Copper strip corrosion testing in hydrocracked base oil in the presence of different inhibitors

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Abstract

In this paper, the corrosion test of copper in hydrocracked base oil HC-6 was performed in the presence of an additive for extremely high pressures (EP additive) in different concentrations. EP additives are used to reduce wear in industrial applications, under high load conditions. Since most of these additives are sulfur-based, whose compounds can be corrosive at high temperatures, their use leads to corrosion of some materials. To prevent corrosion in the base oil with the EP additive, three commercial corrosion inhibitors are added. By chemical composition, the inhibitor RC 8210 is a derivate of dimercaptothiadiazole, RC 4220 is a synthetic neutral calcium sulfonate, and IRGAMET 39 is a derivative of tolutriazole. Efficiency of the inhibitors was monitored by standard test methods for corrosiveness to copper arising from petroleum products by the copper strip test (ASTM D-130) and the gravimetric method, while oxidation stability of the base oil was monitored by peroxide number determination. Oxidation was performed at 100±1 °C for 3 and 24 h. Results of these studies have shown that IRGAMET 39 is the most effective inhibitor in the presence of the EP additive at both examined oxidation times.

Keywords: ASTM D-130; copper coupons; lubricating oil; extreme pressure additive.

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1. INTRODUCTION

Corrosiveness of petroleum products indicates the product ability to cause metal corrosion. The main cause of corrosivity of lubricating oils is sulfur and its compounds, such as organosulfur molecules, thiophenes, disulfides, polysulfides, dialkyl sulfides (thioethers), and mercaptans (thiols). The origin of sulfur in lubricating oils can be from the base oil obtained by a basic oil production technology and refining process, or from various additives. Each compound exhibits its own unique reaction rate with copper, which ultimately forms copper sulfide species, as solid corrosion products or complexes, depending on the concentration of the compounds, copper surface condition, temperature, and aging time [1].

The use of extreme pressure (EP) additives leads to the formation of protective layers upon high loading in the friction process. These additives consist of chlorine, sulfur, and phosphorous compounds, which react tribochemically with the metal surface during the mechanical interaction and develop a well adhered and easy-to-shear protective layer [2]. Sulfur in EP additives reacts with the metal forming a tribofilm that improves the friction and wear behavior [3], but also causes corrosion of metals with which it comes into contact.

As copper is susceptible to corrosion, it is often used as an indicator of corrosiveness of petroleum products. There are a number of different standards to measure copper corrosion. The copper strip corrosion test is one of the most frequently used methods designed to assess the relative degree of corrosiveness of petroleum products [4,5].

Many studies on the issue of corrosion in lubricating oils are available in the literature, such as studies on transformer oils [6-11], as well as on the most commonly used inhibitors for these and other types of lubricating oils [12-14].

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For efficient prevention of copper corrosion, metal passivators and deactivators can be used, which can be sulfur- and nitrogen-based. The most commonly applied are benzotriazole, toluylbenzotriazole, and aminomethyl-substituted toluylbenzotriazole (better known as Irgamet 39). In a study on the effect of Irgamet 39 on the copper strip oxidation after 4 h at 150 °C formation of a complex of impermeable film on the metal surface was found, which protected copper from corrosion and improved oxidative stability [9]. Also, effects of Irgamet 30 and Irgamet 39 on the oxidative stability of oil and copper corrosion were compared showing that Irgamet 30 inhibits oil oxidation but does not passivate the copper surface like Irgamet 39 [10].

In another study, corrosive sulfur in the form of dibenzyl disulfide (DBDS), dodecanethiol (DDM) or a combination of both and different concentrations of passivators Irgamet 39, 1-methylbenzotriazole (marked as T571) and *n,n*-Bis(2-ethyl-hexyl)-4-methyl-1h-benzotriazole-1-methanamine (marked as TTA) were added to the non-corrosive transformer oil [11]. All examined passivators have shown a protective effect against DBDS. Irgamet 39 at a concentration of 200 mg kg^{-1} was shown to be the best choice for protection against DDM, while T571 proved to be the best passivator of copper in the presence of a combination of DBDS and DDM [11].

