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Ion-Exchange Cleaning of Oil Washing Water from Chloride Ions

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Abstract

The process of ion-exchange cleaning of oil cavitation water from chloride ions with its subsequent return to the recycle of oil refining has been studied. Based on the presented experimental data, the technological parameters of its main stages were established: cleaning water from fractions of oil by mixing it with activated carbon powder and its subsequent running through a filter; cationite water treatment in a periodic mode using the CU–2 cation exchanger in the H⁺-form; decarbonization of water; anionite treatment of water in a periodic mode using anion exchanger AW–17–8 in OH⁻-form; regeneration of the cation exchanger with a 0.75 % solution of sulfate acid and its washing with decarbonated water; regeneration of anion exchangers with 3.0 % sodium hydroxide solution; washing the anion exchanger with decarbonated water. It is shown that ion-exchange regeneration includes the technology of reducing the concentration of chloride ions in oil washing water to the technological standards of wastewater treatment, ensures the return of oil washing water to the technological cycle, suggests low waste and efficiency of the process due to the use of available ion exchangers.

Keywords: Oil washing water; Chloride ions; Regeneration; Cleaning; Ion exchangers.

1. Introduction

The volume of world industrial oil refining has been increasing year by year recently ^[1-2]. The recovered crude oil contains a number of impurities that get into the resulting oil products, having a negative impact on the technological processes of oil refining; they cause poisoning of catalysts, corrosion of equipment, deteriorate the quality of oil products, etc. ^[3-6]. The presence of high concentration NaCl electrolyte in oil promotes interaction between oil components and the sandstone surface, which increases the stability of suspensions during oil recovering ^[7]. To improve the quality of oil products and prolong the operation of technological oil refining units, industrial technologies for processing crude oil include the stages of its desalting and dehydration ^[5, 8-9]. So, in the process of oil treatment by the cavitation method, the washing water is gradually enriched with chloride ions. The concentration of Cl⁻ ions increases up to 70 times in comparison with the permissible amounts of 2-5 mg/L^[4]. The presence of chlorine ions causes the formation of hydrochloric acid in further technological stages of thermal treatment of oil. The HCl vapors reduce the output of oil products, disrupt the operation of oil refineries, lessen the calorific value and quality of oil fuels, and cause corrosion of the equipment of oil refineries. In addition, the air in the working area is polluted by the toxic HCl compound, which impacts negatively the health of the people working at the enterprise, causes corrosion of technological equipment and significantly worsens working conditions.

Traditional technologies of crude oil refining existing at oil refineries ^[8-9] are often ineffective in terms of environmental safety and economy. Due to the absence of closed water consumption in most of them, there is a common practice of discharging large volumes of wastewater into industrial sewage as a result of processing. Therefore, treatment of industrial effluents at oil refineries is the main technology for protection and reuse of water resources in many countries around the world. Besides, the use of wastewater for the production of industrial water will significantly reduce the enterprises' need in water and efficiently solve the problem of protecting the water basin from pollution ^[10].

The existing diversity of methods for cleaning of oil washing water in the scientific literature and an active search for new technologies proves the urgency of the existing problem ^[3–15]. However, most of the methods proposed by the authors are unprofitable in terms of both capital and operating costs. The applied approaches are often ineffective for the treatment of chloride-containing wastewater ^[5, 10–13]. Traditionally, the petrochemical industry uses adsorbents: activated aluminum oxide, silica gel, activated carbon and molecular sieves. The factors influencing the choice of the sorbent are the composition of raw materials, operating conditions and requirements for the product ^[16].

The selection of methods for cleaning of chloride-containing water at the level of milligram concentrations proved the ion exchange methods to be the most expedient ^[17-19]. The technologies proposed by the authors of these works make it possible to clean waste water from chloride ions by passing it through a layer of ion exchanger. The presented results of laboratory and experimental studies of cleaning processes enable to evaluate the features and effectiveness of their application. However, the multiplicity of operations performed and the additional consumption of materials increase the cost of technological processes and make these methods economically unprofitable. Besides, the proposed methods do not provide recommendations for the removal of residual amounts of oil fractions contained in the wastewater of the oil refining industry.

The purpose of the research is studying the ion-exchange process of regenerating oil cavitation washing water of from chloride ions.

