polydispersity index of the mixture occurs, that brings to violation of the structuring degree and association of aggregates. Ultimately, this facilitates the coalescence of water droplets and leads to partial dehydration.

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