

COKE QUENCHING PLENUM EQUIPMENT CORROSION AND ITS DEPENDENTS ON THE QUALITY OF THE BIOCHEMICALLY TREATED WATER OF THE COKE-CHEMICAL PRODUCTION

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Abstract. Steel corrosion processes that occur due to the effects of the biochemically treated water in coke quenching plenums have been studied. Model investigations into the processes of the corrosion failure of carbon St. 3 steel and alloyed 18Kh1MF steel were carried out to study the behavior of the metal exposed to the action of the primary but treated water used for the coke quenching after the metal is heated to 373 K and 773 K. Different types of the corrosion that results from the contact of the carbon steel and alloy steel with the sodium hydroxide biochemically treated water of the coke-chemical production have been described. It was shown that the corrosion failure of the steels results from the formation of the films of hydrated iron oxides that appear in all the test media and these films show different behavior of adhesion to specimen surfaces. It was proved that the water treatment results in a certain decrease of the values of the mass and in-depth corrosion factors for St.3 and 12Kh1MF steels, however it fails to produce an essential effect when the steel is in constant touch with hot water.

Key words: Corrosion, Coke Quenching, Iron Oxide, Carbon Steel, Gravimetry, Polarization Dependence.

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1. Introduction

The coke quenching or cooling is a mandatory stage of the coke production at coke-chemical companies and it defines to a great extent its output and quality.¹ The coke is pumped from coke-kilns to hot coke cars to perform the primary quenching in order to eliminate a spontaneous ignition of the products exposed to the action of the air. In this case the coke temperature is decreased from 1273–1323 K to 273 K, and after that the coke is sent to quenching towers in which the coke temperature is dropped to 473–523 K.²

The hot coke cars are made of carbon steel of the St.3 grade. This steel is inexpensive and has satisfactory mechanical properties. The major drawback of this steel is its unsatisfactory corrosion resistance, especially in conditions of the insufficient replenishment of the consumed circulating water that results in the need for the multiple use of the effluent water with increased mineralization by the company. An intensive corrosion deterioration of the St.3 steel results in that the hot coke cars are subject to the running repairs after each month of their use.

The steel deterioration during the coke quenching occurs due to a high-temperature oxidation of the heated metal surface by oxygen, the corrosion in the mineralized circulating water (especially under closed cycle conditions with the reuse of biochemically treated water of a high salt content), and the mechanical wear caused by the abrasive action of the loaded and unloaded coke.³ In this particular case, the rate of the corrosion deterioration of the cars can be decreased by taking appropriate measures that include the selection of steel grades with an increased corrosion-resistance⁴ and the treatment of

circulating water carried out to decrease the water aggressiveness.⁵⁻⁸ At the moment, coke quenching car elements are manufactured from carbon steels St.3 of a standard quality. The steels of this type are used for the production of hot-rolled products operating at a relatively low mechanical stresses and temperatures that dramatically limit their service life when used for coke quenching plants.⁹ These steels can be replaced by heat-resistant alloyed steels that can operate for a long time at elevated temperatures with no essential degradation of mechanical properties. These materials include the alloyed 12Kh1MF steel used for the manufacturing of the elements for steam plants used by the power-plant industry. This steel can operate for a long time when exposed to the action of hot water and steam. However, the real opportunity for the use of this steel can only be assessed after subjecting it to corrosion resistance tests when the metal is cooled by circulating coke quenching waters.

The purpose of this research was to study the processes of the corrosion deterioration of the steel of a different grade exposed to the action of biochemically treated waters simulating the conditions of the operation of hot-coke cars.

Tasks of the research:

- studying the behavior of St.3 and 12Kh1MF steels heated to 373 K with their quenching in the initial water

and in the alkaline-treated water when the water vapor acts as the corrosion environment, simulating thus the operation conditions of the elements of the lower part of the walls and the bottom of the hot coke receiving cars that are in touch with the cooled coke;

- studying the behavior of St.3 and 12Kh1MF steels heated to 773 K with their quenching in the initial water and in the alkaline-treated water when the water vapor acts as the corrosion environment, simulating thus the operation conditions of the elements of the upper and the middle parts of the walls of the hot coke receiving cars that are in touch with the hot and partially-cooled coke;

- taking the polarization measurements of St.3 and 12Kh1MF steels in the hot initial water and alkaline-treated water when the solutions act as the electrochemical corrosion environment, simulating thus the operation conditions of the bottom of the hot coke-receiving cars with the cooling water running to the collector.

