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INFLUENCE OF PHENOLIC COMPOUNDS ON THE OPERATIONAL CHARACTERISTICS OF COAL TAR WASH OIL

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ABSTRACT

The quality of coal tar wash oil plays a key role in industrial processes for the absorption of benzene hydrocarbons from coke oven gas, and oil stability as well as resistance to polymerisation and corrosive oxidation processes are important aspects for ensuring efficient system operation. To ensure low oil consumption, it is necessary to suppress oxidative degradation processes. In this study, the effects of the phenolics of coal tar and its fractions on the antioxidant properties of the absorption oil were investigated. The results showed that phenols are effective oxidation inhibitors, promoting the formation of a protective film on the metal surface and reducing the corrosion rate of carbon steel from 0.56 to 0.33 mm year $^{-1}$. A particularly strong protective effect was observed when the anthracene fraction of phenolic compounds was used, which possesses slightly reduced acidity with respect to low-molecular-weight phenols. This result can be explained by the weakening of the strength of the intermolecular interactions between phenolic compounds and pyridine bases. This reduces the negative effect of a decrease in the antioxidant abilities of phenols and increases the antioxidant effect of phenols in the anthracene fraction. The addition of various phenolic derivatives to the working absorbing oil did not have a significant effect on the viscosity or flow characteristics of the oils under study. The introduction of phenolic derivatives into the working oil did not change its coke-forming properties. The introduction of the N,N-dimethylformamide additive into coal tar wash oil did not lead to a decrease in coking properties but slightly decreased the viscosity of the working oil. In general, the obtained results are arguments in favour of the inexpediency of phenol extraction from coal tar wash oil to reduce its consumption due to oxidative processes of thickening.

Keywords: *coal tar wash oil, phenolic derivatives, oxidative processes, inhibition.*

INTRODUCTION

The recovery of chemical coking products is a mandatory and cost-effective measure at coke-chemical enterprises in Ukraine and the world [1 - 3]. The recovery of crude benzene from coke oven gas is an economically favourable stage aimed at improving coke oven gas purification, as some carbon disulphide, naphthalene and solid suspended impurities are recovered. The absorption fraction is recovered during the rectification of coal tar and after the alkaline extraction of phenols; this fraction is called “wash oil” and is used for the absorption of benzene hydrocarbons from coke oven gas.

The choice of oil for the absorption of benzene hydrocarbons and naphthalene from coke oven gas is still an urgent problem [4], as is the preservation of its performance characteristics in the process of cyclic heating/cooling [5]. For this purpose, coal tar wash oil is sometimes modified with additives, for example, to lower the precipitation temperature [6]. An improvement in oil quality can also be achieved by optimising its fractional composition during distillation [7] or crystallization [8].

The extraction of phenols is carried out through a sodium phenolate stage, which is then processed into pure phenols in centralised plants. It is believed that dephenolized wash oil improves its operating characteristics; in particular, it is more difficult to emulsify with water, and its tendency to polymerise is reduced. However, this issue is still a matter of debate. In our opinion, it is necessary to consider that wash oil of any quality still absorbs phenols from coke oven gas, which adds to the arguments against dephenolation.

It has been established that 67 % of the phenols between coking products pass to coal tar, 32.5 % to coal tar water and 0.5 % to coke oven gas, and up to 25 % of phenols are distilled off to coke oven gas at the ammonia column and finally go to the benzene plant [9]. At one of the plants, the phenolic content in fresh oil was 0.3 %, while it increased to 1.6 % in working oil, which can only be explained by the absorption of phenols from coke oven gas [10]. As a result, phenol, *o*-cresol, *p*- and *m*-cresols [11] are found in the crude benzene distilled from the oil, with a total content of 0.1 % [12]. During the separation of benzene from vapour condensate, up to 0.3 g dm^{-3} phenol [12] is transferred to separator water, which also confirms the dynamic equilibrium between the phenol content in fresh oil, working oil, coke oven gas and crude benzene extracted.

