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PILOT TESTS REAGENT FOR THE PREPARATION OF RECYCLED WATER ON SITE WATER CIRCULATION № 627 NPZ JSC "GAZPROM NEFTEKHIM SALAVAT"

ОПЫТНО-ПРОМЫШЛЕННЫЕ ИСПЫТАНИЯ РЕАГЕНТОВ ДЛЯ ПОДГОТОВКИ ОБОРОТНОЙ ВОДЫ НА ВОДООБОРОТНОМ УЗЛЕ № 627 НПЗ ОАО «ГАЗПРОМ НЕФТЕХИМ САЛАВАТ»

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Abstract. Based on the results of experimental and industrial water circulation mileage on site (hereinafter EWC (equipment of water circulation)) № 627 Refinery JSC "Gazprom neftekhim Salavat" testing reagents for the preparation of recycled water produced by "Research and technology center Salavatnefteorgsitez" (hereinafter RTC) an assessment of their effectiveness in comparison with basic reagents.

Defined Langelier saturation index (LSI - Langelier Saturation Index) and the stability index Riznera (RSI - Ryznar Stability Index). Found that part of the

water circulation cooling water unit is more prone to scale formation than to corrosion.

It is shown that in the period of mileage had an average evaporation rate of the order of 1.1-1.4 units, a relatively low figure for EWC. Presented and analyzed the results of the analytical control of feed water and circulating water in different periods of mileage (before the flood period, flood period and after the flood period).

It was revealed that the application of base biocide - sodium hypochlorite biocide activator leads to a sharp increase in the chloride ion in the recirculating water, in contrast to non-oxidizing biocide action AddiTOP B production RTC, with better efficiency and a significantly lower dosage of the latter.

Shows dosage and the operating flow of base reagents in comparison with reagent AddiTOP. Found that the annual economic cost of processing with the use of base reagents to 3 times higher than the cost of production of reagents AddiTOP RTC.

In general, the proposed program of circulating water treatment reagents RTC production at lower cumulative doses and costs, as effective as the program treatment basic reagents, and on the effectiveness of corrosion and microbiological protection surpasses it.

Аннотация. На основе результатов опытно-промышленного пробега на водооборотном узле (далее ВОУ) № 627 НПЗ ОАО «Газпром нефтехим Салават» по испытанию реагентов для подготовки оборотной воды производства ООО «НТЦ Салаватнефтеоргситез» (далее НТЦ) проведена оценка их эффективности в сравнении с базовыми реагентами.

Определены индекс насыщения Ланжелье (LSI – Langelier Saturation Index) и индекс стабильности Ризнера (RSI - Ryznar Stability Index). Установлено, что оборотная вода данного водооборотного узла более склонна к накипеобразованию, чем к коррозии.

Показано, что в период пробега коэффициент упаривания имел среднюю величину порядка 1,1-1,4 единиц, что является относительно

невысоким показателем для ВОУ. Приведены и проанализированы результаты аналитического контроля подпиточной и оборотной воды в различные периоды пробега (допаводковый, паводковый и послепаводковый).

Выявлено, что применение базового биоцида – гипохлорита натрия с биоцидом-активатором приводит к резкому увеличению хлорид-ионов в оборотной воде, в отличие от биоцида неокисляющего действия АддиТОП Б производства НТЦ, при лучшей эффективности и значительно меньшей дозировке последнего.

Приведены рабочие дозировки и расход базовых реагентов в сравнении с реагентами АддиТОП. Установлено, что ежегодные экономические затраты на обработку с применением базовых реагентов в 3 раза превышают затраты на реагенты АддиТОП производства НТЦ.

В целом предложенная программа обработки оборотной воды реагентами производства НТЦ, при меньших суммарных дозировках и затратах, не уступает по эффективности программе обработке базовыми реагентами, а по эффективности коррозионной и микробиологической защите превосходит ее.

Key words: equipment of water circulation, reagent, mileage, non-oxidizing biocide action, sodium hypochlorite, scaling, corrosion.

