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Research of Purification of Oil Sludge Waste Mixture Using $Fe_2(SO_4)_3$ As A Coagulant

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Abstract
During the treatment of industrial waste water (IWW) formed in technological processes at the Heydar Aliyev refinery at the plant's treatment facilities, a deposition of oil sludge waste mixture (OSWM) with various composition and properties takes place on the bottom of these facilities. The definition of composition and properties of OSWM is not the main purpose of this research work. But according to the information given in the technical literature, it can be noted that OSWM contains ~5-90% oil (or oil products), 1-52% water, 0.8-65% solid impurities, the density of oil sludge is 1.5-2 g/cm^3, pour point from -3°C to +80°C, flash point from 35°C to 120°C. Not only in our country, but throughout the world the problem of deep (up to 100%) ecologically effective purification of this mixture from the oil products waste mixture (OPWM) has not been completely solved until now. Despite the use of various coagulants ($AlCl_3$, $FeCl_3$, polyacrylamides, polyaluminum silicate chloride, polyferric silicate chloride), the maximum efficiency of purification of OSWM from OPWM was ~95% when carrying out the purification process in several stages. We conducted a study of purification of this mixture samples taken from the input of "Alpha-Laval" facility, processing OSWM at the above-mentioned plant (from the area where OSWM was collected for processing) using $Fe_2(SO_4)_3$ as a coagulant. The choice of this coagulant is explained by the fact that the purification process of OSWM is simple, economically beneficial, meets all environmental safety requirements, and is carried out at room temperature. Based on the results of the study, it was established that the highly effective purification of the OSWM sample using a solution of $Fe_2(SO_4)_3$ as a coagulant initially depends on the choice of an extractant, the volume of a coagulant, mixing time, the development of special optimal conditions. Treatment of OPWM from the OSWM up to 96.87% was achieved. Optimal conditions specially developed for this consist of using 5 ml of a primary gasoline fraction which is cheaper than petroleum ether as an extractant, 25 ml of a 5% solution of $Fe_2(SO_4)_3$ as a coagulant, 10 ml of H_2SO_4 as a flocculant and interval mixing of the mixture during 6 hours at room temperature..
Keywords: Oil sludge, coagulant, extractant, production, waste water, petroleum ether, gasoline fraction, optimal conditions.

Introduction

As noted in the scientific and technical literature, the 100% separation of the oil or OPWM contained in the oil sludge waste formed in the oil production and refining industry has not been achieved so far. It can be noted that the OSWM formed in the oil refining industry is very complex due to the composition and nature in comparison with the oil sludge waste of the oil production industry. That is why, together with IWW, which is formed during various technological processes in the oil refining industry, oil sludges with various composition enters the treatment facilities of the enterprise. The methods used in production and also developed in the research works on the separation of OPWM from the OSWM accumulated at the bottom of these treatment facilities by many researchers earlier for the stated purpose are not 100% effective.

As mentioned in the literature (Bagryantsev, et al., 1995; Vaisman, 2010), the deep and ecologically effective cleaning of OSWM with modern methods has not yet been fully achieved, because each of them has ecological and economic shortcomings.

At present, in general in the oil industry, a large amount of energy and financial costs are spent during the operation of each facility that carries out the processing of the OSWM (the separation of oil or OPWM from its composition).

Taking into account the brief explanations given above, we carried out research works on the ecologically effective purification of the OSWM from the OPWM in several directions in order to determine the purification effectiveness of coagulant substances. One of these research works was carried out using $Fe_2(SO_4)_3$ in accordance with the research works developed by us and noted in the literature (Bayramov, et al., 2018; Osmanova, 2022), the study on the purification of the OSWM sample was carried out in the following way.

Experimental Parts

In accordance with the research works presented in the literature (Bayramov, et al., 2018; Osmanova, 2022), we report the progress of determining the effectiveness of treatment of the mentioned sludge waste with complex composition with this substance by using the initial gasoline fraction as an extractant and a 5% solution of $Fe_2(SO_4)_3$ salt as a coagulant for chemical treatment of the OSWM sample in the laboratory conditions.

