

The research of anti corrosive properties of various compositions on samples of standard metals

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In this proceeding, the compositions of the T-30 turbine oil with liquid rubber. Co. Ni. Zn. Mg and Ba salts of the natural petroleum acids (NPA) boiling in the range 220–340°C and nitro compounds which are produced on the basis of C₁₄H₂₈ α-olefins have been investigated in different ratio and contents as metalworking fluids (MWF). The physical and chemical properties of the produced compositions have been studied, and the morphology of inhibited carbon steel surface was analyzed and characterized by using Fourier transform infrared (FTIR) and scanning electron microscopy (SEM) techniques. The effect of the temperature on the inhibition efficiency and thermodynamic parameters have also been reported. The thermal analyzer was used for the thermogravimetric analysis (TG/DTA) of the prepared MWF. The experiments have been operated with different concentrations of the inhibitor on the steel plates in condensation and environment phases in the experiment chamber.

Keywords: metalworking fluids and lubricants, corrosion, liquid rubber, natural petroleum acids.

INTRODUCTION

Corrosion is defined as the deterioration of a material due to reaction with its environment. Regardless of their operation conditions, metal constructions show the thermodynamically unstable state as they react with the aggressive part of the environment and corrode. This harmful attack can cause the defect in the mechanical, physical and chemical properties of metal^{1–3}. Therefore, protection methods should be developed to reduce the damage effects of the metal. Inhibitors have very wide application in a variety of industrial applications such as cooling systems, refinery units, pipelines, chemicals, oil and gas production units, boilers and water processing, paints, pigments, lubricants, etc.^{4–8} Organic inhibitors are very effective way for the reducing of corrosion of metals and alloys due to generate a thin film that controls and prevents access to corrosive agents to the metal surface. The inhibitory effect of an organic inhibitor is reinforced by the presence of hetero adsorption active centers such as sulphur (S), nitrogen (N) and oxygen (O) atoms in its molecule^{9–11}. In addition to the molecular electronic structure with a number of these active centers, the molecular size, the mode of adsorption, the formation of metallic complexes, and the projected area of the inhibitor on the metallic surface also affect the efficiency of inhibition^{12–14}.

In recent years, some experimental studies have been done to describe the corrosion and electrochemical behavior of stainless steel in concrete environment^{15–18}. The widespread investigation of the inhibitors in the metalworking fluids is based on its economic efficiency and simple structure of application technology. The addition of a small amount of an inhibiting substance to the aggressive system is enough for protecting the metal without any changes to the current technical system^{19–21}. The purpose of the work is to synthesize multifunctional nitrogen containing corrosion inhibitors with different molecular masses on the basis of local feedstocks with enough reserves and to prepare high – performance MWF by using them as additives.

In the given proceeding, the solution of the composition of inhibitor that is obtained by the complex of the Co, Ni, Zn, Mg və Ba metal salts of the natural petroleum acids with nitro compounds and liquid rubber in T-30 oil has been applied as a corrosion protector. The compounds those have inhibitor properties in the prepared MWF mainly consist of 3 components: metal salts of natural petroleum acid, aliphatic based nitro compounds and liquid rubber.

EXPERIMENTAL

The metal (Co, Ni, Zn, Mg, Ba) salts of technical petroleum acids which are separated from Azerbaijan kerosene and diesel fractions and is soluble in organic solvents have been synthesized by the general methods. The NPA (It is obtained from the refinery of the Azerbaijan petroleum in the “Merichem” (USA) unit of the Baku Oil Refinery) which is used in the process has been obtained by the dearomatization of the diesel fraction with the boiling range of 220–340°C. The dearomatization process has been operated with the extraction method. N-methyl pyrrolidone has been used as an extract. The physical and chemical properties of the fraction which is obtained from the extraction process were like this: $\rho_4^{20} = 0.8294 \text{ g/cm}^3$; $n_d^{20} = 1.4677$; $M_b = 187$; freezing point = -51°C ; boiling point = 220–340°C. kinematic viscosity at 20°C $\sim 5.52 \text{ mm}^2/\text{sec}$. total sulfur content $\sim 0.03\%$. the amount of aromatic hydrocarbons $\sim 1\%$ (by mass).