Ключевые слова: водооборотный узел, реагент, пробег, биоцид неокисляющего действия, гипохлорит натрия, солеотложение, коррозия.

In the context of closed water circulation systems of refineries and petrochemical plants, there is a constant deposition of hardness salts deposited on the equipment, corrosion develops, aerobic microorganisms multiply and sulfate-reducing bacteria. All this affects the operation of heat exchangers and other equipment can shorten battery life, leads to the inevitable complications in the process, increased costs, increased consumption of water and leads to

contamination of drains. Emerging issues - multifactorial requiring complex solutions.

Addressing the negative factors leading to complications of water circulation systems, carried out by different methods. The most effective and least expensive is by far the chemical treatment of circulating water cycles [1]. The use of reagents capable of stabilizing the production process, reduces downtime associated with repair of equipment allows the use of cheaper materials of construction [2].

Water circulation at sites of JSC "Gazprom neftekhim Salavat", as in other similar enterprises, use a special chemical treatment of water.

Equipment of water circulation (EWC) number 627 is an open, flowing, open system. Main characteristics EWC number 627 are presented in table 1, a block diagram of the EWC number 627 shown in figure 1.

Table 1. Main specifications EWC number 627

Index	Valid values
Volume of the system, м^3	6500
Circulation, м^3/hr	3600 – 3850
Temperature, °C	winter from plus 6 to minus 7; summer from plus 6 to plus 10
Feed water, м^3/hr, no more	150
Evaporation and entrainment, м^3/hr, no more	1,2-2,0
Purging and losses, м^3/hr, no more	5
Time working in a year of operation, days	365
Evaporation coefficient	2
Constructional materials	concrete, carbon steel, wood
Exterior features recycled water system and open areas	muddy, the smell, the presence of algae, foam films, slurries, sludge

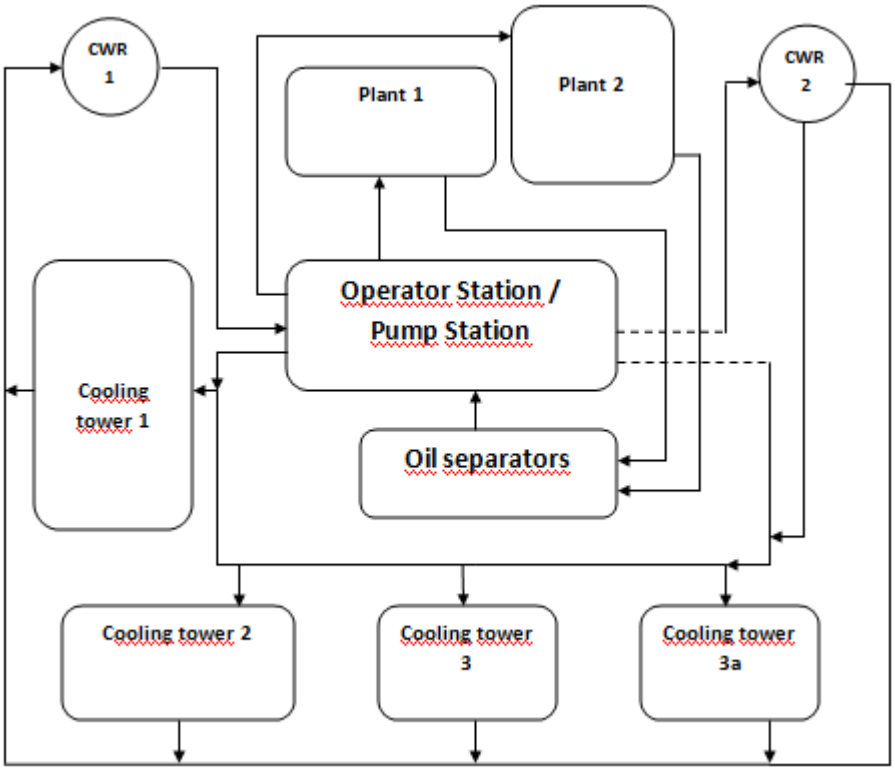

Figure 1. Block diagram EWC number 627
CWR 1,2 - cold water reservoir 1,2

Consumers of recycled water EWC number 627 are plants Hydrotreatment - 2, Catalytic cracking -1, Catalytic cracking - 2, Gas fractionation plant-1 refinery JSC "Gazprom Salavat neftekhim."