2. Experimental

The research was done using the steel specimens of St.3 and 12Kh1MF grades. Table 1 gives the chemical composition of the steels.

Table 1. Composition of steel specimens

Steel	Content of the element, %										
	C	Si	Mn	Ni	Cu	Cr	As	S	P	Mo	V
St.3	0.16	0.07	0.50	0.25	0.22	0.23	0.06	0.05	0.04	–	–
12Kh1MF	0.11	0.25	0.51	–	–	1.07	–	0.006	0.005	0.3	0.24

The specimens of biochemically treated coke quenching water initial quenching water (IQW) and circulating sodium hydroxide treated water (TQW) were used as the corrosion environment. The chemical composition of corrosion environment was defined using the methods of the titrimetric analysis;¹⁰⁻²² a more detailed information is given in Table 2.

The investigation consisted in the simulation of the operation conditions of the hot coke – receiving cars. The adopted simulation scheme consisted in the following: the specimens have been heated for 5 minutes in the muffle furnace at the temperatures of 373 and

773 K and then the specimens were placed into the tanks filled with IQW and TQW and were held there for 2 minutes. On the whole, fifty (50) heating and cooling test cycles were conducted and the specimens were weighed after each five cycles. The weigh data were used to calculate a change in the steel mass Δm that enabled the estimation of the extent of their wearing during the operation. The specimens were prepared by subjecting them to the grinding with abrasive flap – wheels with the subsequent reduction of abrasive grain sizes and degreasing with petrol, synthetic detergent solution and sodium carbonate suspension followed by the

rinsing in the running water and in the distilled water. The specimens were weighed using the analytical scales.

The corrosion products were removed using the chemical method, *i.e.* the H_2SO_4 solution in the volume of 100 cm^3 ($\rho = 1.84\text{ g/cm}^3$) and 5 g of CH_4N_2S in 1000 cm^3 of water.

After the removal of corrosion products, we calculated the mass k_m and in-depth k_h corrosion factor and estimated the steel resistance point. Electrochemical measurements were taken using the MTech PGP-550S potentiostat-galvanostat with the software provided by the developer. Polarization measurements were taken in the electrochemical cell with separated cathode and anode spaces. The working electrode was represented by test steel specimens and the auxiliary electrode was the steel of a Kh18N10T grade. The working electrode was prepared by subjecting it to the grinding with abrasive flap – wheels and subsequent abrasive grain size reduction and degreasing with petrol, synthetic detergent solution and sodium carbonate suspension followed by the rinsing in the running water and in the distilled water. Electrode potentials were measured using the saturated chloride silver reference electrode of an EVL-1M1 type that was arranged in a separate vessel and connected to the cell by the two-arm electrolytic key with the Lugin capillary.¹⁰ The obtained potential values were converted to the normal hydrogen electrode scale.

The measurements were taken in the initial water and in treated water at 348 K. The temperature of 348 K corresponds to the temperature of water streaming down to the collector. Hence, the polarization investigations carried out at this t value enabled the simulation of the operating conditions for the bottoms of quenching cars. The thermoregulator PT-049 was used to maintain the preset temperature. Based on the data of polarization measurements we derived the potential E_k and the current density j_k for the steel corrosion and calculated the mass k_m and in-depth k_h corrosion factors.

3. Results and Discussion

3.1. Chemical analysis data obtained for the biochemically treated water

The water composition analysis data are given in Table 2.