Research objectives:

- studying the process of ion-exchange cleaning of oil cavitation water from chloride ions;
- optimizing the stages of the process of ion-exchange cleaning of oil washing water from chloride ions;
- developing the technology for the process of ion-exchange cleaning of oil washing water from chloride ions with their subsequent return to the oil refining cycle.

2. Research methodology

The object of the study was the washing water of the cavitation treatment of the oil produced by the industrial engineering company "BI-Rezon". The water contained 7.04–8.45 mmol/L of Cl⁻-ions, which exceeds the permissible concentration of 0.06–0.14 mmol/L.

Chloride ions in periodically taken samples were determined by potentiometric method using a working chlorine selective electrode and a silver chlorine reference electrode. The content of sodium ions in the solution after the cavitation treatment of oil was monitored on the atomic absorption spectrophotometer "Saturn" at a wavelength of λ =328.1 nm; slit=0.1 nm; J=10.0 mA. The acidity of water was measured with a pH-meter measuring device – a millivoltmeter, which includes a selective electrode and a modified reference electrode in polypropylene housings. The duration of a single measurement was 1–2 s.

The oil washing water was regenerated by ion exchange. Using this method makes possible to remove an excess amount of chloride ions and ensure the return of oil washing water to the washing cycle. Cation exchanger CU–2 and anion exchanger AW–17–8, which combine ion exchange capacity and high-performance indicators, were chosen as ion exchangers. The ion-ogenic group for the cation exchanger is the $-SO_3$ group, and for the anion exchanger – the $-N(CH_3)_4^+$ group. Therefore, the CU–2 cation exchanger belongs to the group of strongly acidic cation exchangers, and the AW–17–8 anion exchanger belongs to the strongly basic anion exchangers. Ion exchangers have high sorption properties in relation to the corresponding sodium and chlorine ions. Their total exchange capacity for dry ion exchanger is, mmol/g: for CU–2 it is 4.45; for AW–17–8 it is 2.8–3.5. The total exchange capacity of ion exchangers (CE) was determined by the formula (1)

 $CE = (C_1 - C_2) \cdot V \cdot m^{-1}, \text{ mmol} \cdot g^{-1},$

(1)

where: C_1 and C_2 are the concentration of ions, respectively, in the waste washing water and in the regenerated one; V is the volume of water passed through the ion exchanger until it is saturated; m is the mass of the ion exchanger.

The process of cleaning of oil cavitation water includes the main stages:

- cleaning of water from oil fractions by mixing it with activated carbon powder and then passing water through a filter;
- cationite treatment of water in a periodic mode using the CU-2 cation exchanger in the H⁺-form;
- decarbonization of water;
- anionite treatment of water in a periodic mode using anion exchanger AW-17-8 in OH⁻-form;
- regeneration of the cation exchanger with a 0.75 % solution of sulfate acid and its washing with decarbonated water;
- regeneration of the anion exchanger with 3.0 % sodium hydroxide solution and its washing with decarbonated water.

3. Results and discussion

3.1. The process of pre-treatment of oil washing water

During ion exchange treatment of oil washing water, it is necessary to provide for the possibility of residual oil entering the water. Repeated flushing of oil with water leads to the formation of a fairly stable "oil-in-water" emulsion. Organic compounds can block the functional groups of ion exchangers, as a result their sorption capacity sharply decreases. To achieve the maximum possible working capacity of ion exchangers, it is necessary to remove oil fractions from the water. In this regard, it is proposed to introduce two successive stages – cleaning of water from oil fractions by mixing it with activated carbon powder and then passing water through a filter. For complete water cleaning from oil fractions, it is necessary to repeat these operations twice. Activated carbon is used in filters once, then it is completely replaced, since no economically justified methods of its regeneration have been developed yet. The already used activated carbon can be recommended as a combustible material, which increases the resource-saving parameters of the process.

3.2. The process of cation exchange of oil washing water

Regenerated water undergoes ion exchange treatment, which consists of successive processes of its cation exchange and anion exchange. Chlorine ions are removed on the AW-17-8 anion exchanger in the OH⁻-form. The content of the working fraction in the anion exchanger reaches 94 %, the minimum density in the OH⁻-form is 1.1 g/cm³. Activation of the functional groups of the anion exchanger and the achievement of the maximum possible dynamic sorption capacity are possible in an acidic medium, but the washing water has an alkaline reaction. This circumstance was one of the reasons for the introduction of the previous technological stage, which is cationite water treatment using the CU-2 cation exchanger. This cation exchanger has a spatial structure with cavities. Upon swelling, its structure virtually does not change, and the increase in volume occurs due to the spread of polymer chains. The second reason for the introduction of water cation exchange is removal of sodium ions, in whose presence the capacity of the anion exchanger is significantly reduced. The results of the cationite treatment of oil washing water are presented in Table 1.

