

# Development of Ecological Effective Cleaning Method in Oil Industrial Wastewater (Iw) Mechanical Cleaning Plants

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## Abstract

Due to the complex composition and dispersed systems of industrial wastewater generated in the oil refining industry, which have a colloidal solution, deep cleaning of these waters, up to 100% of oil product waste, is one of the main unresolved environmental problems. Due to the high content of MOPW in the IW obtained from ORI, the initial treatment of these waters is carried out by the method of mechanical precipitation due to the minimum of 1–2 g/l and the maximum concentration of 5–10 g/l. Depending on the amount of 1000–2000 mg/l of MOPW included in these devices, the purification of IW is often carried out in the actual range of 250–500 mg/l. In the next stages, physicochemical, chemical, biological, etc. the methods are carried out only in accordance with the environmental standards of the remaining MOPW to a certain extent. Although some of this water is transported to the periodic water system after the final biological treatment of the IW of the industry, the MOPW mixture, which remains in the ecological norms due to the discharge of the rest into the water basins, has a negative environmental impact on the flora and fauna of the basin. The ecological problem of the destruction of ecosystems still persists.

**Keywords:** Oil refining, industry, production, wastewater, mechanical, extractant, coagulant, flocculant

## Introduction

Wastewater containing oil has its own specific characteristics in terms of composition and behavior of oil products in the aqueous environment, and ultimately determines the methods of treatment of this type of wastewater. The main features that determine the behavior of oil products in water are that they have a lower density than the density of water (gasoline 0.70–0.73, diesel fuel 0.8–0.9, jet fuel 0.8–0.85, fuel oil 0.94–1.0 g / cm<sup>3</sup>) and low solubility. The latter does not exceed 20–30 mg / l in water for light oil fractions (gasoline), 70–90 mg / l for kerosene, and is practically zero for heavy fractions. When petroleum products fall into the water,

their bulk is in the form of coarse dispersions (droplets), and because of their low density, they easily rise to the surface of the water to form a floating layer or layer. The rest of the petroleum products are in a finely dispersed state and can form an "oil in water" emulsion. It is assumed that the true emulsion is formed in the colloidal dimensions of oil droplets (approximately 0.1  $\mu\text{m}$ ). However, persistent emulsions are also observed in larger droplets in wastewater containing oil products. The durability of the emulsion is determined by the surface tension, the kinetic resistance of the particles, their small density, the stabilizers of emulsions can be particles, depending on the wastewater. At present, various surfactants are finding a wider field of application, and their exposure to petroleum-containing effluents significantly disrupts the cleaning process by stabilizing the emulsion (Khaidarov et al., 2005; Abrosimov et al., 2002; Komissarov et al., 2002; Nevsky et al., 2004; Glukhova et al., 2003; Bukhgalter et al., 2003; Kuchera et al., 2007; Glueckstern et al., 2000; Magid et al., 2006).

Coagulation is one of the most commonly used treatment methods for wastewater, refined and insoluble oils, and petrochemicals. Numerous studies in this area show that aluminum and iron oxides are used as coagulants in wastewater treatment (Kuchera et al., 2007; Glueckstern et al., 2000; Magid et al., 2006; Magid et al., 2002; Magid et al., 2007; Gentsler et al., 2004; Ksenofontov et al., 2004).

As noted in many technical literatures, the IW formed in the oil refining industry (ORI) is very different in terms of complex composition, flow rate and volume compared to the industrial wastewater generated in the oil extraction (extraction) industry. That is why ORI mixes wastewater of different compositions formed in very different technological processes and initially enters the mechanical treatment plant with a common sewage system (Kuchera et al., 2007).

Due to the high content of MOPW in the IW included in these units, these units are initially precipitated in the oil or oil separator sections and cleaned in large additional sediments belonging to the specified treatment area. Then, in the stages of physical-chemical, chemical and biological treatment, the cleaning technological processes are continued, part of the IW purified in accordance with the ecological norms is discharged into the recirculating water system and part into the water basins. Despite treatment in accordance with environmental standards, long-term environmental problems arise due to the presence of MOPW in the IW, even in the amount of less than 5–9 mg/l, which has a negative environmental impact on the flora, fauna and ultimately the ecosystem of the IW (Glueckstern et al., 2000).