In the coal tar wash oil (absorption fraction), the phenol content depends on the degree of pyrolisation of the tar and the clarity of tar rectification. The absorption fraction contains 5 % cresols, 30 % xylenols and 65 % more phenols. The higher boiling anthracene fraction contains 10 % xylenols and 90 % high phenols. In the absorption fraction, the concentration of phenols reached 3.6 %, and in the anthracene fraction, it reached 2.8 % [12]. In Ukraine, the phenol content in oil is regulated by the technical conditions “Coal oil absorbent TU U 20.1-00190443-117:2017” and increases from 0.7 to 3.5 % (wt. fraction) as the value of the absorbent grade decreases.

Phenolics are known to be used as additives in motor oils to increase the oxidation induction period and have been applied as antioxidants in other fields [13, 14]. Various plant derivatives of phenols and cresols exhibit antioxidant activity towards hydroperoxides, and this activity is comparable to that of known antioxidants (α-tocopherol) [15]. As the molecular weight of phenolic derivatives increases, their antioxidant activity increases [16]. The inhibitory effect of naphthols is greater than that of cresols and xylenols; however, resinous oxidation products are formed [17].

Based on the above data, there is a need to clarify the effect of phenols on the antioxidant properties of wash oil and some of its operating characteristics. Of particular interest is the effect of higher phenols, both the absorbing fraction and the higher boiling fraction (anthracene fraction). It was logically assumed that phenolic compounds with boiling fractions higher than the absorption fraction had the strongest influence on the inhibition of oxidative processes. The objective of the present study was to determine the effect of different phenols on the performance characteristics of absorption oil, such as viscosity, coking index, and corrosion aggressiveness.

EXPERIMENTAL

The influence of the composition of phenolic derivatives on the properties of absorbing oil was studied by adding them to the working circulating wash oil of the Ukrainian enterprise. The oil quality data are given in Table 1.

The oil of the presented characteristic belongs to absorbents with deteriorated performance properties; it has an increased coking index, rather than an acidic reaction of water extract, a low content of distillation up to 300°C, a low content of naphthalene, and a low content of methylated homologues of naphthalene.

The following individual compounds of “chemically pure” grade were used as antioxidant additives to the oil: phenol C_6H_5OH as a model substance of coke gas phenols; *o*-cresol, 2,4-xylenol and 2-naphthol as substances of coal tar wash oil; and an additive to automotive motor oil “anticoke”. This additive is designed to reduce the consumption of motor oil in automobile engines and to dissolve coke-like deposits, as the main substance contains N,N-dimethylformamide (DMF).

To study the antioxidant properties of heavy phenols

Table 1. Characterisation of circulating oil.

Parameters	Indicators
Density, kg m^{-3}	1086
Content of benzene hydrocarbons, %	1.2
pH of the water extract	5.42
Salt content of water extract, mg dm^{-3}	171
Ash content, %	0.123
Viscosity (25 °C), mPa s	12.9
Temperature of crystal appearance, ^{o}C	5
Coking number, %	7.65
Fractional composition:	
Initial boiling point, °C	227
Volume distilled to 230°C, %	1.4
Volume distilled to 270°C, %	70
Volume distilled to 285°C, %	80
Volume distilled 230-300°C, %	84.6
Content of components, mass fraction, %:	
naphthalene	8.15
α-methyl naphthalene	2.97
β-methyl naphthalene	5.81
diphenyl	7.13
acenaphthene	8.57
dibenzofuran	9.03
fluorene + indole	6.17
anthracene + phenanthrene	1.45

Table 2. Characterisation of the samples under study.

Sample number	Additive (3 % wt.)
0	-
1	phenol
2	*o*-cresol
3	2,4-xylenol
4	2-naphthol
5	phenols of anthracene oil
6	Additive to automotive engine oil “anti-coke”

in coal tar, the anthracene fraction was dephenolized with 15 % alkali solution, followed by decomposition of the obtained phenolates with 20 % sulfuric acid. The composition of the obtained oil samples is presented in Table 2.

The rheological characteristics of the investigated oils with additives were determined on a Brookfield DV2T rotary viscometer with a thermocell at a fixed temperature and variable shear rate.