After the washing water is regenerated, the sodium ion content decreases. At a constant concentration of C_{Na^+} , CE depends on the volume of regenerated water. The optimal ones are No. 7–17 (Table 1). For the effective use of the ion exchanger in a periodic mode the ratio of the volume of washing water to the mass of the cation exchanger should not exceed (1.6–2.1):1.

When using an ion exchanger in the amount of 50 % (No. 20, Table 1) in relation to the optimal one (No. 17, Table 1), the volume of regenerated water is maximum, but the ion exchanger KU-2 cannot absorb all sodium ions. Their residual amount in the eluate is high, it is $C_{\text{Na}^+} = 3.216$ mmol/l. If we take an ion exchanger in the amount of 150 % (No. 21, Table 1) in relation to the optimal one (No. 17, Table 1), then the concentration of sodium ions in the

eluate will be low. In this case, the ion exchanger is not fully used, its capacity is only 67 % of the optimal value of 9.34 mmol/g (No. 17, Table 1).

No.	Final concentration of Na+ ions (C2), mmol/L	Solution volume up to saturation of the ion exchanger, V, L	CE, mmol/g
1	0	10.0	2.74
2	0	15.0	4.11
3	0	20.0	5.48
4	0	25.0	6.85
5	0.001	27.0	7.4
6	0.002	30.0	8.19
7	0.161	32.0	8.51
8	0.351	34.0	8.72
9	0.602	36.0	8.78
10	0.718	37.0	8.81
11	0.733	38.0	9.02
12	0.788	39.0	9.15
13	0.880	40.0	9.20
14	0.963	41.0	9.26
15	0.971	41.3	9.31
16	0.984	41.5	9.33
17	1.032	42.0	9.34
18	2.058	42.2	7.22
19	2.553	42.5	6.22
20	3.251	42.0	9.36
21	1.003	42.0	6.26

Table 1. Quantitative criteria for the process of cationite treatment of oil washing water at an initial concentration of Na⁺ – ions $C_1 = 5.48$ mmol/l and an ion exchanger mass m = 20 g

3.3. The process of decarbonization of oil washing water

During cationite treatment, as a result of the interaction of H⁺ ions, which are released into water due to ion exchange, and $CO_3^{2^-}$ ions, which are present in oil washing waters, carbonic acid is formed. As a result, the outlet water has a pH of ~ 3.0. To increase the pH (to the required 4.5–5), carbonic acid is removed by heating to temperatures of 30–45°C (Table 2).

No. Decarbonization time, min pН 3.0 1 5 2 10 3.2 3 15 3.5 4 20 3.7 5 25 4.0 30 6 4.2 7 35 4.5 8 40 4.7 9 45 5.0 10 50 5.2 11 55 5.5

Table 2. The results of decarbonization of regenerated water

According to Table 2, the decarbonization time at the set temperature is 35-45 minutes. It is the optimal period of time for the establishment of pH = 4.5-5.0, which is required for anionite treatment.

3.4. The process of anionite treatment of oil washing water

After cationite treatment the regenerated water, containing 8.45 mmol/L of Cl⁻ ions, is filtered through anion exchanger AW-17-8. The results of the process of anionite treatment of oil washing water are presented in Table 3.