The composition of the solution which contains synthesized the metal salts of the NPA and nitro compound with the polybutadiene based liquid rubber has been prepared and tested on steel plates. The liquid rubber which is used in the process has been synthesized on the basis of stereo regular structured 1.4 cis – polybutadiene (low molecular weight, $M_r \sim 1800\text{--}3000$), which has a wide range of industrial application (micro structure: 1.4 cis-75~80%; 1.4 trans-18~22%; 1.2-units-2~7%. number of units~80). The cis- position of the methyl groups in liquid rubber facilitates the adsorption on the metal surface^{22–23}. Nitro compound is synthesized by using treat-

ing $C_{14}H_{28}$ which is produced by the oligomerization of ethylene with nitro compound with nitric acid in optimum condition (sodium nitrite is used as an initiator). The amount of the active components was 1:1:1 (in grams). "T-30" turbine oil has been used as a solvent.

The physical and chemical properties of the composition of inhibitor have been investigated and its content has been proved by IR spectroscopy method²⁴⁻²⁵.

Name of the samples

I. T-30 oil + Co salt of the NPA + Nitro compound+liquid rubber;

II. T-30 oil + Ni salt of the NPA + Nitro compound+liquid rubber;

III. T-30 oil + Zn salt of the NPA + Nitro compound+liquid rubber;

IV. T-30 oil + Mg salt of the NPA + Nitro compound+liquid rubber;

V. T-30 oil + Ba salt of the NPA + Nitro compound+liquid rubber.

In order to determine the identity of the conservation liquid. 5 different points have been chosen on given sample and IR spectra have been evaluated²⁶ (Fig. 1.1. – Fig. 1.6.). The following wavelength has been obtained by the spectral analysis of the conservation content of the inhibitor (Co salt of NPA + Nitro compound + liquid rubber) which shows the maximum efficiency:

The content of the composition has been tested by IR-Furye LUMOS (BRUKER) microscope in the range of 600–4000 cm^{-1} wavelength. The deformation (1376–1457 cm^{-1}) and valence (2921–2950 cm^{-1}) oscillations of structure groups (CH_3 and CH_2) which belong to NPA are observed in the spectrum of the approved sample. At the same time, the resonance signals for C – H bond and valence oscillations for C – NO_2 group are seen at 723 cm^{-1} and 1558 cm^{-1} wavelength, respectively. The comparison of the IR spectrum of all 5 points on the sample shows that they are practically the same which