Table 2. Chemical composition of the biochemically treated water

Indicator	Mass concentration, mg/dm^3	
	IQW	TQW
Total ammonia ¹¹	5186	1346
Volatile ammonia ¹¹	122.5	1140
Fixed ammonia ¹¹	5063	206
Phenols ¹²	0.23	0.2
Sulfates ¹³	1908	1848
Sulphides ¹⁴	1.33	–
Calcium ^{15,16}	320	170
Thiocyanates ¹⁷	1360	1440
Cyanides ¹⁸	3.1	3.1
Chlorides ¹⁹	10084	11208
Sodium ²⁰	667.7	7241
Iron ²¹	39.8	48.6
Dry residue ²²	19935	25675

The pH factor of initial water is 6.79 and that of the alkaline treated water is 10.32.

3.2. Gravimetric investigations were carried out by heating the specimens to 373 K

Fig. 1 gives the dependence diagrams that reflect the changes in the mass of the specimen heated to 373 K and cooled in IQW. The ordinate axis shows a specific change in the specimen mass per 1 m^2 of the steel surface obtained using the weigh data. The minus sign in front of the values is indicative of a decrease in the specimen mass during the tests and the plus sign is indicative of the gain in the specimen mass.

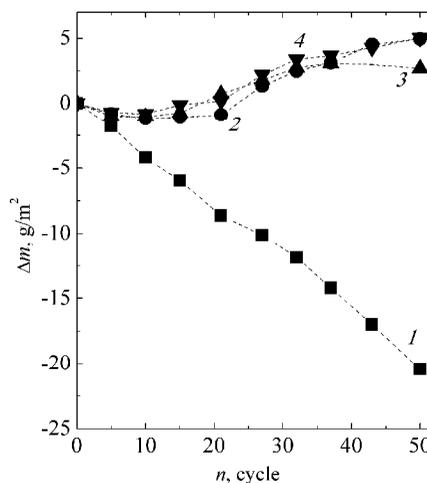
The obtained data allow us to draw the following conclusions. The mass of the steel specimens St.3 is decreased and it is indicative of the bad adhesion of corrosion products to the substrate (Fig. 1, curve 1). The mass of steel specimens 12Kh1MF is increased and it is indicative of the formation of dense films with good adhesion to the substrate (Fig. 1, curve 2). The corrosion of steel specimens immersed into IQW shows a continuous uniform pattern independently of their grade and it is accompanied by the formation of the brown layer of hydrated iron oxides.

After cooling in TQW, the mass of the specimens of all tested steels is increased (Fig. 1, curves 3 and 4) and it is a consequence of the formation of the dense layer of corrosion products. The steel corrosion pattern is

nonuniform and during the first 10 cycles we observed the formation of individual dotted deterioration zones.

Later on, we observed the appearance of brown spots with some black zones on the steel surface (Fig. 2).

Fig. 1. Change in the mass of steel heated to 373 K and cooled in the biochemically treated water:
 1 – IQW, grade St.3; 2 – IQW, grade 12Kh1MF;
 3 – TQW, grade St.3; 4 – TQW, grade 12Kh1MF



a)



b)



c)



d)

Fig. 2. Image of the state of the steel surface after 50 cycles of heating and cooling at 373 K:
 a – St.3, IQW; b – 12Kh1MF, IQW; c – St.3, TQW; d – 12Kh1MF, TQW

3.3. Gravimetric measurements taken during the heating up to 773 K

Gravimetric measurements data obtained for IQW are given in Fig. 3 (curves 1 and 2). The obtained data show that the IQW provides approximately the same rate of a decrease in the specimen mass. The corrosion pattern is uniform and smooth when the steel specimen is cooled in IQW. The layer of products of a grey and black color with the good adhesion to the metal is formed on the steel surface. As the number of test cycles is increased to 30 and more, the cooling of specimens results in the partial cleavage of the layer due to the difference in the coefficients of the thermal expansion of the film and substrate. The St.3 steel is more inclined to the clinker cleavage during the cooling in comparison to the 12Kh1MF grade.