Table 3. Quantitative criteria of the process of oil washing water anionite treatment at the initial concentration of Cl⁻-ions $C_1 = 8.45$ mmol/l and the mass of the ion exchanger m = 60 g

No.	Final concentration of Na+ ions (C2), mmol/L	Solution volume up to saturation of the ion exchanger, V, L	CE, mmol/g
1	0	10.0	2.74
2	0	15.0	4.11
3	0	20.0	5.48
4	0	25.0	6.85
5	0.001	27.0	7.4
6	0.002	30.0	8.19
7	0.161	32.0	8.51
8	0.351	34.0	8.72
9	0.602	36.0	8.78
10	0.718	37.0	8.81
11	0.733	38.0	9.02
12	0.788	39.0	9.15
13	0.880	40.0	9.20
14	0.963	41.0	9.26
15	0.971	41.3	9.31
16	0.984	41.5	9.33
17	1.032	42.0	9.34
18	2.058	42.2	7.22
19	2.553	42.5	6.22
20	3.251	42.0	9.36
21	1.003	42.0	6.26

The working capacity of the anion exchanger depends on the mass of chloride ions in the regenerated waters, that is, on their volume at a constant concentration of C_{CI} . As it can be seen from the data in Table. 3, ratios No. 14–18 are considered optimal. Therefore, for effective use of the ion exchanger in a periodic mode, the ratio of the volumes of washing water to the mass of the ion exchanger should not exceed (0.33–0.36):1.

When using the ion exchanger in the amount of 50 % (No. 21, Table 3) in relation to the optimal (No. 18, Table 3), the volume of regenerated water is maximum. However, in this case, the anion exchanger cannot absorb all Cl⁻ ions. Their residual concentration in the eluate is high, 4.32 mmol/L. If the ion exchanger is used in the amount of 150 % (No. 22, Table 3) in relation to the optimal one (No. 18, Table 3), then the concentration of chloride ions in the eluate will be low, but the ion exchanger will not be not fully used. Its capacity at this is only 66.8 % of the optimal one.

After anionite treatment the content of chloride ions in the regenerated water is reduced to the concentrations required by the oil treatment technology (0.14 mmol/L). Therefore, it can again be returned to the technological cycle of oil washing.

3.5. The regeneration process of ion exchangers

Ion exchangers need regeneration, therefore, the mode of cation exchange and anion exchange is periodic. Ion exchangers must be replaced when their ion exchange layer is depleted. For the cation exchanger the condition for replacement is when the concentration of Na⁺ ions in the eluate reaches 1.0 mmol/L, for the anion exchanger it is when the concentration of Cl⁻ ions is 0.14 mmol/L. Regeneration of ion exchangers is carried out in a periodic mode from bottom up for 6–10 hours at a speed of 3–5 m/h. Regeneration of the cation

exchanger is carried out with a solution of 0.75 % sulfate acid (2), and the regeneration of the anion exchanger – with the 3 % sodium hydroxide solution (3)

 $2NaR + H_2SO_4 = Na_2SO_4 + H_2R.$

(2)

(3)

RCI + NaOH = ROH + NaCI

To check the stability of the ion exchangers, 10 cycles of washing water regeneration were carried out, followed by the ion exchanger regeneration using a solution of the following composition: for the cation exchanger – C_{Na^+} = 5.48 mmol/L; for anion exchanger – C_{Cl^-} = 8.45 mmol/L. In this case, about 2 % of the ion exchanger is lost as a result of its transfer to the solution. In addition, to improve the technical and economic indicators of ion exchangers and loosen their upper layers, the ion exchangers are washed with decarbonated water in the direction from bottom up (Table 4). The washing operation is considered complete when pH = 7.

No.	The ratio of volumes of cat- ion exchanger and water	рН	The ratio of volumes of anion exchanger and water	pН
1	1:1	4.2	1:1	9.2
2	2:1	4.8	2:1	9.0
3	3:1	5.5	3:1	8.5
4	4:1	6.3	4:1	8.2
5	5:1	6.9	5:1	8.0
6	6:1	7.0	6:1	7.6
7	7:1	7.0	7:1	7.4
8	-	-	8:1	6.9
9	-	-	10:1	7.0
10	-	-	12:1	7.0

Table 4. Volumes of decarbonized water for washing the cation exchanger

According to the results of Table 4, the optimal parameters of ion exchanger washing were determined. As it can be seen from the given data, for the cation exchanger 5-7 volumes of water per volume of the cation exchanger (No. 5-7, Table 4) are optimal, for the anion exchanger 8-12 volumes of water per volume of the anion exchanger (No. 8-10, Table 4) are optimal.

3.6. Technology of the process of regenerating oil cavitation washing water from chloride ions

Cleaning oil wastewater from chloride ions and subsequent regeneration of ion exchangers are combined into a single technological scheme (Fig. 1), which allows almost complete removal of Cl^- ions from oil washing waters. The technological scheme includes the reserve columns with regenerated ion exchangers.