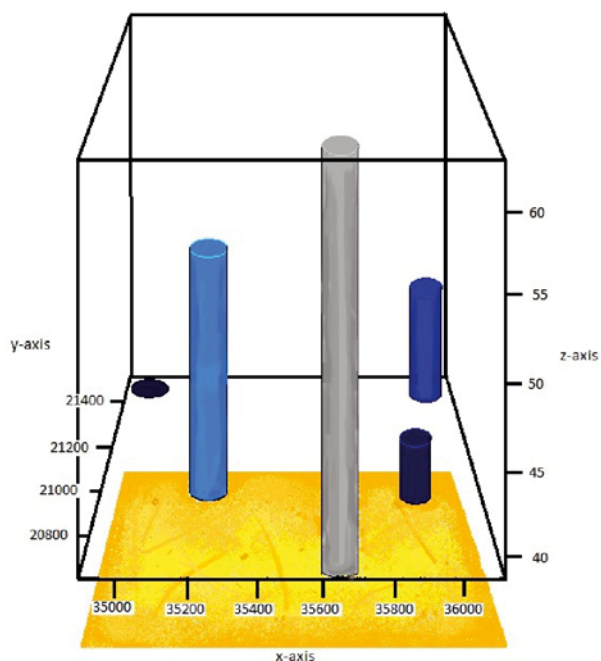


Figure 1.1. The microscopic view of 5 selected points in sample

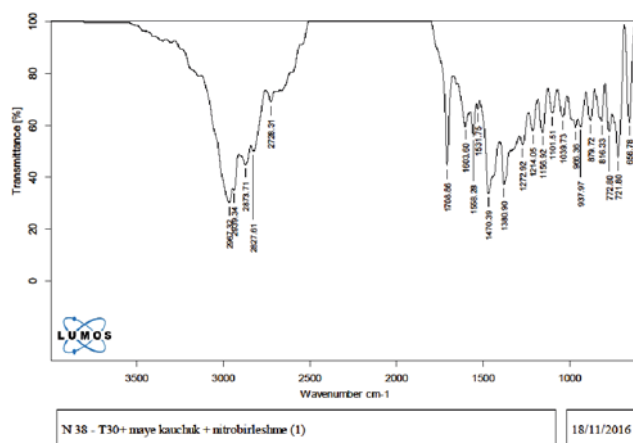


Figure 1.2. IR spectra of the 1st point

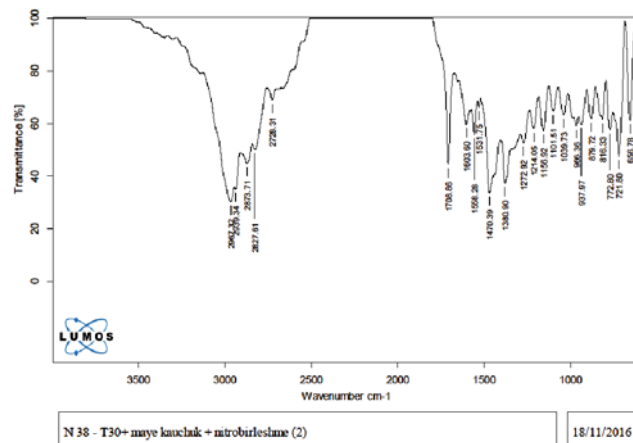


Figure 1.3. IR spectra of the 2nd point

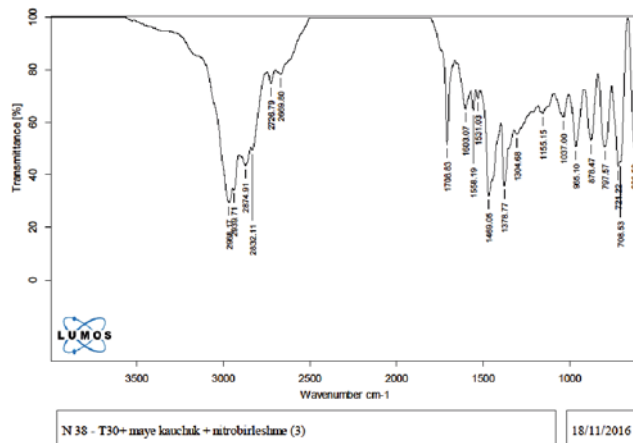


Figure 1.4. IR spectra of the 3rd point

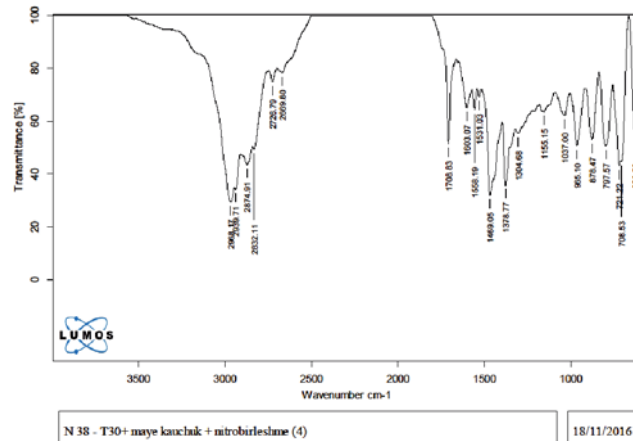


Figure 1.5. IR spectra of the 4th point

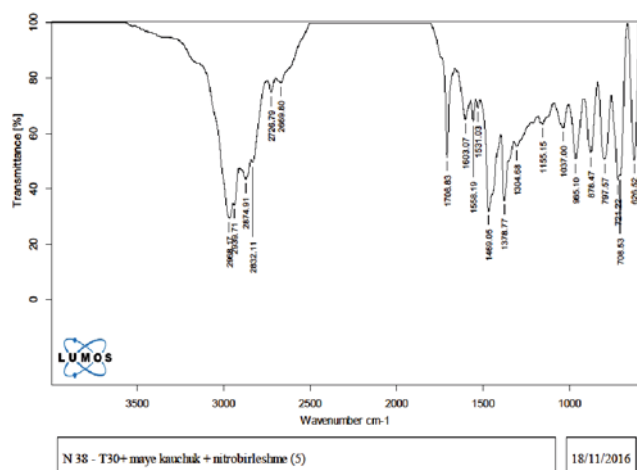


Figure 1.6. IR spectra of the 5th point

proves that the conservation materials are distributed on the metal surface homogeneously.

STA-449 F3. NETZSCH thermal analyzer was used for the thermogravimetric analysis (TG/DTA – change in mass depending on the temperature and differential thermal analysis) of the prepared metalworking fluids (Fig. 2). The processes have been operated by using nitrogen gas flow and by increasing the temperature by 20°C per minute. The evaluation of thermal analysis of Sample 5 (Table 1) shows that there is no chemical change in the STA (Simultaneous Thermal Analysis) curve. the peak at 360°C coincides with the complete disintegration of the composition. The curve for the mass loss is observed in the TG (Thermogravimetry) curve which proves the homogeneity of the composition. The component that has less thermal stability in the studied MWF is nitro compound. Hence. nitro compounds are decomposed at temperatures above 600°C. then the metal salts of the NPA and liquid rubber are decomposed

respectively. The dependence of the change in the mass loss of the temperature is given in Table 2.