Ling and coworkers investigated the interaction mechanism of 2,5-dimercapto1,3,4-thiadiazole (DMTD) with copper and concluded that a reaction occurs at room temperature, building a Cu-DMTD complex. The adsorption nature of DMTD on copper surface is chemical. Sulfur atoms from DMTD molecules react with copper surface and polymerize into a polymer chain that covers the copper surface [12].

In addition to corrosion inhibitors, additives that improve the performance of EP additives, as well as provide antioxidant and dispersive and detergent properties, can be added to base oils. Synthetic calcium sulfonates have been shown to be the most effective for these purposes. In addition to cleaning, detergents also neutralize acidic combustion and oxidation products, thereby, minimizing corrosion, rust, and deposit formation in the engine [13,14].

The aim of this paper was to evaluate the influence of a sulfur-based EP additive on the corrosivity and oxidative stability of hydrocracked base oil HC-6. Since the tested additive causes corrosion of nonferrous metals, the protection efficiency of various inhibitors on the corrosion of copper was also examined.

2. EXPERIMENTAL

In the experimental part the corrosivity of copper strip in hydrocracked base oil HC-6 (Modriča Oil Refinery, Bosnia and Herzegovina) was tested in the presence of the EP additive and with the addition of three different (commercial) corrosion inhibitors. HC-6 base oil was obtained by the hydrocracking process at the Modriča Oil Refinery (Bosnia and Herzegovina). According to properties presented in Table 1, this oil has a high viscosity index, excellent oxidative stability, low sulfur and aromatic hydrocarbon contents and very low volatility.

Table 1. Characteristics of HC-6 [15]

Characteristic	Test method	Value
Viscosity at 40 °C, mm^2 s^{-1}	BAS ISO 3104	34.25
Viscosity at 100 °C mm^2 s^{-1}	BAS ISO 3104	5.96
Viscosity index	BAS ISO 2909	119
Flow point, °C	BAS ISO 3016	-8
Color (ASTM)	BAS ISO 2049	1.0
Flash point, °C	ISO 2592	260
Density at 15 °C, kg m^{-3}	ASTM D 5002	854.9
Sulfur content, ppm	BAS ISO 20846	30.0
Volatility (NOACK test), wt.%	DIN 51581	6.75

Used EP additive 7038 N (Additiv-Chemie Luers GmbH, Germany) contains sulfur based on vegetable ester and olefines [16], and three commercial inhibitors were used as corrosion inhibitors: IRGAMET 39, RC 4220 and RC 8210, whose chemical compositions and technical characteristics are given in the accompanying MSDS [17-19]. By chemical composition the inhibitor IRGAMET 39 (BASF Corporation GmbH, Germany) is based on the derivative tolutriazole, RC 4220 (Rhein Chemie Rheinau GmbH, Germany) is synthetic neutral calcium sulfonate, and the inhibitor RC 8210 (Rhein Chemie Rheinau GmbH, Germany) is a dimercaptothiadiazole derivative. All these inhibitors are soluble in mineral and synthetic base oils, and compatibility with other additives needs to be determined experimentally.

Copper strip corrosion test was performed in accordance with the standard method ASTM D130 [20]. This standard contains a color chart for the copper strip corrosion test (Fig. 1). Oxidation of copper coupons (99.95 % Cu, Hemija Patenting d.o.o Lukavac, Bosnia and Herzegovina) in base oils, base oils with the EP additive and base oils with the EP additive and different concentrations of corrosion inhibitors was performed at 100±1 °C for 3 and 24 h. After oxidation, copper strip tests are degreased and dried and their color is compared with the standard color chart.