To evaluate the performance characteristics, the influence of phenols on the yield of coke residue (coking ability) was determined in accordance with the requirements of TU U 322-00190443-060-96. The method of determination involved removing volatile substances from the bulk of the analysed product in a closed porcelain crucible at a temperature of 850 ± 25°C for 10 min, after which the mass of the coke residue was further determined. The yield of coke residue reflects the tendency of the oil to form coke at high temperatures and indicates the ability of the oil to retain its properties at elevated temperatures. In addition, the coking index can indicate the resistance of oil to oxidation; oils with low coking indices can provide longer life at high temperatures without significant coke formation.

To establish the corrosion aggressiveness of the oils, aqueous extracts of the oils were isolated. A tenfold amount of distilled water was added to a sample of oil and boiled for 0.5 h with a reflux condenser, after which the aqueous layer was filtered through a paper filter.

Electrochemical corrosion laboratory studies were carried out based on the plotting of polarisation curves obtained from measurements in a three-electrode cell with separated electrode spaces. The measurements were carried out using a voltammetric potentiostat (MTech COR-50F) combined with a personal computer in a standard three-electrode cell. The temperature of the solution was maintained at 50°C during the measurements using a U-10 thermostat. The corrosion current density is proportional to the corrosion rate of the metal in the studied medium. The latter was determined by extrapolation of Tafel plots on the obtained polarisation curves to the value of the free corrosion potential. The test specimen was an electrode made of constructional carbon steel (St. 3). The rate of metal corrosion in the solution under study was determined by the following formula:

$$K,\ \mathrm{g\ m^{-2}\ h^{-1}} = \frac{i \times A}{n \times 2.68 \times 10^{-2}}, \qquad (1)$$

where A is the atomic mass of iron, n is the number of electrons participating in the electrochemical reaction, and i is the corrosion current density, A cm^{-2}.

Based on the determined corrosion rates, the protective effect of the corrosion inhibitor was determined by the following formula:

$$Z = \frac{Kм - Kм(inh)}{Kм} \times 100\ \%, \qquad (2)$$

where K_M is the corrosion rate of a metal in a given medium and $K_{M(inh)}$ is the corrosion rate of a metal in a given medium with the addition of a corrosion inhibitor.

RESULTS AND DISCUSSION

Fig. 1 shows the viscosity curves of the tested oil samples.

The addition of phenols with high polarity with respect to the neutral components of the wash oil leads to an increase in the viscosity of the oil mixture. As the molecular weight of phenols increases, the viscosity of the mixture increases by 0.4 - 1.6 mPa s in the Newtonian flow region. The introduction of a solvent (DMF) possessing a high dielectric constant lead to a decrease in the viscosity of the working oil by 0.8 mPa s, which is apparently due to the low viscosity of the basic substance (DMF) and the corresponding dilution of the dispersion medium of the colloidal structure of the working wash oil.

The dependence of viscosity on shear rate allows us to estimate the rheological behaviour of the liquid. The viscosity of the working wash oil without additives, as well as its mixture with various additives, does not depend on the shear rate at values above 20 - 30 s^{-1}. Thus, the oils exhibit non-Newtonian flow behaviour, and power-law model coefficients were established for the investigated oils, expressed by the following equation [18]:

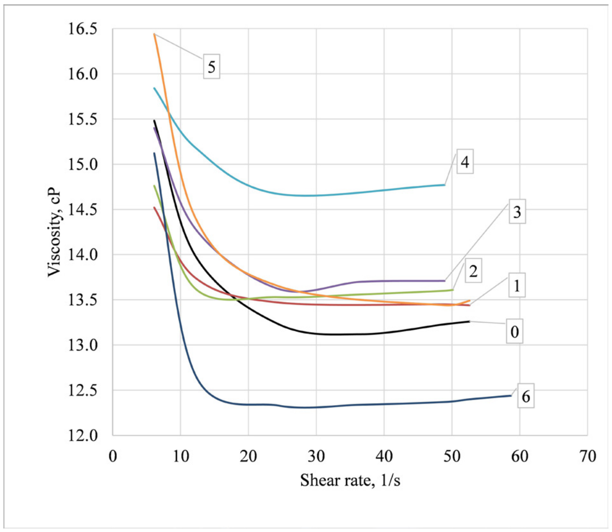

Fig. 1. Dependence of viscosity of oils with additives on shear rate (30°C).

$$\tau = K\times(\gamma)^n \qquad (3)$$

where τ is the shear stress; γ is the shear rate; K is the consistency index; and n is the flow behaviour index.