Because of the uniform distribution of the components in the T-30 oil medium and thermostability property of the metal salts of the NPA. metalworking fluids can be utilized for a long time without losing quality.

Table 2. Change in the mass loss

Temperature [°C]	Mass loss [mg]	Mass loss [%]
120	0	–
140	0.2	1.48
160	0.4	2.96
180	0.6	4.44
200	1.0	7.41
220	1.4	10.37
240	2.6	19.25
260	4.0	29.26
280	6.0	44.44
300	8.6	63.70
320	11.0	81.48
340	11.4	84.44
360	11.4	84.44
380	11.6	85.92
400	11.8	87.64
420	12.0	88.88
440	12.0	88.88
460	12.2	90.37

The specific contact method has been used in order to determine the resistivity (specific conductance) of the compositions. The measurement process has been operated by the E6-13A teraohmmeter. The measurement places are cleaned with alcohol before each process and the distance between electrodes is determined. Then. the substance is poured until the area between electrodes is filled and the resistivity is measured. The resistivity is calculated according to $r = \rho r^2/d$ equation and the conductance is determined with the following equation: $\sigma = 1/r$. The physical components

Table 1. The main physical properties of the metalworking fluids

Name of the property	Name of the device	ASTM	Samples				
			I	II	III	IV	V
Freezing point °C	Stanhope Seta	ASTM D2386	-35	-30	-25	-25	-25
Viscosity mm ² /s. 40°C	TW4000	ASTM D445	60.88	56.47	61.96	66.93	53.99
Viscosity. mm ² /s. 100°C	TW4000	ASTM D445	8.31	7.98	8.12	7.54	7.63
Density. g/cm ³	DMA 4500 M	D5002	0.9072	0.9112	0.9136	0.9093	0.9090