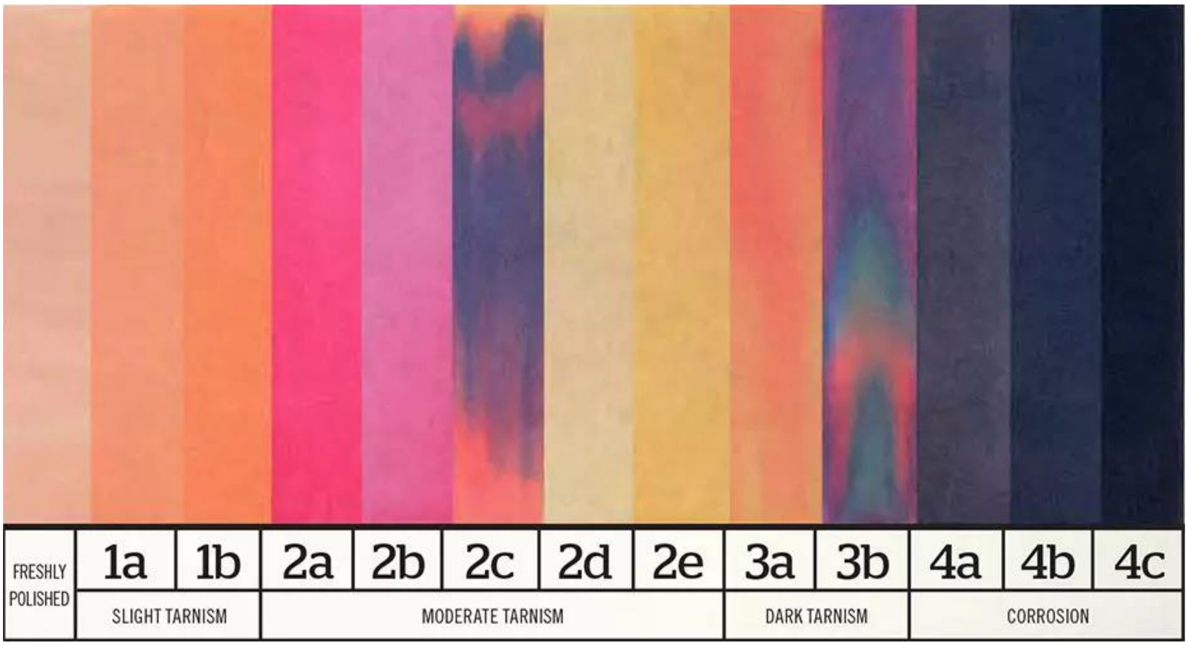

Figure 1. Color chart according ASTM D 130 copper strip corrosion standards

After 24 h of oxidation, change in the mass of copper coupons - weight growth, Δm / g was determined as well as the positive mass corrosion index, K_m^+ /g m^{-2} h^{-1}. Based on the value of K_m^+ the negative mass corrosion index, K_m^- / g m^{-2} h^{-1} was determined and the corrosion rate, π / mm year^{-1} was thus determined by the gravimetric method and calculated by the following equations:

$$K_m{}^+ = \frac{\Delta m}{st} \tag{1}$$

$$K_m{}^- = K_m{}^+ \frac{nA_{\text{Me}}}{A_{\text{O}}} \tag{2}$$

$$\pi = K_m{}^- \frac{8.76}{d_{\text{Me}}} \tag{3}$$

where s is the copper coupon surface (m^2), t is the oxidation time (h), n is the valence of the metal, A_{Me} is the atomic mass of the metal (g mol^{-1}), A_{O} is the atomic mass of the oxide (g mol^{-1}) and d_{Me} is the metal density (g cm^{-3}) [21].

Oxidation impact on the quality of base oil was monitored by peroxide number in accordance with the standard ASTM D 3703-99 [22]. During the process of oil oxidation, free radicals are formed first, followed by formation of peroxides and hydroperoxides, aldehydes, ketones, acids, esters, and finally resins and asphaltenes. The standard method is based on iodide oxidation to iodine by peroxide in an acidic medium, followed by the reaction with sodium thiosulfate until the blue color of starch indicator disappears and a colorless solution is obtained. The purpose of the oxidative stability determination is to assess the service life and behavior of the lubricating oil during exploitation [23].

3. RESULTS AND DISCUSSION

The first part of the research consisted of testing copper corrosivity in the base oil HC-6 alone and in the oil with addition of the EP additive at different concentrations, during 3 h at the temperature of 100±1 °C. Due to the limited solubility of sulfur contained in EP additives in mineral oils [24], the tested range of the additive concentrations was 0.25 – 1 wt.%

At all tested EP additive concentrations, the color of the copper strip changed (different grades of tarnish) and copper corrosion started at the concentration of 0.25 wt.% (Table 2).