We have been conducting long-term research and experimental work to clean up 100% of MOPW with various chemical components in several directions from ORI samples with very complex composition taken from the entrance of ORI mechanical treatment plants.

As noted in the literature of many technicians, we have conducted an ecological analysis of the reasons for the failure to separate and purify the IW of ORI, which is

a colloidal solution with a continuous dispersed system, up to 100% MOPW emulsion.

An emulsion of an oil, petroleum product waste mixture, which is used to purify some selective substances, such as solvent  $\text{CCl}_4$ , as well as other coagulants in different concentrations, precipitates at the bottom of the IW with the same solvent or coagulant. This leads to the loss of large amounts of MOPW in the subsequent treatment and the problem of separation from the water.

That is why, based on our environmental research - analysis and the results of experimental work, we have achieved a new ecological approach to the deep purification of IW samples from ORI, which is of great economic and environmental importance, up to 100% from MOPW, dependent substances an effective chemical method has been developed (Ilyin et al., 2006; Hadjieva et al., 2021; Bayakhmetova et al., 2011; Sapina et al., 2011; Shapkin et al., 2012).

During the purification of IW, which is formed in ORI and has a very complex composition, in several stages, ie during the process of transporting IW by centrifugal pump to each stage, the hydrophobic emulsions of these waters increase, and a continuous dispersed system colloidal solution is arises.

Therefore, the maximum amount of IW included in mechanical treatment plants (300–1000 mg/l), ie the maximum amount of MOPW containing 100–250 mg/l MOPW, depending on the amount of emulsion of MOPW in the IW it is possible to carry out at least 3–5 stages in the mentioned devices.

That is why, with the new chemical method we are working on, in the initial stage of the existing mechanical treatment plants of ORI, regardless of the highest concentration of MOPW in IW, these waters can be cleaned up to 100% from MOPW, dependent substances and completely discolored achieving transparency has been identified (Ilyin et al., 2006; Hadjieva et al., 2021; Bayakhmetova et al., 2011; Sapina et al., 2011; Shapkin et al., 2012).

## **Materials and methods**

According to the new method developed by us, the degree of purification of the IW sample was also determined by the device QX-KS 6890–5975 (Manufacturer Agilent Technologies, USA). Thus, the analysis of the device was carried out in a system including an Agilent 6890N gas chromatograph which has an interface with an Agilent 5975 high-performance mass selective detector manufactured by Agilent Technologies (USA). The chromatograph is equipped with an injector without flow splitting and a ZB-5 capillary column (Phenomenex, USA). The ZB-5 column has the following specifications - copolymer 5% diphenyl 95% dimethylpolysiloxane, length 60 m, inner diameter 0.25 mm, film thickness 0.25  $\mu\text{m}$ . Helium (purity

99.999%) is used as the carrier gas at a flow rate of 1.5 ml/min. Ionizing source voltage 70 eV, source temperature 230°C, quadrupole temperature 150°C, injector temperature 270°C.

The amount of organic (PAH) compounds remaining in the treated water was 10 times less than the reference substances taken for spectral sampling, as can be seen from the spectrum wiped out by the device taken in Example VI. An overview of the spectrum device is shown in Figure 1.



**Figure 1. QX-KS 6890-5975 overview**

Chemical treatment of IW samples taken from the entrance of ORI mechanical treatment plants in different directions was carried out in the laboratory. During the experimental research works, petroleum ether as an extractant with high ecological effect, 5% solution of  $\text{Al}_2(\text{SO}_4)_3$  salt as a coagulant, 5%  $\text{H}_2\text{SO}_4$  solution as a flocculant and neutralizer and, if necessary, 5% - the optimal technological regime for the use of  $\text{NaHCO}_3$  salt as a neutralizer to obtain  $\text{pH}=7$  from 5 solutions was determined. That is why the IW sample was cleaned in the following direction by mixing in the following sequence and at each stage.