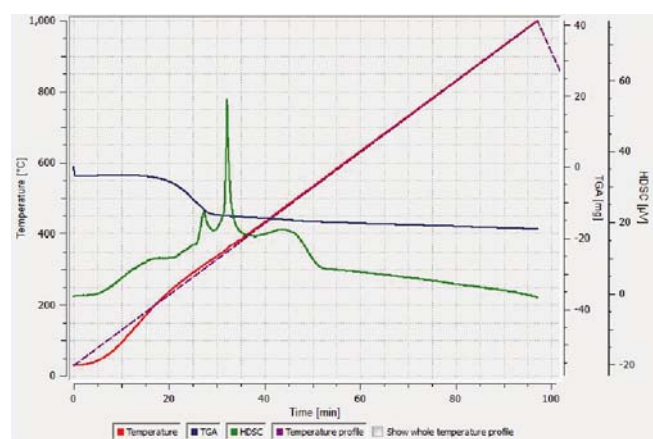


Figure 2. TG/DTA curves of the Sample 5

in the formulae are following: r-resistance. p-constant. σ -conductance. r-radius of the measurement electrode. d-distance between electrodes. The determined electrical conductivity values of the samples are shown in Table 3.

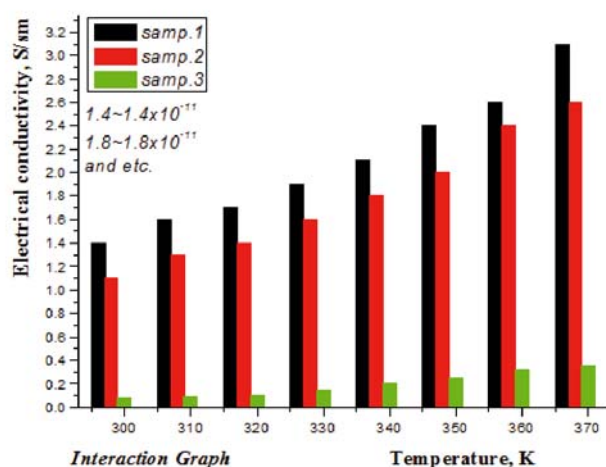
The graph (Fig. 3.) showing the change of the electrical conductivity of the metalworking fluids due to temperature is given below:

As can be seen from the tables (Table 3 and 4). there is a direct dependence between the electrical conductance and the corrosion protection effect of the metalworking fluids; the higher electrical conductance. the greater corrosion protection effect.

Obtained composition has been tested as MWF on steel plates by dissolving T-30 oil (5%, 7% and 10%).