Table 2. Copper corrosion at different concentrations of EP additive (3 h at 100 ± 1 °C)

Concentration of the EP additive in base oil HC-6, wt.%	Copper strip corrosion according to the ASTM D 130
0.00	1a
0.25	3a
0.50	3b
0.75	4a
1.00	4a

In the further test, corrosion inhibitors were added at various concentrations, in the range of 25 - 200 ppm. The results are presented in Table 3, which indicates that IRGAMET and RC 8210 inhibitors were effective already at 25 ppm (1b). By applying the RC 4220 inhibitor at concentrations of 25 and 50 ppm, the corrosion degree is same as without the inhibitor, while at concentrations higher than 75 ppm the corrosion degree is even increased. This inhibitor is based on calcium sulfonate, so there is a possibility that by the increase in inhibitor concentration as more sulfur is introduced, the corrosion process is promoted with the formation of copper sulfide.

Table 3. Copper strip test in HC-6 oil with 0.25 % EP additive and various concentrations of corrosion inhibitors (3 h at 100 ± 1 °C)

Inhibitor concentration, ppm	Copper strip corrosion in according to ASTM D 130		
	IRGAMET 39	RC 4220	RC 8210
0	3a	3a	3a
25	1b	3a	1b
50	1b	3a	1b
75	1b	3b	1b
100	1b	3b	1b
200	1b	3b	1b

In this series of experiments the mass change of copper coupons was negligible, due to the short oxidation time. Since lubricating oils need to ensure good lubrication under extreme conditions and for several hours of operation, examined oxidation time has been extended and the results are presented in Table 4.

Table 4. Copper strip test in HC-6 oil with 0.25 % EP additive and various concentrations of corrosion inhibitors (24 h oxidation at 100±1 °C)

Inhibitor concentration, ppm	**Copper strip corrosion in according to ASTM D 130**		
	IRGAMET 39	**RC 4220**	**RC 8210**
0	3b	3b	3b
25	1b	4a	2c
50	1b	4a	2b
75	1b	4a	2e
100	1b	4b	2a
200	1b	4b	1b

Due to the longer oxidation time, the change in mass was measurable, so the corrosion rate was calculated by the gravimetric method (Fig. 2).

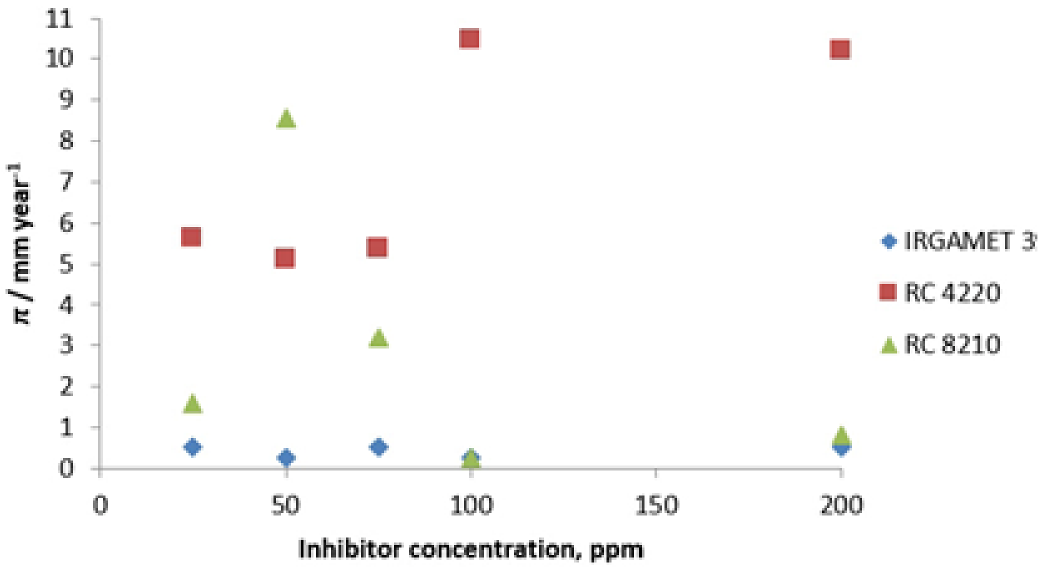

Figure 2. Copper corrosion rate in HC-6 oil with 0.25 % EP additive and various concentrations of corrosion inhibitors (24 h oxidation at 100±1 °C)