According to the technological process (Fig. 1), the regenerated washing water of oil enter reservoir 1, where, in the process of active mixing, the oil fractions are sorbed by activated carbon.

After passing through filter 2, the regenerated water is sent to the second stage of cleaning from oil fractions, respectively, passing through the reservoir with coal 3 and filter 4. After the sorption of oil fractions, the activated carbon can be utilized, as a combustible material among others. The water cleaned from oil fractions enters the working column with the cation exchanger 5. Control of the water pH at the exit from column 5 is carried out in tank 6. After cationization, carbon dioxide is removed from the water in calciner 7. Then the water enters the column with anion exchanger 8 for cleaning from anions, including chlorine ions. After anion exchange, the cleaned water is returned to the process cycle for oil washing.

After saturation of ion exchangers, they are periodically regenerated. During regeneration, spare columns are included in the water cleaning process: with cation exchanger 9 and with anion exchanger 10. Regeneration and washing of ion exchangers are carried out sequentially through regeneration units (RU I, RU II) and cleaning units (CU I, CU II). Initially, the regenerating solution (acid or alkali) from reservoir 11 (12) is fed from the bottom through the reservoir 1rce (1ra), respectively, to columns 9, 10. The acid (alkali) solution is filtered through the ion exchanger in the direction from the bottom up and enters the regeneration unit (RU I, RU II) into a container 2rce (2ra). At the next stage, the acid (alkali) solution,

respectively, enters the 3rce (3ra) reservoir. After passing through the regenerating solution, the ion exchanger washing operation begins. In this case, decarbonized water from reservoir *13* through the container 1wce (1wa) is supplied from below to regeneration columns *9*, *10*, filtered through the ion exchanger in the direction from the bottom up and enters the cleaning unit (CU I, CU II) into the reservoir 2wce (2wa). At the next stage, respectively, it enters the reservoir 3wce (3wa).



Figure 1. The scheme of the method of ion exchange treatment of oil washing water from chlorine ions: (RU) is the regeneration unit; (CU) is the cleaning unit; (rce) is the reservoir of cation exchanger regeneration; (wce) is the reservoir of cation exchanger washing; (ra) is the reservoir of anionite regeneration; (wa) is the reservoir of anionite washing

Thus, the developed technology provides for cleaning of washing water from residual oil fractions, allows for the cleaning of chloride ions to the norms outlined by the technological process and ensures the return of water to the production cycle after regeneration. In addition, the technology is economical due to resource conservation and the use of the most common and affordable brands of ion exchangers.

4. Conclusion

The process of ion exchange cleaning of oil cavitation washing water from chloride ions has been studied. On the basis of the presented experimental data, the optimal parameters of its main stages were determined:

- cleaning of washing water from oil fractions by mixing it with activated carbon powder and then passing water through a filter. To completely clean the water from oil fractions, it is necessary to repeat these operations twice;
- cation exchange treatment of washing water in a periodic mode: the ratio of the volume of washing water to the mass of the ion exchanger should not exceed (1.6–2.1):1; pH value of water after cation exchange treatment is about 3.0. To increase the pH (to the required 4.5–5), carbonic acid is removed by heating to temperatures of 30–45 °C for 35–45 min;
- anionite treatment of washing water in a periodic mode: the ratio of the volume of washing water to the mass of the ion exchanger should not exceed (0.33–0.36):1; the concentration of chloride ions in water after anionic treatment should be reduced to the technological standards for oil treatment of 0.06–0.14 mmol/L;
- regeneration of the cation exchanger: in a periodic mode from bottom up with 5–7 volumes of 0.75 % sulfate acid solution per one volume of cation exchanger at a rate of 3–5 m/h for 6–10 hours;
- regeneration of the anion exchanger: in a periodic mode from bottom up with 8–12 volumes of 3 % sodium hydroxide solution per one volume of anion exchanger at a rate of 3–5 m/h for 6–10 hours.

The technology has been developed for the process of ion exchange cleaning of oil washing waters from chloride ions. It is shown that ion-exchange regeneration includes the technology of reducing the concentration of chloride ions in oil washing waters to the technological standards of wastewater treatment, ensures the return of oil washing waters to the technological cycle, assumes low waste and efficiency of the process due to the use of available ion exchangers.

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