Calculation of the model coefficients is possible by taking the logarithm of equation (3):

$$\log(\tau) = \log(K) + n\times\log(\gamma) \qquad (4)$$

The results of the calculation of the coefficients are presented in Table 3.

The calculated coefficient n is in the range of 0.91 - 0.98, which indicates that the viscosity decreases with increasing shear rate. Fresh wash oil, which was introduced into the working cycle, had a value of 0.98.

Table 3. Viscosity of wash oil of different compositions and coefficients of the power-law model.

Sample number	Viscosity, mPa s, at shear rate 30 - 40 s^{-1}	Consistency index, K	Flow behaviour, n
0	13.1	0.17	0.93
1	13.5	0.15	0.97
2	13.6	0.15	0.97
3	13.6	0.15	0.97
4	14.7	0.16	0.98
5	13.7	0.19	0.91
6	12.3	0.16	0.93

The introduction of individual phenolic derivatives into the working oil affected the molecular or structural arrangement of the oil molecules, as it increased the value of n from 0.93 to 0.97 - 0.98. This alteration in the internal structure may contribute to more Newtonian flow behaviour and reduce the shear-thinning effect.

The greatest deviations from the ideal liquid flow (n = 0.91) were observed for mixtures with additives of higher amounts of phenolics in the anthracene fraction, which can be explained by the presence of neutral oils extracted during the process. In general, the introduction of all the additives insignificantly changes the viscosity of the working oil, especially the phenol-cresol and phenol fractions of anthracene.

In addition to the viscosity of oils, the coking index (coke residue yield) is also an integral characteristic of oils, as it is correlated with density, viscosity, component composition, fractional composition, content of substances insoluble in toluene, and ash content. Monitoring the quality of working oils of benzene plants in Ukraine has shown that the coking ability of good-quality oils is up to 4 %, while that of satisfactory-quality oils is up to 7 - 8 %. In the absence of oil regeneration, frequent watering, equipment failure for cleaning and the elimination of corrosion damage, the coking ability reached 15 - 18 %.

Polycyclic hydrocarbons, which condense under the influence of temperature to form coke nuclei, are considered initiators of coking processes. The results of determining the value of the coking behaviour of the investigated oil mixtures are given in Table 4.

As the results of the determination showed, the introduction of the studied additives did not lead to an increase in the coking index of the mixtures, and the greatest decrease in the yield of coke residue (by 0.2 %) was shown by the addition of 2-naphthol. Based on the obtained data, none of the additives can be considered conditional "anticoking", and the introduction of phenols does not change the coking properties of the ash oil.

Thus, the action of phenolic antioxidants under these experimental conditions did not affect the formation of polycyclic hydrocarbons involved in the formation of the "coke skeleton". This may have been because phenols, as antioxidants, are effective at inhibiting oxidation during the initial period after their addition to the oil. They can neutralise free radicals and prevent the initial stages of oxidative processes. However, when coke numbers are determined at higher coke formation temperatures, there is a more intensive thermal effect on the oil. In this case, phenols may no longer be as effective in preventing the formation of coke deposits that may result from long-term thermal effects.

Table 4. Coke residue yield.

Sample number	Coke number (average of three experiments)
0	7.70
1	7.70
2	7.70
3	7.78
4	7.50
5	7.80
6	7.68

The undoubted positive property of the investigated additives is that they do not increase the coking behaviour of wash oil. Previous studies have shown that the coke residue yield of fresh oils fluctuates in the range of 0.02 - 0.05 %, and that of recycled oils from unsatisfactorily operating benzene plants reaches 15 - 20 % [19]. The results of the coking ability determination showed that the absence of the oil dephenolization stage does not worsen the tendency of oils to form precipitates and coke-like deposits during heating.