Table 3. Change in the electrical conductivity due to temperature

№	Temperature. [K]	Special resistance ρ . [Om·m]	Electrical conductivity σ . [S/sm]	№	Special resistan. ρ . [Om·m]	Electr. cond. σ . [S/sm]	№	Special resistan. ρ . [Om·m]	Electr. cond. σ . [S/sm]
1	298	$6.9 \cdot 10^8$	$1.4 \cdot 10^{-11}$	2	$8.4 \cdot 10^8$	$1.1 \cdot 10^{-11}$	3	$1.08 \cdot 10^{10}$	$9.2 \cdot 10^{-13}$
	303	$6 \cdot 10^8$	$1.6 \cdot 10^{-11}$		$7.9 \cdot 10^8$	$1.3 \cdot 10^{-11}$		$1.04 \cdot 10^{10}$	$9.6 \cdot 10^{-13}$
	308	$6 \cdot 10^8$	$1.6 \cdot 10^{-11}$		$7.4 \cdot 10^8$	$1.4 \cdot 10^{-11}$		$1.04 \cdot 10^{10}$	$9.6 \cdot 10^{-13}$
	313	$5.6 \cdot 10^8$	$1.7 \cdot 10^{-11}$		$6.9 \cdot 10^8$	$1.4 \cdot 10^{-11}$		$1 \cdot 10^{10}$	$1 \cdot 10^{-12}$
	318	$5.6 \cdot 10^8$	$1.7 \cdot 10^{-11}$		$6.5 \cdot 10^8$	$1.5 \cdot 10^{-11}$		$9.2 \cdot 10^9$	$1.1 \cdot 10^{-12}$
	323	$5.2 \cdot 10^8$	$1.9 \cdot 10^{-11}$		$6.1 \cdot 10^8$	$1.6 \cdot 10^{-11}$		$8 \cdot 10^9$	$1.3 \cdot 10^{-12}$
	328	$5.2 \cdot 10^8$	$1.9 \cdot 10^{-11}$		$5.9 \cdot 10^8$	$1.7 \cdot 10^{-11}$		$6.8 \cdot 10^9$	$1.5 \cdot 10^{-12}$
	333	$4.8 \cdot 10^8$	$2.1 \cdot 10^{-11}$		$5.7 \cdot 10^8$	$1.8 \cdot 10^{-11}$		$6 \cdot 10^9$	$1.7 \cdot 10^{-12}$
	338	$4.6 \cdot 10^8$	$2.2 \cdot 10^{-11}$		$5.3 \cdot 10^8$	$1.9 \cdot 10^{-11}$		$5 \cdot 10^9$	$2 \cdot 10^{-12}$
	343	$4.2 \cdot 10^8$	$2.4 \cdot 10^{-11}$		$4.9 \cdot 10^8$	$2 \cdot 10^{-11}$		$4 \cdot 10^9$	$2.5 \cdot 10^{-12}$
	348	$3.8 \cdot 10^8$	$2.6 \cdot 10^{-11}$		$4.6 \cdot 10^8$	$2.2 \cdot 10^{-11}$		$3.6 \cdot 10^9$	$2.8 \cdot 10^{-12}$
	353	$3.8 \cdot 10^8$	$2.6 \cdot 10^{-11}$		$4.1 \cdot 10^8$	$2.4 \cdot 10^{-11}$		$3.2 \cdot 10^9$	$3.1 \cdot 10^{-12}$
	358	$3.2 \cdot 10^8$	$3.1 \cdot 10^{-11}$		$3.8 \cdot 10^8$	$2.6 \cdot 10^{-11}$		$2.8 \cdot 10^9$	$1.3 \cdot 10^{-12}$

**Figure 3.** The graph of electrical conductivity against temperature

The experiments were carried out with DC01 (CR4) carbon steel specimens. The main chemical composition (wt%) of the carbon steel was listed as follows: C%~0.07; Si%~0.01; Mn%~0.2–0.35; Ni%~0.06; S%~0.025; P%~0.02; Cr%~0.03; Al%~0.02–0.07; Cu%~0.06; Fe balance. The test sample size was 150 x 100 x 1 mm. The carbon steel plates were prepared, degreased and cleaned with deionized water and alcohol.

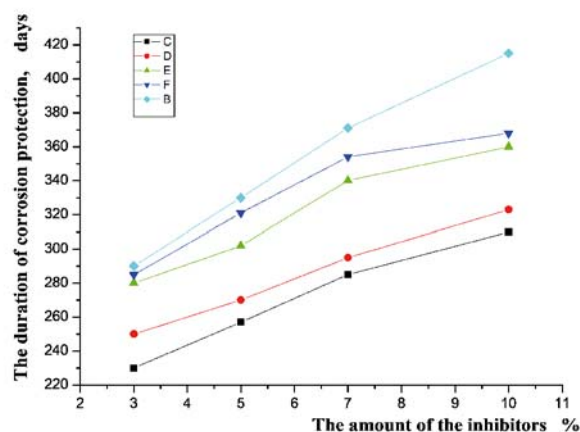
The experiment has been operated according to the current standards in the chamber called «CORROSIONBOX-1000E» which is considered as a modern technological device. The process has been carried out in two phases: condensation and atmospheric phase. Electronic appliances have been used in order to adjust the standard parameters for obtaining sustainable trials in the experiment chamber. For the condensation phase, these parameters are the temperature of the chamber and the duration of the trial. For the environmental phase, these are the temperature of the moisturizing agent and the chamber and the duration of the trial. The duration of the trial was in the interval of 1 minute – 9999 hours and the temperature of the chamber was 20–50°C for the condensation phase, whereas the temperature of the moisturizing agent was 20–80°C and the remaining parameters were the same for the environmental phase.