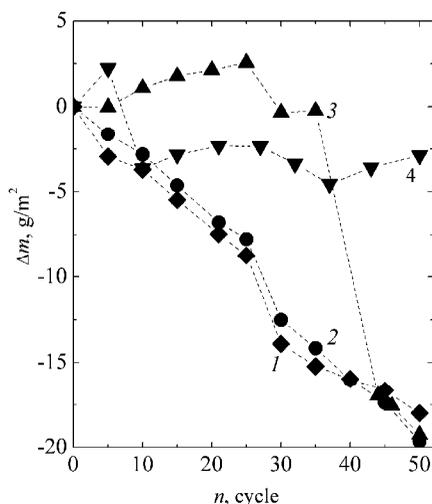


Fig. 3. A change in the steel mass when heated to 773 K with further cooling in the biochemically treated water: 1 – IQW, St.3; 2 – IQW, 12Kh1MF; 3 – TQW, St.3; 4 – TQW, 12Kh1MF

Gravimetric measurements taken in the TQW (Fig. 3, curves 3 and 4) show that the mass of the St.3 specimens is increased after the first 25 test cycles. It is indicative of the formation of the well adherent layer of corrosion products. As the number of cycles is increased the specimen mass is abruptly decreased due to the clinker separation.

The 12Kh1MF specimens show the gain in the mass during the first 5 test cycles. As their number continues to increase, the steel mass is decreased as a result of the film separation during the cooling process. The 12Kh1MF steel specimens show a lower mass loss by 4.5 to 5 times in comparison to the St.3 grade. The corrosion of the steels cooled in TQW shows a uniform pattern with the formed products of a black color.

Based on the data given in Fig. 3, we can draw a conclusion that a decrease in the mass of the specimens of all the steels cooled in the IQW has similar values. The value of a change in the St.3 mass after 50 test cycles is close to that obtained for the specimens cooled in the IQW. The loss in the mass Δm of 12Kh1MF specimens cooled in the IQW after 50 test cycles is equal to 2.53 g/m² in comparison to 19.1 g/m² for the St.3 steel. Images of the state of the steel surface after the tests are shown in Fig. 4.

The values of the mass and in-depth corrosion factors that were calculated using the data of gravimetric measurements are given in Table 3.

The data in Table 3 are indicative of that the steels heated to 373 K and cooled in the IQW and those heated to 773 K and cooled in the TQW are attributed to the group of the materials with low resistance, as for their chemical resistance class (point 9, $k_h > 5$ –10 mm/year). The St.3 and 12Kh1MF steels heated to 773 K and cooled in the IQW are attributed to nonresistant materials (point 10, $k_h > 10$ mm/year).

Table 3. Corrosion rate values obtained for the steels cooled in the IQW and TQW after the 50 test cycles of heating and cooling (the 5 minutes for heating and the 2 minutes for cooling)

№	Environment	St.3		12Kh1MF		Type of corrosion
		Mass corrosion factors k_m , g/(m ² ·h)	In-depth corrosion factors, mm/year	Mass corrosion factors k_m , g/(m ² ·h)	In-depth corrosion factors, mm/year	
1	IQW, 373 K	8.04	9.03	7.86	8.80	Solid uniform
2	TQW, 373 K	6.37	7.13	5.16	5.78	Spots
3	IQW, 773 K	9.00	10.08	11.58	12.97	Solid uniform
4	TQW, 773 K	8.82	9.88	8.37	9.37	Solid uniform