### **Experimental part**

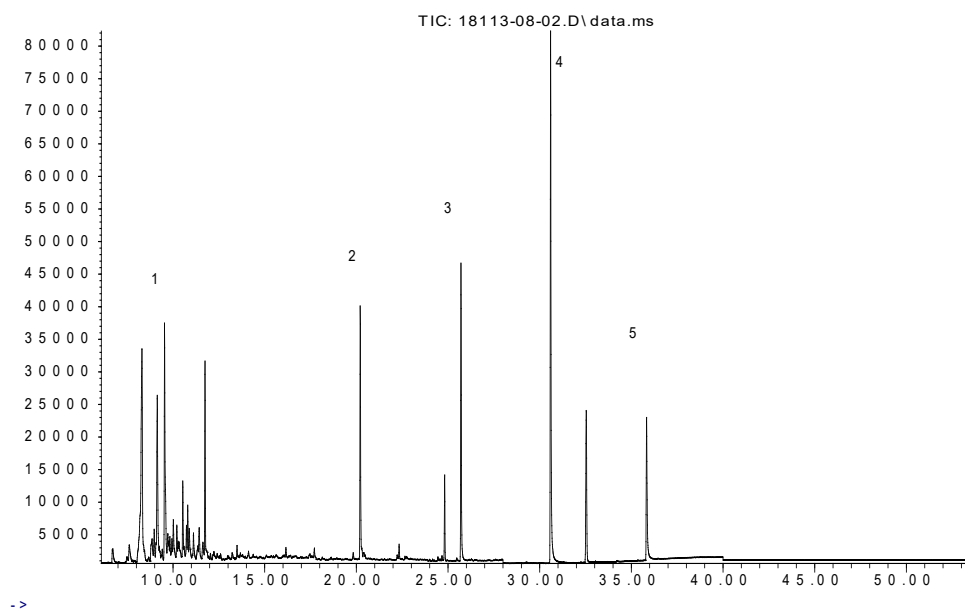
In the first stage: 1L IW sample is filled into the separating funnel, 0.5% of petroleum ether is added to it and mixed several times at 10–20.C for 0.5 minutes add 0.5% IW of 5%  $\text{Al}_2(\text{SO}_4)_3$  solution coagulant to the extracted mixture and mix for 1 minute.

To speed up and complete the coagulation process, a flocculant (simultaneously neutralizing) 5% H<sub>2</sub>SO<sub>4</sub> solution of 0.1% IW is added to the mixture and the mixture is stirred several times for 2 minutes. The coagulation process takes 15 minutes. The organic layer is separated from the water layer. The analysis of the amount of MOPW (total organic compounds) in the aquifer was performed by known methods and the spectrum was recorded with the device "QX-KS 6890-5975".

The spectrum captured by this device is shown in Figure 2 below.

## Conclusions and discussions

### Test T-VI



**Figure 2. Spectrum was recorded with the device "QX-KS 6890-5975".**

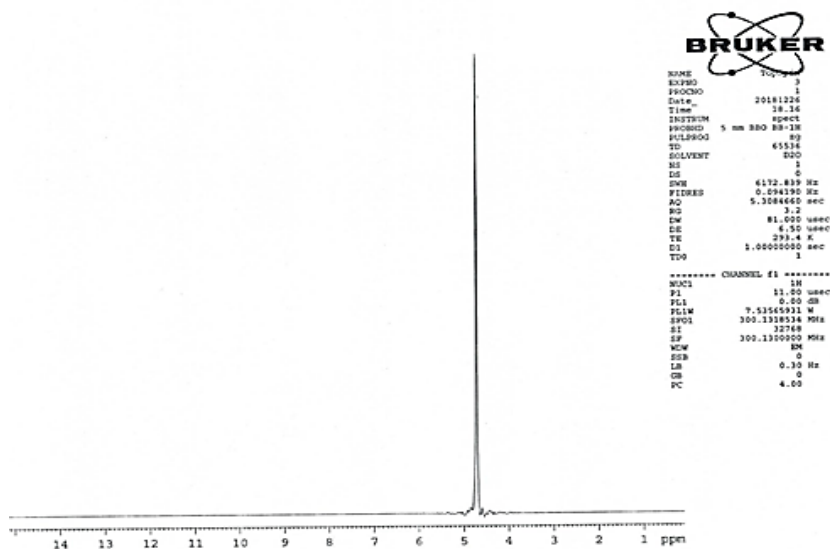
Peaks of the internal standard: 1.Naphthalene -d8; 2.Phenanthrene -d10;3.Pyrene -d10;4.Chrysen -d12';5.Washing machine -d12