The study of the antioxidant abilities of phenols was carried out by determining the corrosion activity of oil with different antioxidant additives by analysing the corresponding aqueous extracts (Tables 5, 6).

The salt composition and hydrogen index (pH) of aqueous extracts largely determine the corrosion rate of carbon steel, and at the established values, the predicted index is 0.60 - 0.70 mm year^{-1}, according to the data [20].

The practice of benzene plant operation shows that the corrosion activity of absorbing oil and its polymerization, thickening and degradation are related processes. The corrosion rate of carbon steel in aqueous extracts of working oil sampled from stable operating benzene plants is 0.1 - 0.25 mm year^{-1}. A similar indicator for unsatisfactorily operating benzene plants was an increase of up to 1.0 - 1.2 mm year^{-1} [20].

The ammonium salts of strong acids hydrolysed in aqueous extracts determine the acidity of the medium, and the introduction of the studied additives does not significantly change the salt content or the hydrogen

Table 5. Characteristics of aqueous extracts of the produced oils.

Sample number	pH	Conductivity, μS cm^{-1}	Salinity*, mg dm^{-3}
0	5.50	233	155
1	5.48	253	167
2	5.38	245	163
3	5.42	247	164
4	5.20	249	166
5	5.37	242	161
6	5.47	250	167

**- determined by a conductometric electrode*

Table 6. Salt composition of the aqueous extract of the working oil.

Total dissolved salts, mg dm^{-3}	Anions:				
	CNS^-	Cl^-	CN^-	S^{2-}	SO_4^{2-}
231	75	19	12	23	34

index. The main cause of equipment corrosion is the presence of water-soluble thiocyanate compounds and, to a lesser extent, ammonium chlorides in oil. Ammonium chloride is introduced into the working absorption oil with coke oven gas containing aerosols of chloride-containing supernatant water. Ammonium thiocyanates are a product of oxidation processes under the influence of catalytic complexes and oxygen:

$$Fe^{3+} + xHS^- \rightarrow Fe^{2+} + S_x^- + xH^+ \qquad (5)$$

$$2Fe^{2+} + 0.5O_2 + H_2O \rightarrow 2Fe^{3+} + 2OH^- \qquad (6)$$

$$xCN^- + S_x \rightarrow xCNS^- \qquad (7)$$

The addition of phenols can inhibit this oxidative mechanism and prevent the formation of strong acid salts (thiocyanates). The results of the electrochemical determination of the corrosion rate are given in Table 7, and the characteristic potentiodynamic curves are shown in Fig. 2.

The results show that the highly boiled phenols in the anthracene fraction have the greatest protective effect, and *o*-cresol and 2-naphthol also reduce the corrosion rate well and have a strong protective effect. These results agree with the findings of greater oxidation protection of benzene products when naphthols are introduced than when cresols are introduced [21]. This suggests the inexpediency of the dephenolation of coal tar absorbing oil, which contains heavy phenols (cresols, naphthols and some phenolic compounds of the anthracene fraction).

The greatest shift in the free corrosion potential towards an increase for oil extracts with additives of phenols in the anthracene fraction and *o*-cresol is consistent with the greatest protective effect of these antioxidants.

The most effective antioxidant additives are crude phenols isolated from the anthracene fraction, which are represented mainly by relatively high amounts of phenols. The composition of these phenols has not been completely studied; usually, the isolation of crude phenols from the anthracene oil fraction is carried out up to the fraction with a boiling range of 330 - 340°C [22].

The action of phenols as inhibitors of oxidation is not limited to preventing the reaction (6); in the wash oil, parallel tar formation processes occur. Analogous to tar formation processes in crude benzene, these processes are preceded by oxidation processes leading to the appearance of peroxide compounds, and the action of inhibitors is based on their introduction into the fresh product to prevent the formation of the aforementioned peroxide compounds [21]. Under the action of peroxide compounds, phenols are able to oxidise to form stable phenoxyl radicals, which results in substituted phenol inhibitors of chain reactions [17]. Apparently, the phenols in the anthracene fraction form the most stable free radicals during oxidation. The most likely representatives of inhibitors (high-boiling phenols of the anthracene fraction) are butyl derivatives in the

Table 7. Results of the determination of the corrosion rate of carbon steel in aqueous extracts of oils at 60°C.