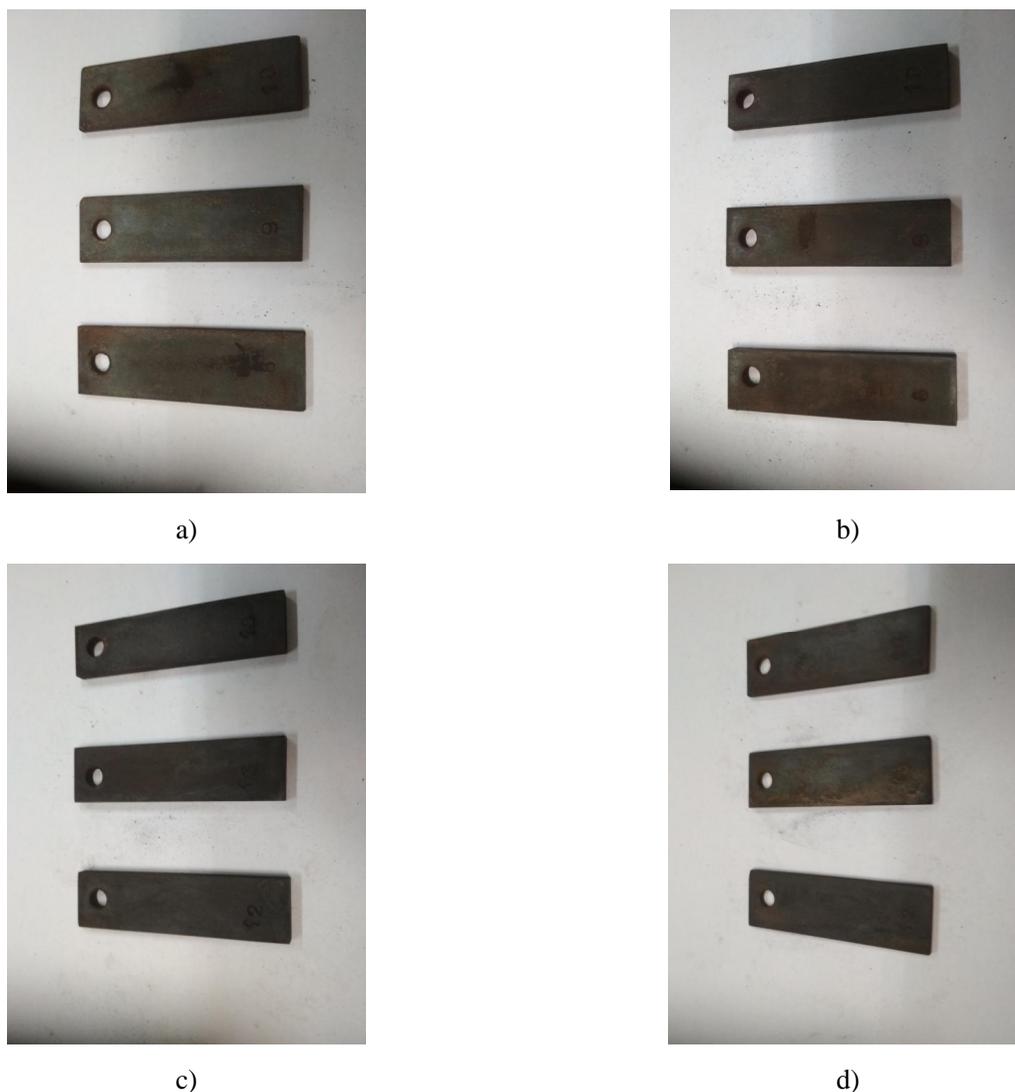


Fig. 4. Image of the state of the steel surface after 50 cycles of heating and cooling at 773 K:
a – St.3, IQW; *b* – 12Kh1MF, IQW; *c* – St.3, TQW; *d* – 12Kh1MF, TQW

3.4. Polarization measurement data

Fig. 5 gives anode polarization dependences obtained for the St.2 and 12Kh1MF steels in the biochemically treated water. The obtained curves allow us to draw a conclusion that the steel corrosion in the initial water develops in an active state due to the availability of a significant amount of chloride ions in the solutions.^{23,24}

In the treated water, the current drop is observed on the anode branch of the curves for the St.3 steel at -0.25 V and for the 12Kh1MF steel at -0.29 . Possibly, this drop is caused by the formation of the films of hydrated iron oxide. However, an efficient steel

passivation fails to occur and it is conditioned by a high content of chloride ions in the solutions.^{25,26}

During the potential sweep from the stationary value we can observe the current spread to the domain of negative values in all the cases and it is indicative of a high efficiency of the cathode process and the availability of the chemical etching of the specimens in the absence of the outer polarization.^{27,28}

Table 4 gives the data obtained during the processing of the plotted curves.