Indicator determining the tendency of water to corrosive process and the scaling is Langelier saturation index (LSI - Langelier Saturation Index), which in practice is calculated as a function of the electrical conductivity, calcium hardness, alkalinity, pH, and the maximum temperature of the heating surface [3]. To assess the thermal stability of the solution along with the Langelier index used Riznera stability index (RSI - Ryznar Stability Index), which connects the empirical data on the thickness of the film deposition observed for water recycling water from the chemical characteristics of water. As well as the Langelier Index, Index Riznera is based on the concept of water level saturable. Rizner tried to digitize the relationship between the saturation level for the formation of calcium carbonate deposits (table 2) [4].

Table 2. Langelier index and stability index Riznera for recycled water of EWC number 627

Period	Langelier Index (LSI)		Riznera stability index (RSI)	
	value	conclusion	value	conclusion
Before the flood period (up to 15.03.2013)	1,3	scale formation, corrosion minor	5,8	scale formation
Flood period (15.03.2013 - 01.05.2013)	1,6	scale formation, corrosion minor	4,8	scale formation
After the flood period (from 01.05.2014)	0,9	scale formation, corrosion minor	6,2	corrosion

After calculation of the Langelier and Riznera index obtain that recycled water of EWC number 627 is more prone to scale formation than to corrosion.

Ltd «RTC Salavatnefteorgsintez" developed reagents for the preparation of recycled water EWC under the trademark AddiTOP.

To assess AddiTOP reagents in an industrial environment with a view to their possible further implementation and comprehensive analysis of their impact on the quality indicators specific to the recycled water of recycled water system was conducted pilot production mileage reagents AddiTOP EWC number 627 Refinery JSC "Gazprom neftekhim Salavat" in the period from 1 February 2013 to 15 May 2013.

According to the approved program mileage was conducted in comparison with the base reagents (currently used EWC number 627) reagents in four stages (figure 2).