The amount of organic matter remaining in the treated water sample was ten times lower than the not only environmental but also sanitary norms, as shown in Table 1. At the same time, the IW samples were spectrured on a BRUKER device to determine the amount of residual organic compounds in the water sample purified by the new method developed above by us.

**Table 1.** QX-KS 6890-5975 Concentration of individual PAHs automatically obtained on the device

Compound	naphthalene	acenaphthylene	acenaphthene	fluorene	phenanthrene	anthracene	fluoranthene	pyrene	Benzo(a)anthracene	chrysene	benzo(b,k)fluoranthene	benzo(a)pyrene	indeno(1,2,3-cd)pyrene	benzo(g,h,i)perylene	Dibenzo(ah)anthracene	Total EPA 16 PAH
T-VI (ug/l)	1.14	0.11	0.12	0.41	0.82	0.19	0.13	0.26	0.15	0.32	0.30	0.21	<0.01	0.07	<0.01	4.23

As can be seen from Figure 3, the hydrogen atoms in the water were obtained at high peaks. However, the peaks of the groups of hydrocarbon compounds remaining in the water are very small. This shows that the organic compounds in the IW sample purified by the new method are ten times lower than the environmental and sanitary norms. Thus, based on the above spectra and the results of the analysis of water treated by other gravimetric methods, it can be noted that up to one hundred percent treatment of oil industry wastewater can be carried out by this method. In addition to the above-mentioned experimental work, up to 5% of the IW purified to remove the extractant from the MOPW mixture obtained from the initial method of purification of IW samples of ORI is added to the IW in the separating funnel and mixed for 1 minute. It was then proved that up to 5% of the IW on the mixture could be used as a substitute for a coagulant solution from the purified IW obtained from the initial treatment and containing sulfate and chloride salts  $Al(OH)_3$ .

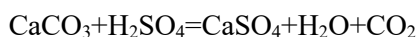
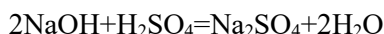
**Figure 3.** Spectrum of purified IW water sample taken with NMR BRUKER device.

In general  $\text{Al}(\text{OH})_3$  is formed by hydrolysis of the coagulant process after its addition to the IW sample after mixing, as noted in the literature (Ilyin et al., 2006; Hadjieva et al., 2021; Bayakhmetova et al., 2011; Sapina et al., 2011; Shapkin et al., 2012).



In this case, the metal compounds in the released  $\text{H}_2\text{SO}_4$  IW are converted by their hydroxides (as  $\text{pH}=9-10$ ) and their salts into sulfate salts. These salts are transferred to the water separated from the MOPW.  $\text{Al}(\text{OH})_3$ , on the other hand, sinks to the bottom of the water phase of the IW by attracting positively charged mechanical compounds..

To increase the speed of the coagulation process after the addition of an extractant, coagulant to the IW sample, as a flocculant, and also to bring the IW medium to  $\text{pH} = 9-10$  to  $\text{pH} = 7.0-7.5$ , using  $\text{H}_2\text{SO}_4$  acid.  $\text{NaOH}$ ,  $\text{CaCO}_3$ , etc.  $\text{H}_2\text{SO}_4$  reacts with compounds and enters the aqueous phase.