Sample number	Free corrosion potential, mV	Corrosion current I, mA	Corrosion rate, mm year^{-1}	Protective effect of inhibitor, %
0	-0.589	0.0468	0.56	-
1	-0.544	0.0398	0.47	16.1
2	-0.500	0.0331	0.39	36.2
3	-0.527	0.0501	0.59	-
4	-0.533	0.0302	0.36	33.9
5	-0.506	0.0275	0.33	63.9
6	-0.547	0.0457	0.54	6.1

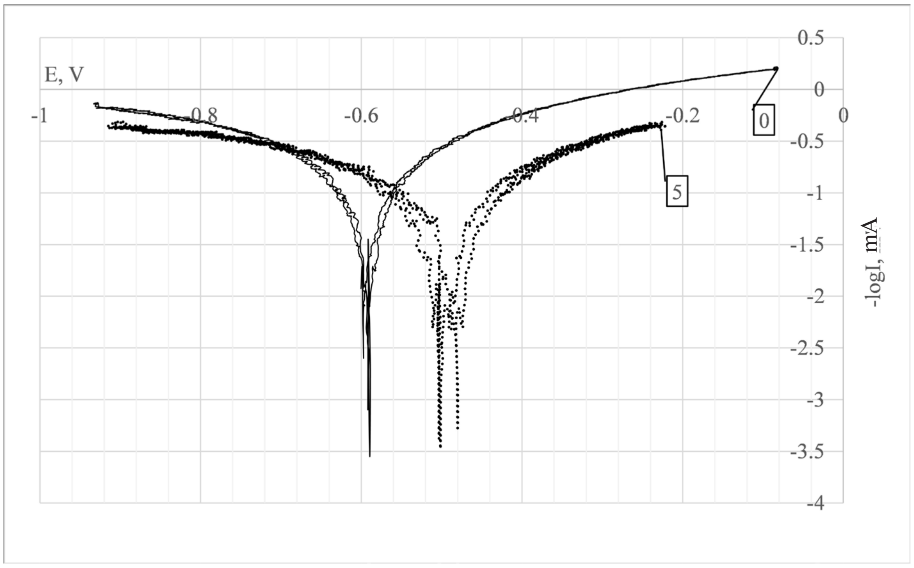

Fig. 2. Polarisation curves of carbon steel in aqueous extracts from working absorption oil (item 0) and from oil with added anthracene fraction phenols (item 5).

meta- and *para*-positions since the increase in the boiling point upon introduction of such radicals is maximal [12].

The protective effect of phenols against the corrosion of carbon steel is also possible due to the formation of a film on the surface because of the interaction of iron (III) ions with phenols. The molecular structure of the complex formed with 2-naphthol is presented in Fig. 3.

The formed film of iron (III) complex phenolates can serve as an additional factor that provides a strong protective effect and sterically hinders the access of water and oxygen molecules to the metal surface.

The obtained data show that the positive effect of the presence of more phenols in the anthracene fraction in the wash oil is associated with a shift in the free corrosion potential of the carbon steel in the positive direction and a decrease in the corrosion rate. This inhibitory effect may be related to the creation of a protective film on the metal surface and to the effect of phenols on the oxidative processes occurring in the working oil. If phenols are successful in slowing these oxidative processes, this may help to reduce the intensity of oil degradation processes, resulting in increased oil density, viscosity and related performance characteristics.