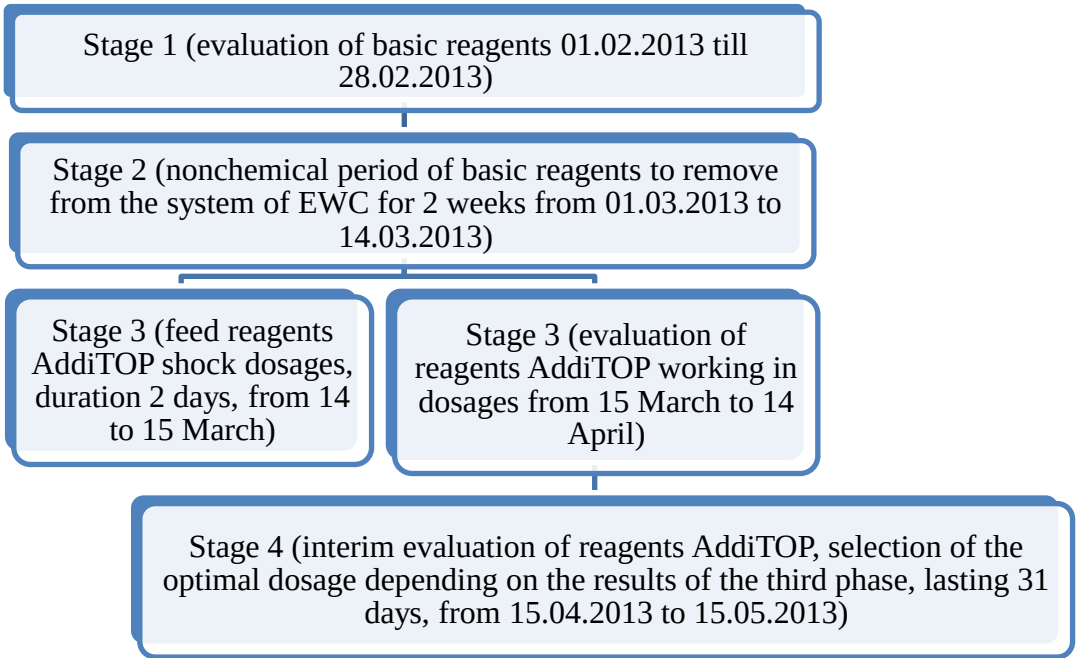

Figure 2. Stages of experimental-industrial mileage on EWC number 627 refineries

Operating doses and flow rate of base reagents when the evaporation coefficient equals 2 given in table 3.

Table 3. Operating doses of base reagents when K_{evap}= 2 and the average volume of feeding 650 m^3/day, calculation of annual consumption

Reagent	Dosage, g/t	Consumption, kg/d	Annual consumption (365 days), t
Base corrosion inhibitor	21,4	13,91	5,08
Base scale inhibitor	5,2	3,38	1,23
Base biodispersant	10,7	6,96	2,54
Base bromo biocide activator	36,8	23,92	8,73
Base bactericide - aqueous solution of sodium hypochlorite	608,8	395,72	144,44
			Total: 162,02

Dosage AddiTOP reagents produced by analogy with the base reagents technological personnel water circulation unit number 627 with a frequency of 1 per shift or 3 times a day.

Operating doses of AddiTOP when the evaporation coefficient equals 2 and calculation of annual consumption given in table 4.

Table 4. Operating doses of AddiTOP reagents when Kevap=2 and the average volume of feeding 650 m3/day, calculation of annual consumption

Reagent	Dosage, g/t	Consumption, kg/d	Annual consumption (365 days), t
Scale inhibitors AddiTOP IS-1	30	19,5	7,12
Corrosion inhibitor AddiTOP INV	20	13,0	4,75
Dispersant AddiTOP DP	10	6,5	2,37
Bactericide AddiTOP B	50	18,3	6,68
			Total: 20,92

Data analytical control of key parameters feed water and recycle water, characterizing the efficiency of the reagents in the suppression of corrosion processes and sediment hardness are shown in tables 5,6.

Table 5. Results of analytical control of feed water during the mileage

№	Index			Average value for period		
1	Index	unit of measure	Norm	Before the flood period (up to 15.03.2013)	Flood period (15.03.2013 - 01.05.2013)	After the flood period (from 01.05.2014)
2	pH	unit.pH	6,5-8,5	7,8	7,9	7,8
3	conductivity, УЭП	mkSm / cm	-*	326	260	227
4	the mass fraction of total dissolved iron	mg/dm^3	-	0,2	0,73	0,97
5	total alkalinity, $Щ_о$	mg-ekv/dm^3	-	2,9	2,4	1,85
6	total hardness, $Ж_{общ}$	mg-ekv/dm3	no more 5,8	3,1	3,2	2,3
7	temporary stiffness, $Ж_{врем}$	mg-ekv/dm3	-	2,2	1,8	0,7
8	rigidity of calcium, $Ж_{Ca}$	mg-ekv/dm3	no more 2,5	1,1	1,4	1,6
9	mass fraction of suspended solids	mg/dm^3	no more 25	5	6,7	10
10	mass fraction of chlorides	mg/dm3	no more 300	10	9,3	8
11	mass fraction of sulfate	mg/dm3	no more 100	24	12,3	13