Using the new method developed based on the results of the research, it is possible to achieve the complete in-depth purification of the IW formed in the ORI from the required MOPW-dependent substances, as well as the transparency of these waters.

The application of this method in production can be considered scientifically based. During the experiment, it was determined that the process of coagulation of the IW sample, the effectiveness of the purification depends on the sequence of application of the extractant, the concentration and amount of coagulant, as well as the consistency and amount of flocculant. The results of the coagulation process, depending on the sequence and concentration of the reagents used in the developed method, are given in Figure 4–7. The results of the method developed for the environmental treatment of the IW sample of ORI are given in Table 2–4.

IW sample during purification 5%  $\text{Al}_2(\text{SO}_4)_3$  solution in the first stage, 5%  $\text{H}_2\text{SO}_4$  acid solution in the second stage and petroleum ether as an extractant in the third stage a graph showing the coagulation process when used is shown in Figure 5 below.

At the same time, the technological scheme related to the method of environmentally effective cleaning of NES in mechanical cleaning units of ITS is shown in figure 8.

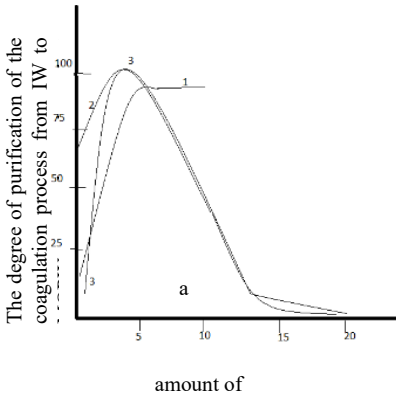


Figure 4. Dependence of the coagulation process (purification of MOPW and dependent substances from IW) on the amount of extagent (petroleum ether), coagulant 5% Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> solution and flocculant (5% H<sub>2</sub>SO<sub>4</sub> solution) 1) petroleum ether, 2) 5 % Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> solution, 3) 5% H<sub>2</sub>SO<sub>4</sub>.

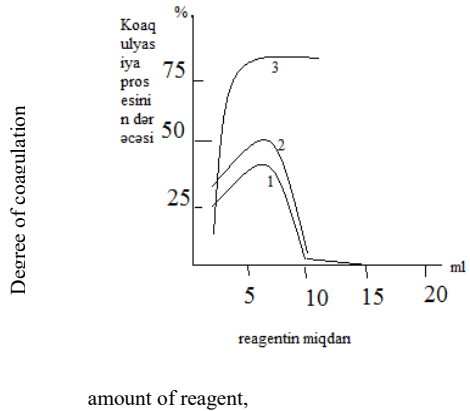


Figure 5. Dependence of the coagulation process (purification of MOPW and dependent substances from IW) on the amount of extagent (petroleum ether), coagulant 5% Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> solution and flocculant (5% H<sub>2</sub>SO<sub>4</sub> solution). 1–coagulant (5% Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> solution), 2– flocculant (5% H<sub>2</sub>SO<sub>4</sub> acid), 3– extractant (petroleum ether).

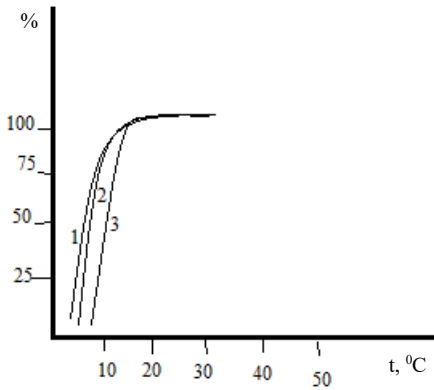


Figure 6. Graph of temperature dependence of the coagulation process. 1– extagent; 2– coagulant; 3– flocculant.