RESULTS AND DISCUSSION

As can be clearly seen from the table, metal sheets were protected 31 and 56 days by using T-30 turbine oil and liquid rubber respectively. However, these pointers were higher when the inhibitor was used. When 7% concentration of the inhibitor (Co salt of NPA + nitro compound + liquid rubber) was used, in the condensation phase, the protection of the metal sheets from the corrosion lasted 301 days. However, in environmental phase, it was 384 days. In 10% concentration, it was 322 and 401 days, respectively. At the same time, the corrosion protection efficiency of the conservative liquids those have been produced on the basis of the complex of the metal salts (Ni, Zn, Mg and Ba) of NPA with nitro compounds was high in comparison to current demand.

The dependence graph (Fig. 4.) of the trial results of composition type metalworking fluids on the concentration of the inhibitor is shown below:

As it is clear from the Figure 4, the highest result was obtained from the metalworking fluid which is synthesized by the composition of Co salt of the NPA (curve B).



B – T-30 oil + Liquid rubber + Co salt of the NPA+ Nitro compound
 F – T-30 oil + Liquid rubber + Ni salt of the NPA+ Nitro compound
 E – T-30 oil + Liquid rubber + Zn salt of the NPA+ Nitro compound
 D – T-30 oil + Liquid rubber + Mg salt of the NPA+ Nitro compound
 C – T-30 oil + Liquid rubber + Ba salt of the NPA+ Nitro compound

Figure 4. The graphical illustration of the trial results of the metal salts of NPA as composition type metalworking fluids

Table 4. The results of the trials of synthesized salts as metalworking fluids both individually and as compositions

№	The solutions of the compositions in the "T-30" oil		The duration of corrosion protection. [days]		
			condensation phase	atmospheric phase	
	Content	The amount of the components [%]			
inhibitor		solution			
1.	T-30 oil + Liquid rubber	–	–	31	56
2.	Co salt of the NPA Liquid rubber	5 5	10	138	219
3.	Co salt of the NPA Nitro compound Liquid rubber	1.66 1.66 1.66	5	228	317
4.	Co salt of the NPA Nitro compound Liquid rubber	2.33 2.33 2.34	7	301	384
5.	Co salt of the NPA Nitro compound Liquid rubber	3.33 3.33 3.34	10	322	401
6.	Ni salt of the NPA Liquid rubber	5 5	10	95	197
7.	Ni salt of the NPA Nitro compound Liquid rubber	2.33 2.33 2.34	7	267	311
8.	Ni salt of the NPA Nitro compound Liquid rubber	3.33 3.33 3.34	10	291	350
9.	Zn salt of the NPA Nitro compound Liquid rubber	3.3.3 3.3.3 3.3.4	10	263	326
10.	Mg salt of the NPA Nitro compound Liquid rubber	3.33 3.33 3.34	10	297	353
11.	Ba salt of the NPA Nitro compound Liquid rubber	3.33 3.33 3.34	10	289	346

The long lasting influence of the aggressive environment on the metallic surface degrades the protective layer. as a consequence. the coating loses its function at the certain level of the experience.

As it is obvious from the Table 5. the lubricant which is produced when 10% of technical paraffin²⁷ is added to the MWF (Sample-I) which has the highest efficiency in "corrosionbox" protects metal plates for 356 and 423 days in condensation and environmental phase. respectively. At the same time. the lubricants which are prepared by other metalworking fluids show high anti – corrosion effect during the trial.