* - indicator is not standardized

Table 6. Results of analytical control of recycled water during the mileage

№	Показатель			Average value for period			
				Before the flood period (up to 15.03.2013)		Flood period (15.03.2013 - 01.05.2013)	After the flood period (from 01.05.2014)
1	Index	unit of measure	Norm	Base reagents	Without reagents	Reagents AddiTOP	Reagents AddiTOP
2	pH	unit.pH	6,5-8,5	8,5	8,3	8	7,95
3	conductivity, УЭП	mkSm / cm	-*	395	358	311	241
4	the mass fraction of total dissolved iron	mg/dm^3	-	0,4	0,23	0,33	0,91
5	total alkalinity, $Щ_о$	mg-ekv/dm^3	-	3,2	3	2,8	1,95
6	total hardness, $Ж_{общ}$	mg-ekv/dm3	no more 5,8	3,8	3,5	3,5	2,5
7	temporary stiffness, $Ж_{врем}$	mg-ekv/dm3	-	2,7	2,5	1,2	0,55
8	rigidity of calcium, $Ж_{Ca}$	mg-ekv/dm3	no more 2,5	1,1	1,1	2	2,05
9	mass fraction of suspended solids	mg/dm^3	no more 25	9	13	11	16
10	mass fraction of chlorides	mg/dm3	no more 300	31	11	9,3	10
11	mass fraction of sulfate	mg/dm3	no more 100	28	34	17	6

* - indicator is not standardized

General analysis of physico-chemical parameters of water during the mileage

Values of the activity of hydrogen ions (pH) of recycled water for the entire period were mileage within the boundaries of acceptable standards and did not exceed 8.5 units, with a tendency to a decrease of reagents AddiTOP. At the same time, when using reagents average pH of of recycled water was equal to the upper boundary of the rated range and the unit was 8.5. That involves the use as a bactericide large quantities of sodium hypochlorite was hydrolyzed to form sodium hydroxide.

The alkalinity of the water caused by the presence in solution of hydrates ,

carbonates, bicarbonates and phosphates of alkali and alkaline earth metals , sodium , calcium and magnesium , causing alkaline water. From the beginning , the path of 01.02.2013 and 08.04.2013 , the rate has remained relatively constant and is not reduced below 2.5 dm 3 . Starting from April 8, 2013 , a sharp decline of total alkalinity ($Щ_о$) , due to the flood period , while maintaining a constant correlation this indicator between feed water and circulating water.

The most important characteristics of water quality are specific electrical conductivity (the УЭП), which reflects the total mineralization of water, and the estimated coefficient of evaporation Ky. During the entire run evaporation coefficient Ku had an average value of about 1.1-1.4 units, a relatively low figure for EWC and associated with significant blowing at facilities (drain recycled water in industrial sewers). From Figure 3, it follows that the УЭП feed water and recycle water expectedly correlated in all periods of mileage.

During feeding the reagents AddiTOP, УЭП as the recycle water, and feed water was slightly higher than during the mileage on base reagents. This is directly related to the increase in flood (contaminated) water in feed water during the snowmelt. The best result was registered 03.04.2013 - 418 mkSm/m (figure 3).