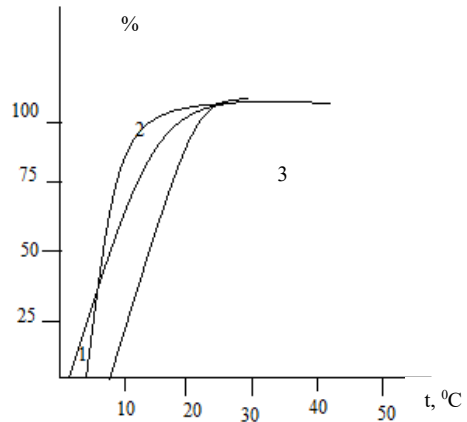


Figure 7. Time-dependent graph of the coagulation process. 1 – extagent; 2– coagulation; 3– flocculant

The table below shows the results of a new method developed for the environmental treatment of IW samples taken from ORI and those in which the amount of MOPW increased in those samples.



**Table 2.** The results of the method developed for the environmental treatment of the IW sample of ORI

IW-composition of samples															
Indicators before cleaning the sample					Reagents and mixtures used in cleaning							Indicators after cleaning			
Number and volume	MOPW amount and mg/l	The amount-of dependent substances is mg/l	Color	pH	Petro-ley ether ml	5% Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ml	5% H <sub>2</sub> SO <sub>4</sub> ml	K*-1	K*-2	Cleaning duration	XIMTQ amount	The amount to dependent substances is mg/l	Color	pH	Note
I 11	500-1000	100-150	Dark black	9-10	2.5	5	1.5	10.0	10.0	20	~0.001	~0.001	Transparent	7.03	
II 11	1000-2000	100-150	Dark black	9-10	2.5	5	1.5	10.0	10.0	20	~0.001	~0.001	Transparent	7.02	
III 11	2000-5000	150-200	Dark black	9-11	5.0	10	3	15.0	15.0	20	~0.001	~0.001	Transparent	7.04	An example taken in an emergency situation
IV 11	5000-10000	200-250	Dark black	9-11	5.0	10	3	20.0	20.0	20	~0.001	~0.001	Transparent	7.05	An example taken in an emergency situation

**Note:** K\*-1 MOPW, which was captured during cleaning and contained as an extractant, was used as an extractant substitute.

K\*-2purified IW was used as a coagulant substitute.

**Table 3.** Results of the developed method for the deep, new environmentally effective treatment of industrial waste water obtained in the oil refining industry

Examples of industrial waste water for ecological scientific research																		
Composition before treatment: pH, color and components used during treatment															pH, color, composition after treatment			
Conditional number №, volume	The amount of amount of mixture of waste oil products in the composition, mg/l	pH	color	EK-1, ml	EK-2, ml	EK-3, ml	EKN component, ml	5% K-1 solution, ml	5% K-2 solution, ml	5% K-3* solution, ml	after the components are given				pH	color	The amount of mixture of waste oil products in the composition, mg/l	Organic compounds mixture amount, mg/l
											m.p., min	o.p.o.l., min	p.c.c., min	t.c.p., min				
N-1, 1 l	500-1000	9-10	Deep black	0.3	0.7	–	–	5.0	2.0	0.3	2	3	5	15	7.3-7.4	clear	~ 0	<0.001
N-1, 1 l	500-1000	9-10	Deep black	–	–	1.0	–	5.0	1.0	0.3	2	3	5	15	7.3-7.4	clear	~ 0	<0.001
N-1, 1 l	500-1000	9-10	Deep black	–	–	–	5.0	5.0	1.0	–	2	3	5	15	7.3-7.4	clear	~ 0	<0.001
N-2, 1 l	1000<2000	9-10	Deep black	0.6	1.4	–	–	5.5-6.0	2.5	0.5	2	4-5	7	15	7.3-7.4	clear	~ 0	<0.001
N-2, 1 l	1000<2000	9-10	Deep black	–	–	2.0	–	5.5	2.5	0.5	2	5	7	15	7.3-7.4	clear	~ 0	<0.001
N-2, 1 l	1000<2000	9-10	Deep black	–	–	–	5.0	5.0	1.0	–	2	5	7	15	7.3-7.4	clear	~ 0	<0.001
N-3*, 1 l	2000<5000	9-10	Deep black	1.5	3.5	–	–	6.0	2.0	1.0	2	5	10	18	7.3-7.4	clear	~ 0	<0.001
N-3*, 1 l	2000<5000	9-10	Deep black	–	–	5.0	–	6.0	2.0	1.0	2	5	10	18	7.3-7.4	clear	~ 0	<0.001
N-3*, 1 l	2000<5000	9-10	Deep black	–	–	–	10-15	7.0	2.5	1.5	2	5	10	18	7.3-7.4	clear	~ 0	<0.001