In the case of IRGAMET 39, which is derivative of tolutriazole, the mass of coupons remained almost the same before and after 24 h of oxidation. This result indicates the protective effect of the inhibitor and prevention of corrosive sulfur action from the EP additive on the copper coupon. It can be concluded that even at prolonged oxidation this inhibitor is effective even at the lowest tested concentration of 25 ppm. Benzotriazoles and their derivatives have been shown to be effective inhibitors of copper corrosion and act as passivators. Passivators react by blocking the sites for corrosive sulfur compounds and are used as a remediation technique to protect copper against the corrosive sulfur attack and formation of copper sulfide [7,9,10].

By adding the RC 4220 inhibitor the coupon weight increased and thus the corrosion rate increased with increasing the inhibitor concentration. The ASTM D 130 method indicated that the increase in concentration of this inhibitor increases "tarnish" of the coupon, so that this inhibitor is not effective in the tested concentration range.

In the case of RC 8210 inhibitor (dimercaptothiadiazole derivative), the coupon weight increased with increasing the inhibitor concentration followed by the weight decrease. At concentrations of 100 and 200 ppm the change in coupon mass is negligible, with the lowest degree of coupon corrosion according to the ASTM D 130 method, leading to the conclusion that this inhibitor is effective at concentrations higher than 100 ppm.

The quality of lubricating oil and amounts of oxidation products formed were monitored by the peroxide number. The following table shows the values of peroxide numbers during 3 h in the HC-6 base oil and the oil with the EP additive and different concentrations of inhibitors.

Table 5. Peroxide number in HC-6 oil with 0.25 % EP additive and different concentrations of inhibitors (3 h at 100°C±1°C)

Inhibitor concentration, ppm	Peroxide number, mmol kg^{-1}		
	IRGAMET 39	RC 4220	RC 8210
0	0.22	0.22	0.22
25	0.20	0.23	0.45
50	0.21	0.21	0.51
75	0.15	0.26	0.68
100	0.17	0.32	0.82
200	0.23	0.27	0.98

By inspecting the peroxide number values of the oil with 0.25 % EP additive at 3 h (Table 5) and 24 h of oxidation (Figures 3 - 5) it can be seen that the oxidation time has a negative impact on the oxidative stability of the lubricant, since the peroxide number increased with time.

Inhibitors IRGAMET 39 and RC 4220 had negligible impacts on the HC-6 oil oxidative stability during 3 h of oxidation, based on statistically not different peroxide number values obtained in the systems with and without inhibitors. During 24 h of oxidation, in the systems with mentioned inhibitors the peroxide number was reduced, indicating that both inhibitors improve oxidative stability of the lubricant.

Addition of RC 8210 at increasing concentrations increases the tendency of the oil to form oxidation products, as the peroxide number increased at both 3 h and 24 h of oxidation.

Based on the folowing diagrams (Figures 3 - 5) it can be seen that the addition of inhibitors IRGAMET 39 and RC 4220 at a concentration of 50 ppm, the lowest values of peroxide number and corrosion rate, while with inhibitors RC 8210 these values are lowest at a concentration of 25 ppm.