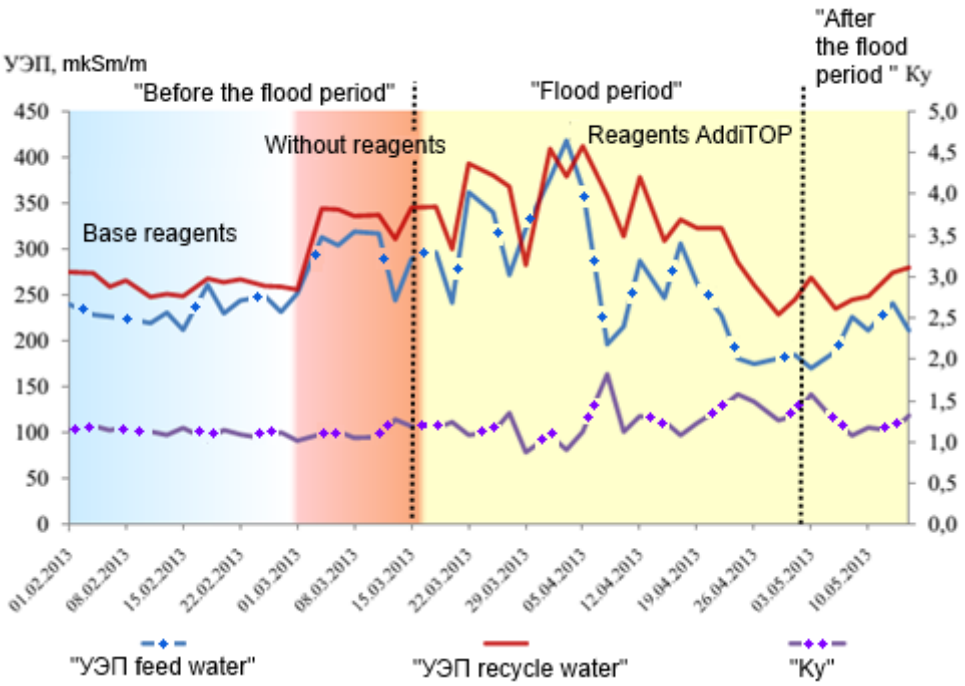

Figure 3. Values УЭП and Ky feed water and recycle water

Stiffness feed water and recycle water also correlated. During the mileage these indicators do not exceed the established norms and characterized by stable with some decrease in the period mileage on AddiTOP reagents, due to changes in the concentration of salts in the feed water in the flood and after the flood periods.

Indicators of suspended solids in the feed and circulating water in the period mileage on base reagents and reagent-free period is not exceeded. Suspended solids in the recycle and feed water during the mileage on the reagents AddiTOP increased dramatically, due to the rate of melting snow. Application in the flood period dispersant AddiTOP DP allowed to minimize the impact of a significant increase in suspended solids in the feed water to the state of the circulating water. After completion of the snow melting suspended solids to normal and no more than indicators on base reagents, indicating the successful removal (leaching) and suspended solids from the system (figure 4).

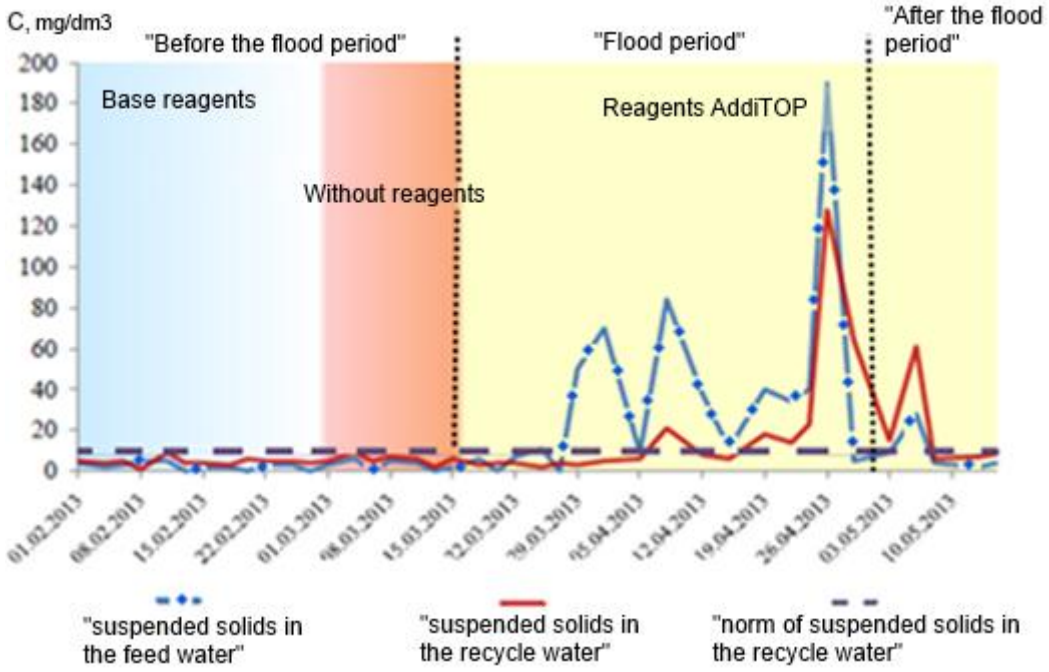

Figure 4. Suspended solids in feed and circulating water EWC number 627

It should be noted that objectively evaluate the effectiveness of scale inhibitors and dispersants on the physicochemical parameters and feed of

recycled water, is extremely difficult because of the need to consider many indicators and influencing factors.