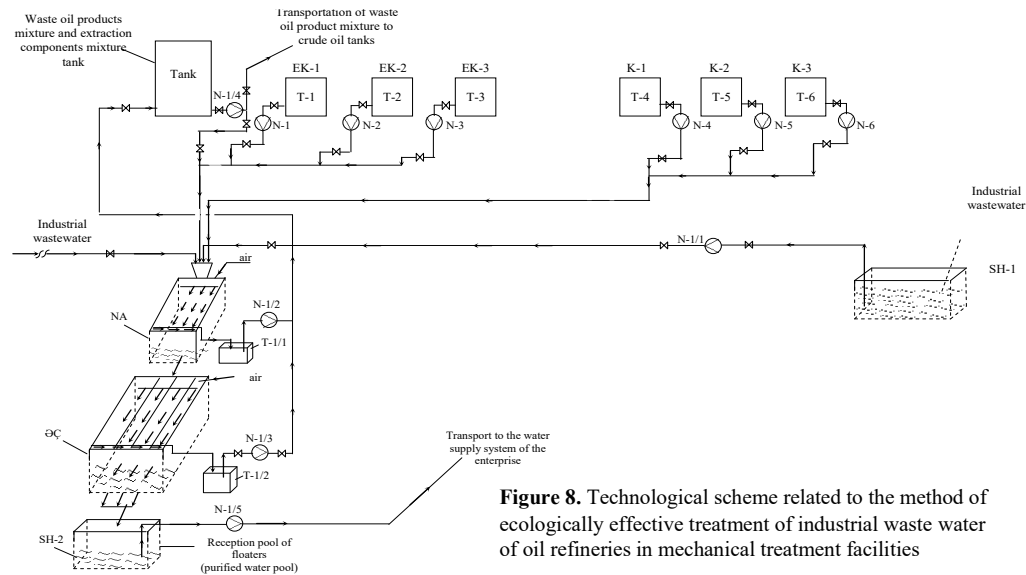
Note: EK-1 – extraction component;  
 EK-2 – extraction component;  
 EK-3 – a component that replaces the mixture of EK-1 and EK-2 extraction components;  
 EKN – mixture of waste oil products containing extraction and coagulation components obtained without purification;

m.p. – mixing period;  
 o.p.o.l. -obtaining period of organic layer;  
 p.c.c. -the period of color change;  
 t.c.p. –treatment completion period;

**Table 4.** Mode of the technological process, the application of a new ecologically effective method of deep treatment of industrial wastewater in the oil refining industry in mechanical treatment facilities