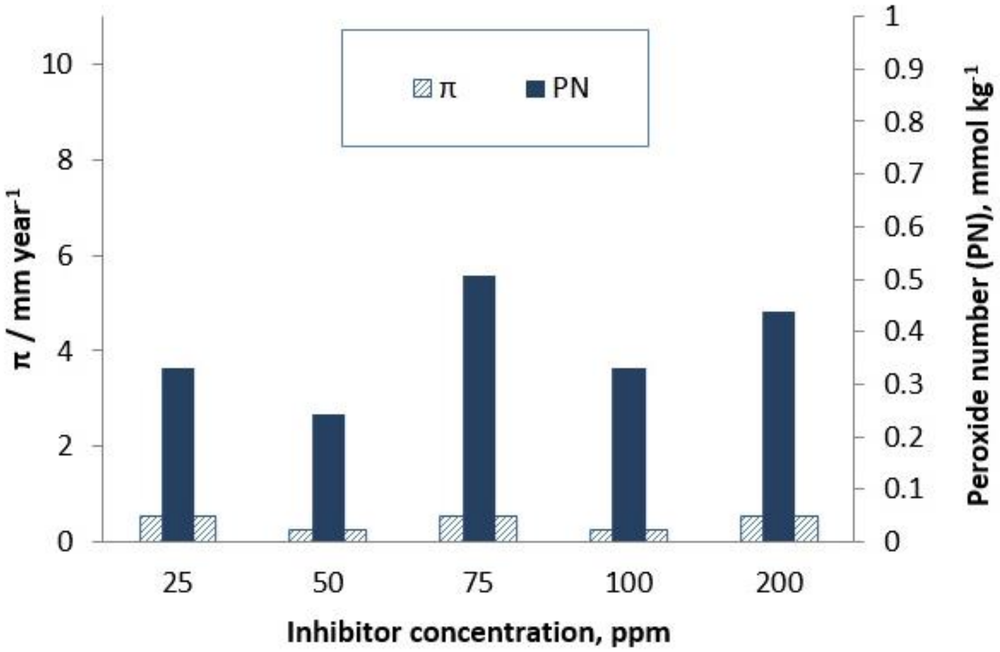

Figure 3. Corrosion rate and peroxide number in HC-6 with the addition of 0.25 % EP additive and various concentrations of IRGAMET 39 (24 h oxidation at 100 ±1 °C)

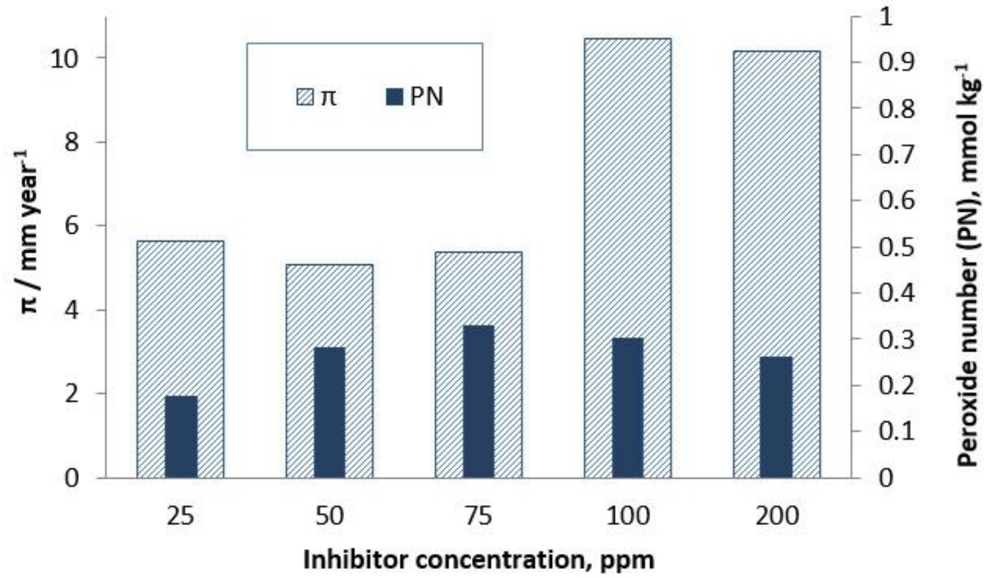

Figure 4. Corrosion rate and peroxide number in HC-6 with the addition of 0.25 % EP additive and various concentrations of RC 4220 (24 h oxidation at 100±1 °C)