The data given above show that the mass factor of the steel corrosion has the value that is equal to one order of magnitude. It is explained by the fact that the cathode reaction of the corrosion process results in the

recovery of oxygen and this reaction rate is limited by diffusion limitations relating to the oxygen solubility in water and it is the constant value under the given conditions for the studied media. Therefore, the water treatment or the replacement of the steel grade in the given case produces no essential effect. The data given

in Table 4 show that the 12Kh1MF steel corrosion rate in the IQW and TQW exceeds the corrosion rates of the St.3 steel. The biochemically treated water actually has no influence on the St.3 steel corrosion rate and allows us to reduce to a certain extent the 12Kh1MF steel corrosion rate.

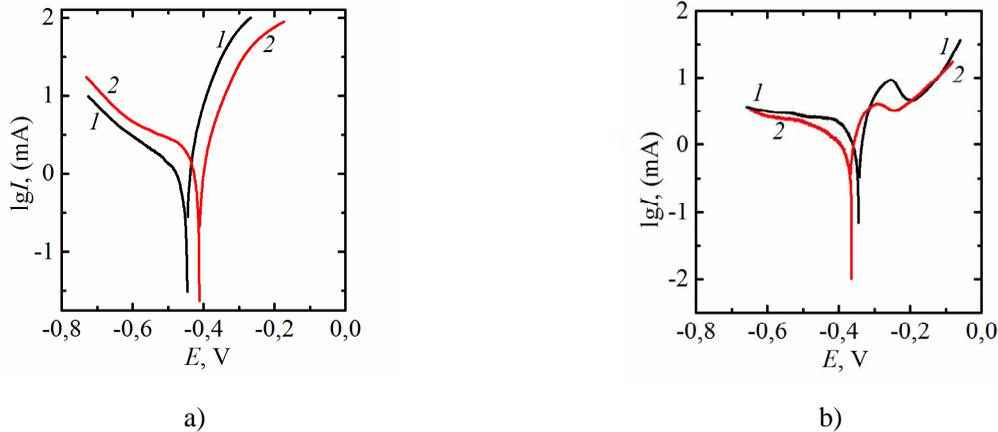


Fig. 5. Anode polarization curves obtained for St.2 and 12Kh1MF steels in the biochemically treated water: a – IQW, St.3 (1); IQW, 12Kh1MF (2); b – TQW, St.3 (1); TQW, 12Kh1MF (2). $v_p = 5$ mV/s

Table 4. Corrosion rate values obtained for the steels cooled in the TQW at 348 K

№	Environment	St.3				12Kh1MF			
		Corrosion current density j_K , mA/cm ²	Corrosion potential E_K , V	Mass corrosion factors k_m , g/(m ² ·h)	In-depth k_h corrosion factor, mm/year	Corrosion current density j_K , mA/cm ²	Corrosion potential E_K , V	Mass corrosion factors k_m , g/(m ² ·h)	In-depth k_h corrosion factors, mm/year
1	IQW, 348 K	0.39	-0.454	0.40	0.49	0.83	-0.410	0.87	0.96
2	TQW, 348 K	0.38	-0.050	0.39	0.44	0.55	-0.371	0.57	0.64

The data given in Tables 3 and 4 show that the alloyed steel demonstrates as a rule lower values of the corrosion rate in comparison to the carbon steel after it is heated to 373 and 773 K and cooled in the quenching water. Exception to the above is when the steels are heated to 773 K and cooled in the primary quenching water and in this case, the 12Kh1MF steel demonstrates a higher rate of the corrosion failure in comparison to St.3 (11.58 and 9.00 g/m²·h, accordingly). These data are important first of all as a basis for the conclusion that the alloyed steel has no special advantages before the carbon steel from the standpoint of corrosion resistance. In most cases, the 12Kh1MF steel demonstrates higher resistance in comparison to the St.3 steel; however simple calculations show that an absolute rate of the alloyed steel corrosion is lower at the worst by 23 % than that of

the carbon steel corrosion. In the third case (No 3, Table 3), the corrosion rate of the 12Kh1MF steel is higher than the corrosion rate of the St.3 steel by 28.7 %. Since a change in the absolute values of the corrosion rate by 50 % of the absolute value is acceptable for the high-temperature corrosion, therefore the data given in Table 3 cannot be used for the robust recommendation of any given steel grade. From the standpoint of the technology, the alkali water treatment will be more efficient in this particular case.