Components to be used for industrial wastewater treatment												The composition of industrial wastewater after treatment		
Volume flow rate	The amount of amount of mixture of waste oil products in the composition, mq/l	pH	the volume									The amount of amount of mixture of waste oil products in the composition, mq/l	Organic compounds mixture amount, mg/l	pH
			EK-1, ml	EK-2, ml	EK-3, ml	EKN component, ml	K-1 solution, ml	K-2 solution, ml	K-3* solution, ml	The period of the water color change	treatment completion period			
1000 m <sup>3</sup> /hour 24000 m <sup>3</sup> /day	500–1000 500–1000	9–10	0.3 l 7.2 l	0.7 l 16.8 l			5.0 l 120.0 l	2.0 l 48.0 l	0.1 l 2.4 l	20 min	20 min	0	<0.001	7.3–7.4
1000 m <sup>3</sup> /hour 24000 m <sup>3</sup> /day	500–1000 500–1000	9–10			1.0 l 24 l		5.0 l 120.0 l	2.0 l 48.0 l	0.1 l 2.4 l	20 min	20 min	0	<0.001	7.3–7.4
1000 m <sup>3</sup> / hour 24000 m <sup>3</sup> / day	500–1000 500–1000	9–10				5.0 l 120.0 l	5.0 l 120.0 l	2.0 l 48.0 l	0.1 l 2.4 l	20 min	20 min	0	<0.001	7.3–7.4
2000 m <sup>3</sup> / hour 48000 m <sup>3</sup> / day	1000–2000	9–10	0.6 l 14.4 l	1.4 l 23.6 l			10.0 l 240.0 l	4.0 l 96.0 l	0.2 l 4.8 l	20 min	20 min	0	<0.001	7.3–7.4
2000 m <sup>3</sup> /saat 48000 m <sup>3</sup> / day	1000–2000	9–10			2.0 l 48.0 l		10.0 l 240.0 l	4.0 l 96.0 l	0.2 l 4.8 l	20 min	20 min	0	<0.001	7.3–7.4
2000 m <sup>3</sup> / hour 48000 m <sup>3</sup> / day	1000–2000	9–10				10.0 l 240.0 l	10.0 l 240.0 l	4.0 l 96.0 l	0.2 l 4.8 l	20 min	20 min	0	<0.001	7.3–7.4
120000 m <sup>3</sup> / day	2000–5000	9–10			5.0 l 120 l		600.0 l	240.0 l	12.0 l	20 min	20 min	0	<0.001	7.3–7.4
120000 m <sup>3</sup> / day	2000–5000	9–10				600.0 l	600.0 l	240.0 l	12.0 l	20 min	20 min	0	<0.001	7.3–7.4

\* – when required.



**Figure 8.** Technological scheme related to the method of ecologically effective treatment of industrial waste water of oil refineries in mechanical treatment facilities

#### Explanation:

SH-1 – receiving water pool of industrial waste water of oil refineries  
 SH-2 – receiving water pool of treated industrial waste water  
 C – Waste oil products mixture and extraction components mixture tank  
 T-1 – tank for the extraction component EK-1  
 T-2 – tank for the extraction component EK-2  
 T-3 – tank for the extraction component EK-3  
 T-4 – tank for the component K-1  
 T-5 – tank for the component K-2  
 T-6 – tank for the component K-3  
 T-1/1 – underground capacity for accumulation of oil refinery waste mixture in oil separator neft ayrıcida  
 T-1/2 – underground capacity for accumulation of oil refinery waste mixture in additional precipitator  
 N-1/5 – The pump for the transfer of industrial waste water purified by a new method from the SH-2 pool to the circulating water system

N-1 – transfer pump for the component EK-1  
 N-2 – transfer pump for the component EK-2  
 N-3 – transfer pump for the component EK-3  
 N-4 – transfer pump for the component K-1  
 N-5 – transfer pump for the component K-2  
 N-6 – transfer pump for the component K-3  
 N-1/1 – Transportation of industrial wastewater from SH-1 to treatment plants  
 N-1/4 – transfer pump for the Waste oil products mixture and extraction components mixture from tank 1  
 N-1/2 – transfer pump for the Waste oil products mixture and extraction components mixture from T-1/1  
 N-1/3 – transfer pump for the Waste oil products mixture and extraction components mixture from T-1/2

NA – oil separator

ap – additional precipitator

## Conclusion

For the first time we have MOPW with the highest concentration of IW in mechanical treatment plants from a 5% solution of  $Al_2(SO_4)_3$  as a coagulant from petroleum ether as a coagulant in a specially defined amount of extractant, flogulant (in some cases also pH<sub>2</sub> 9–10 neutralizer) an environmentally friendly method has been developed to completely clean up to 100% of MOPW and dependent substances using 5%  $H_2SO_4$  acid as a neutralizer and 5%  $NaHCO_3$  as a neutralizer if required. Using this method, it is justified that there is no need to clean the IW formed in the ORI with flotates at other stages by physicochemical and biological purification methods. The new method is developed has of great ecological and economic importance.

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