The mechanism of the antioxidant properties of phenols in homogeneous solutions was considered by Foti and is based on the binding of formed peroxide radicals (ROO*) upon transfer of the hydrogen atom of the phenolic group [23]:

$$ArOH + ROO^* \rightarrow ArO^* + ROOH \qquad (8)$$

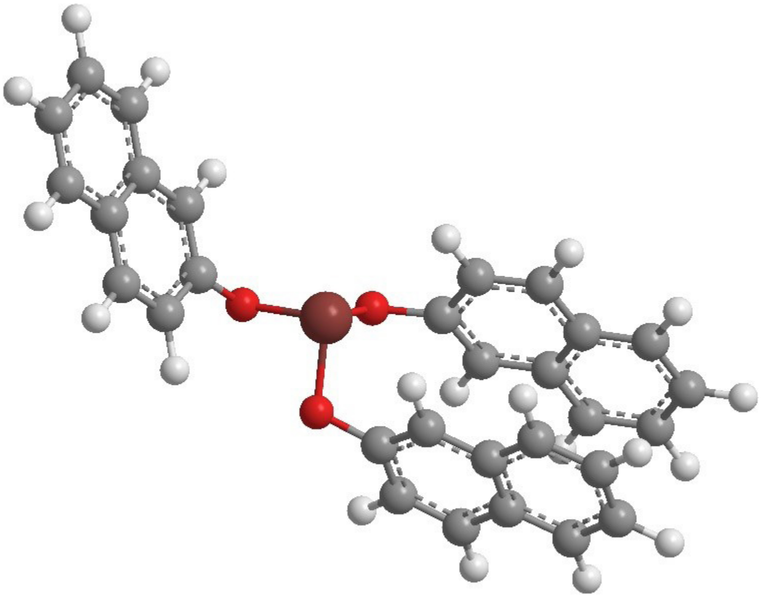

Fig. 3. Complex of iron (III) with 2-naphthol.

with the formation of the resonance-stabilised radical ArO*, a sterically protected and inactive phenolic substance. Moreover, it was found that the antioxidant abilities of phenols are strongly reduced in the presence of solvent acceptors due to the formation of hydrogen bonds of the ArOH---S type, where S is the solvent.

It is known that the strongest intermolecular interactions in the medium of resin, especially when absorbing oil, are observed in the presence of phenols and pyridine bases. The strength of such complexes is related to the difference in the dissociation constants (pK) of these compounds [24]:

$$DpK = pK_s - pK_{ArOH} \qquad (9)$$

It follows that high-molecular-weight phenols of the anthracene fraction, which possess lower acidity than phenols of the absorption fraction and coke gas, form weaker intermolecular complexes with pyridine bases. This approach reduces the negative effect of a decrease in the antioxidant ability of phenols and can serve as one of the explanations for the greater antioxidant activity of the phenols in the anthracene fraction.

Since the combined presence of phenols and pyridine weakens the antioxidant abilities of phenols, their protective effect may be enhanced at low contents of pyridine bases in the working oil. This may explain the ambiguous conclusions from the discussion on the expediency of despyridination and dephenolization of the absorption fraction. At Ukrainian enterprises, the despyridination of fresh oils was obligatory because of the centralised processing of the crude pyridine fraction, but recently, oils have not been despyridinated.

CONCLUSIONS

Phenolic substances of coal tar and its fractions exhibit antioxidant properties in the environment of working wash oil, which was established based on the effect of phenols on oxidative processes occurring in aqueous extracts of working wash oil with the addition of various phenolic derivatives. The action of phenols, as oxidation inhibitors, should extend to the parallel reactions of tar formation, as these processes are preceded by oxidation, leading to the appearance of peroxide compounds.

The reduction in the corrosion rate of carbon steel in the aqueous extract of oils with the addition of phenols is also promoted by the creation of a protective film on the metal surface with the presumed formation of complex compounds with the oxidised form of iron.

The presence of pyridine derivatives in the wash oil leads to the binding of phenols in strong intermolecular complexes, which can weaken the antioxidant properties of phenols.

The greatest protective effect and the strongest influence on the inhibition of oxidative processes are achieved when phenolic compounds in the anthracene fraction are added to the wash oil, as these are the most common high-molecular-weight phenolic derivatives with lower acidity. This reduces the antioxidant properties of higher phenols to a lesser extent, as it leads to a certain weakening of intermolecular bonds with pyridine bases.

The presence of phenolic derivatives in the working wash oil did not significantly affect the viscosity or coking behaviour of the oil.

Authors' contributions: D.M.: conceptualization; A.B.: investigation, whriting; L.B. whriting; S.N.: experimental work.

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