Real picture of efficiency can be obtained only by planned or unplanned inspection of heat exchange equipment (tufts refrigerators, etc.), which was used to cool the circulating water with scale inhibitor. In case inefficient use of reactants possible premature failure of the heat exchanger or to reduce normalized parameters such as temperature and process pressure before and after the heat exchangers. During the mileage of complaints from the shop personnel and technological installations, which provides recycled water EWC number 627, have been reported, which allows at this point in time to assess the basic reagents and series AddiTOP as effective.

Analysis of corrosion protection of equipment

Content of dissolved total iron indirectly characterizes corrosivity environment and, accordingly, the effectiveness of the corrosion inhibitor.

Over the entire period of mileage reagents AddiTOP total iron ion concentration ranged from 0.10 to 1.72 dm 3. Critical value of the mass concentration of the indicator is the amount of total iron 3.0 dm 3. Thus, for the period run the concentration of dissolved iron was in the safe range.

As shown in Figure 5, starting from 17.03.2013, parameter total iron content in recycle water tended to increase, although in the feed water, period of the increase associated with the pollution of river water meltwater, comes later - 08.04.2013. This is probably due to the supply shock dosages AddiTOP reagents and, in particular, high efficiency dispersant AddiTOP DP, causing washing out iron deposits from the surfaces of the process equipment. In the future, the iron content in recycle water does not exceed the performance of makeup water, which indicates the absence of foreign iron in recycle water and sufficiently protect equipment from corrosion.

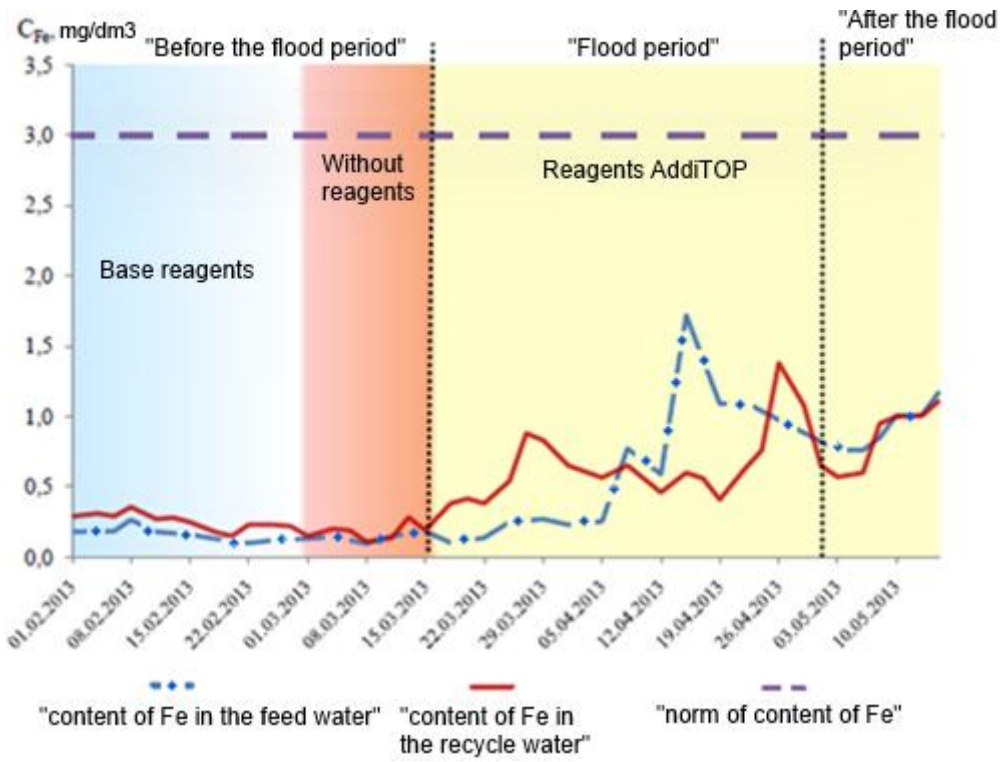

Figure 5. Changing of content the dissolved iron Fe in the feed water and recycle water during the mileage