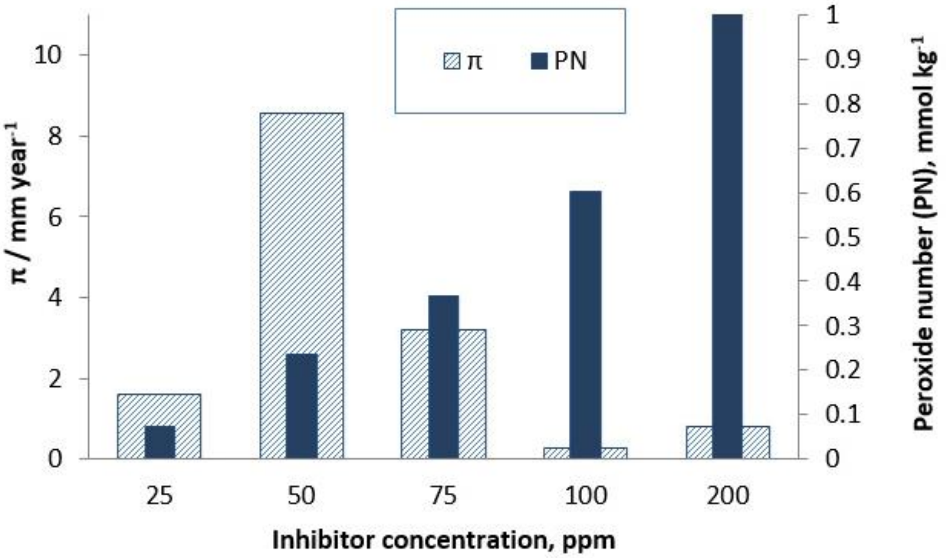

Figure 5. Corrosion rate and peroxide number in HC-6 with the addition of 0.25% EP additive and various concentrations of RC 8210 (24 h oxidation at 100±1 °C)

4. CONCLUSION

The aim of this paper was to evaluate the influence of a sulfur-based EP additive on the corrosivity and oxidative stability of hydrocracked base oil HC-6 used to obtain lubricants and to determine which of the tested inhibitors is the most effective. Based on the obtained results the following conclusions can be made.

1. The tested EP additive has a negative impact on the corrosivity and oxidative stability of the lubricant.
2. The corrosion inhibitor IRGAMET 39 has shown the highest efficiency in protecting copper from corrosion, compared to the other examined inhibitors.
3. In terms of oxidative stability of the investigated lubricant, the RC 8210 inhibitor exhibited the worst results, showing the increase in peroxide number with the increase in concentration. The lubricant has shown the best oxidative stability in the presence of RC 4220, but this inhibitor is the worst in protecting copper from corrosion.

Overall, the inhibitor IRGAMET 39 is the most effective, because it has shown excellent protective properties against corrosion with minimal impact on the oxidative stability of the lubricant.

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Ispitivanje korozije bakarne trake u hidrokrekovanom baznom ulju u prisustvu različitih inhibitora

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Izvod

U ovom radu ispitivana je korozija bakra u hidrokrekovanom baznom ulju HC-6 u prisustvu različitih koncentracija aditiva za ekstremno visoke pritiske (EP aditiv). EP aditivi se koriste za smanjenje habanja u industrijskim primenama, pod uslovima velikog opterećenja. Budući da je većina ovih aditiva na bazi sumpora čija jedinjenja mogu biti korozivna na visokim temperaturama, njihova upotreba dovodi do korozije nekih materijala. Da bi se sprečila korozija bakra u baznom ulju sa EP aditivom dodana su tri komercijalna inhibitora korozije. Po hemijskom sastavu inhibitor RC 8210 je derivat dimerkaptotiadiazola, RC 4220 sintetički neutralan kalcijum sulfonat, a IRGAMET 39 je derivat tolutriazola. Efikasnost inhibitora praćena je u skladu sa stndardnom ASTM D-130 metodom, gravimetrijskom metodom, a oksidaciona stabilnost baznog ulja praćena je određivanjem vrednosti peroksidnog broja. Oksidacija je trajala 3 i 24 časa na 100°C ± 1°C. Ispitivanje je pokazalo da je IRGAMET 39 najefikasniji inhibitor u prisustvu EP aditiva u oba ispitivana vremena oksidacije.

Ključne reči: ASTM D-130; bakarni kuponi; maziva ulja; EP aditiv