As for the polarization measurement data, these show that the corrosion of alloyed steel in hot water is expressly higher in comparison to that of carbon steel (Table 4). The difference in the mass corrosion factor (approximately 2 times higher in the primary water and 1.5 times higher in the treated water) cannot be

explained by the experiment error. Possibly, in this case the electrochemical activity of the alloyed steel occurs under the anode polarization both in subacid (primary water) and alkaline (treated water) mineralized waters. In the case of the alloyed steel, as a multicomponent alloy, the process of the formation of passive films under these conditions can be complicated due to the structural metal inhomogeneity and its corrosion rate will be higher in comparison to that of the carbon steel that has more uniform structure. Perhaps, in this case, the general salt composition of water plays an important role, since alloying with chromium and molybdenum should, on the contrary, increase the corrosion resistance of steels, especially in chloride-containing environments.²⁹ However, these conclusions require additional experimental investigations to reveal the specific features of the electrochemical behavior of the steels in coke quenching waters.

4. Conclusions

Corrosion of the steel heated to 373 K depends on the composition of biochemically treated water. The mass of the specimens of the St.3 steel is decreased after the cooling in the initial quenching water (IQW) and the mass of the specimens of 12Kh1MF steel is increased and it is explained by the adhesion behavior of the layer of corrosion products to the substrate. The mass of steel specimens is decreased after the heating to 773 K and cooling in the initial quenching water (IQW) and has approximately the same value independently of the steel grade. The mass of the specimens of the St.3 steel heated to 773 K and cooled in the treated quenching water (TQW) is increased during the first 25 cycles, however it is decreased due to the clinker separation as the number of test cycles is increased. The mass of the specimens made of the 12Kh1MF steel that are cooled in the TQW is decreased after the first 5 cycles, however the loss of its mass is 7.5 times lower than that of the St.3 steel. The steels heated to 373 K and cooled in the IQW and TQW and those heated to 773 K and cooled in the TQW are attributed to the class of the materials that have low resistance and the steels heated to 773 K and cooled in the IQW are attributed to nonresistant materials. The steel corrosion in IQW and TQW solutions develops in the active state. The absolute values of the corrosion rate of the 12Kh1MF steel are higher in all the cases than those of the St.3 steel. The use of biochemically treated water allows us to reduce the 12Kh1MF steel corrosion

rate and actually has no effect on the corrosion rate of the St.3 steel.

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ЗАЛЕЖНІСТЬ КОРОЗІЇ ОБЛАДНАННЯ УСТАНОВОК ГАСІННЯ КОКСУ ВІД ЯКОСТІ БІОХІМІЧНО ОЧИЩЕНИХ ВОД КОКСОХІМІЧНОГО ВИРОБНИЦТВА

Анотація. Досліджені процеси корозії сталей у біохімічно очищених водах установок гасіння коксу. Проведено гравіметричні дослідження зразків сталей за 373 і 773 К, що дало змогу встановити, що при нагріванні сталей Ст.3 і 12ХІМФ з подальшим охолодженням у воді спостерігається різний характер їх корозійного руйнування. Описано типи корозії, які виникають при контакті вуглецевих і легованих сталей з біохімічно очищеними водами коксохімічних підприємств, що були оброблені гідроксидом натрію. Показано, що результатом корозійного руйнування сталей у всіх досліджених середовищах є утворення плівок гідратованих оксидів заліза з різним характером адгезії до поверхні зразків. Доведено, що обробка води приводить до деякого зниження величин масового і глибинного показників корозії для Ст.3 і 12ХІМФ, однак не дає істотного ефекту при постійному контакті сталі з гарячою водою.

Ключові слова: корозія, гасіння коксу, оксид заліза, вуглецева сталь, гравіметрія, поляризаційна залежність.