Scanning Electron Microscopy Analysis (SEM)

Scanning Electron Microscope (SEM: S-3400N HITACHI) morphologies the steel plates in the corrosion box in the absence and presence of MWF were shown in Figure 5.1–5.4 respectively. Figure 5.1 shows the clean surface of steel sample before immersing in MWF. This figure reveals the homogeneity of the surface. Figure 5.2 shows the general appearance of mild steel without inhibitor (only T-30 oil). It can be seen from the figure that the surface of steel plates was extremely damaged in the absence of the inhibitor. Figure 5.3 and 5.4 reflect the SEM morphology of the steel specimen at lower concentration (5%) and higher concentration (10%) of

the MWF. From the figures it is clear that at low concentration (5%) of the MWF only few constituents cover the surface which are not enough to afford a protective layer and hence dissolution of mild steel is observed to some extent whereas at higher concentration (10%) the constituents present are large enough to form a protective layer regarding the dissolution of steel plates.

As a result of the experiments. it has been revealed that. the inhibitor properties of the compositions of mineral oils with these kinds of mixture of complexes are more effective than the individual compositions with the same concentration those are prepared by adding salt and nitro compounds to the mineral oils separately. It can be explained by the superior ability of chemisorption on the metal surface and producing stronger coating for protection from corrosion of these complexes. On the other hand. these compounds are stronger ligands than the water that is why they can substitute water molecules from the surface of the metal and create strong protection layer.

CONCLUSION

As can be seen from the table which indicates the results of the experiments. the manufactured MWF may be used in order to protect the metals from corrosion

Table 5. The trial results of the lubricants

№	MWF 90% + paraffin 10%	The total amount of the inhibitor in the sample [%]	The duration of corrosion protection. [days]	
			condensation phase	environmental phase
1	I + paraffin	10	356	423
2	II + paraffin	10	341	403
3	III + paraffin	10	251	312
4	IV + paraffin	10	332	378
5	V + paraffin	10	304	352



Fig. 5.1 before the immersion test.

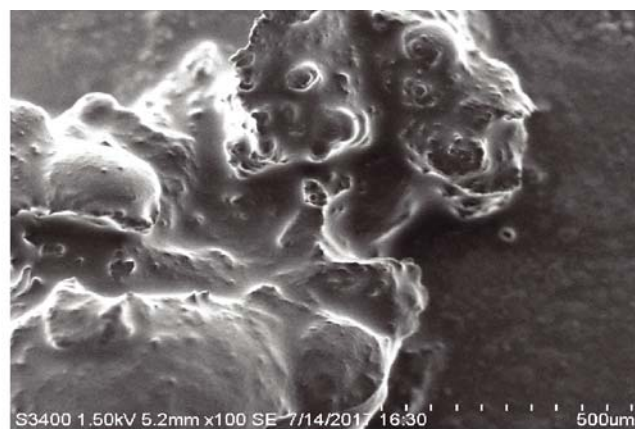


Fig. 5.2 after the immersion test (without inhibitor)



Fig. 5.3 after the immersion test (at 5% concent.)



Fig. 5.4 after the immersion test (at 10% concent.)

Figure 5.1–5.4. SEM Images of MWF specimens; before and after the immersion test

for industrial purposes. The components those have been used for producing MWF are cost effective and environmentally – friendly. on the other hand, they have simple manufacturing technology and rich feedstock. T-30 oil. additives which are used as inhibitors and liquid rubber which are used in the reaction medium are produced on the basis of the feedstock which has enough reserves. So, the comparative analysis of the results reveals that the usage of the compositions of the metal salts of natural petroleum acids with nitro compounds as inhibitors have a practical importance for producing more effective MWF.

It has been revealed that the metalworking fluids that are produced by the composition of the synthesized inhibitor and nitro compound in the presence of liquid rubber has a great protection against corrosion than their individual usages. meets the requirements and has a great practical importance.

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