In the recycle water and feed water also control the concentration of sulfates and chlorides as the most common anions in nature. In general, the greater the amount of sulphates and chlorides, the more corrosive environment. Sulfates and chlorides during the mileage on the base reagent was significantly higher than in the without reagents period and AddiTOP reagents. High chloride content during using a reagent linked to a basic feed a large amount of sodium hypochlorite (hereinafter HPHN) in system. Use as a bactericide HPHN leads to irreversible impacts on the environment and human because of the potential danger of chlorine gas release during storage. HPHN form disinfection by-products, including trihalomethanes, which degrades water quality. HPHN also has a pretty strong corrosive effect on various materials. Large dosage HPHN due nonselective HPHN (active chlorine, primarily consumed for reacting with unsaturated hydrocarbons, ammonia and other organic compounds). In addition, the chlorine gas is extremely poisonous gas, the second class of danger [5, 6].

During the mileage on the reagents AddiTOP chloride content correlates well with the content of the anion in feed water, which means that the AddiTOP

reagents do not contain chlorides and the anion enters the system with feed water only.

For monitoring the rate of corrosion during mileage between the base reagents and the AddiTOP reagents corrosion rates were measured gravimetrically using coupons [7, 8], representing four steel plates (St20), and assembled in the cassette and installed oil separators and installed in the cold water reservoir of EWC number 627.

In the table 7 the data on rate of corrosion of metal specimens in the gravimetric tests EWC number 627.

Table 7. Corrosion rate of the metal plates

Object	Period	Corrosion rate mm/year	
		Base reagents	AddiTOP
Recycled water from oil separators	February 1 - February 28 (before the flood period)	0,286	-
Cold water reservoir	February 1 - February 28 (before the flood period)	0,335	-
Recycled water from oil separators	March 5 - April 14 (flood period)	-	0,221
Cold water reservoir	March 15 - April 14 (flood period)	-	0,253
Recycled water from oil separators	April 15 - May 15 (after the flood period)	-	0,327
Cold water reservoir	April 15 - May 15 (after the flood period)	-	0,414

The test results (Table 7) show that the cooled cooling water in the tank has a higher corrosion activity than the hot water circulating in the oil separators. These data are typical for a base reagent and for AddiTOP reagents and explained by the presence of large amounts of dissolved oxygen in the feed water. After reducing the dosage of corrosion inhibitor AddiTOP INV corrosion rate increased reagents and surpassed several basic reagent. This fact points to the need to return to the original dosage AddiTOP INV (in the period from March 15 to April 14), at which the rate of corrosion on the the AddiTOP reagents was lower in comparison with the base reagent.

Microbiological condition of recycled water during mileage

Efficiency of bactericide was assessed by BART-test as containers with a nutrient medium for the determination of the calculated iron-reducing bacteria (hereinafter IRB) and sulfate-reducing bacteria (hereinafter SRB) bacteria.

During the reagent AddiTOP from 15 March to the end of April 2013 there was a flood in which bacterial contamination of feed water is much higher than in winter. Under these conditions, bactericide AddiTOP B showed similar efficacy with basic reagents. At the end of the flood (sampling 13.05.2013 r) on the number of colonies reagents AddiTOP SRB decreased in comparison with the basic reagents 3.6 times IRB - 22 times [9]. Thus, bactericide AddiTOP B exceeds basic reagents efficacy in suppressing life and SRB and IRB.

Conclusions

Summarizing the results of mileage, we can confidently assert that the proposed program of circulating water treatment reagents AddiTOP as effective treatment program basic reagents, and on the effectiveness of corrosion and microbiological protection surpasses it. Annual consumption of basic reagents in 3 times higher than for the reactants AddiTOP that, combined with a lower total cost of processing reagents AddiTOP makes reasonable transition to the new package of the reagents for the treatment of recycled water WEC number 627.

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