Based on the results of the research conducted by us, 50 g of the OSWM is placed in the reaction flask and 200 g of unrefined IWW from the plant is added to it and mixed during 1 hour. Then the mixture is filled into the separating funnel and mixed. 5 ml of the initial gasoline fraction is added to

the mixture and mixed at the room temperature with breaks during 2 hours. In each experiment, 5% solution of $Fe_2(SO_4)_3$ is added to the mixture in the quantities indicated in the table 1 and mixed with breaks during 4 hours.
After the process of obtaining three phases (solid, water and organic layers) in the separating funnel is completed, each of these layers is filled into small-volume glass flasks, the weight of which is determined in advance, and then the quantity of these layers is determined. The degree of purification of oil sludge waste from the OPWM by the method mentioned above was calculated using the indicators of the specified quantities of the OPWM, water and mechanical mixture contained in the OSWM sample used during treatment.

Result

The results of the research on purification of the OSWM from the OPWM using 5% solution of $Fe_2(SO_4)_3$ are presented in the table 1.

Table 1. The results of the research on purification of the OSWM from the OPWM using 5% solution of $Fe_2(SO_4)_3$

The OSWM sample								effectiveness of the purification with other coagulants, %	
quantity, g., purification temperature	composition before purification			reagents used for the purification		composition after purification		effectiveness of purification with $Fe_2(SO_4)_3$, %	$FeSO_4$	$FeCl_3$
	quantity of the OPWM%	quantity of water %	quantity of the mechanical mixture%	volume of an extractant, ml	volume of a 5% solution of $Fe_2(SO_4)_3$, ml	quantity of the OPWM, %	purification time, hour			
50,	60,15	24,01	15, 84	5,0	5,0	66,46	6	33,5	19,6	34,63
					10,0	48,95	6	51	37,19	51,46
					15,0	09,24	6	90,8	61,33	81,39
					20,0	05,73	6	94,3	89,82	84,98
					25,0	03,18	6	96,8	70,31	88,58
					30,0	16,57	6	83,4	36,91	48,86
					35,0	29,16	6	70,8	13,13	37,49
					40,0	28,54	6	61,5	3,38	27,74

As can be seen from the Table 1, the effectiveness of purification of the OSWM sample from the OPWM decreases if, after addition of the extractant

to the mixture, the volume of 5% solution of $Fe_2(SO_4)_3$ is more than 25 ml. Thus, the above-mentioned optimal conditions and 96.82% effectiveness of purification of the OSWM from the OPWM can be achieved by using mainly 5% solution of $Fe_2(SO_4)_3$ as a coagulant.
The graph of the dependence of the coagulation process effectiveness during the purification of the OSWM from the OPWM on the concentration of the coagulant is shown in Figure 1.

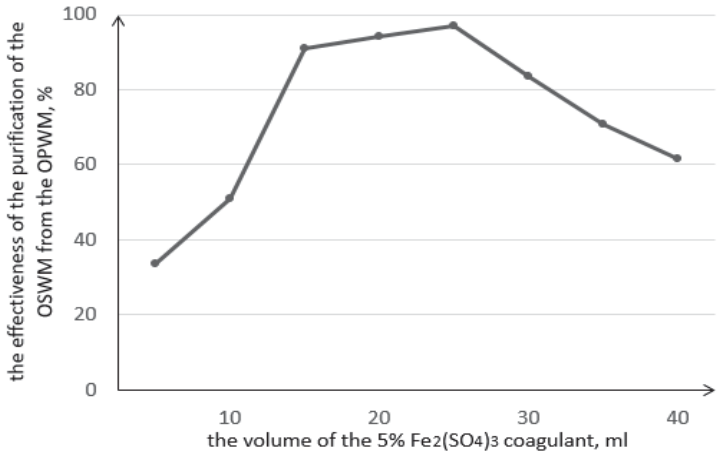

Figure 1. The graph of the dependence of the coagulation process (effectiveness of the purification of the OPWM) on the volume of 5% $Fe_2(SO_4)_3$ coagulant

The presented research work is one of the research works carried out in order to determine the effectiveness of coagulants used for chemical (coagulation) purification of the OSWM samples formed at the Heydar Aliyev refinery and taken from the field of processing at the "Alpha-Laval" facility.
There is $Fe(OH)_3$ together with sulfate salts in the IWW obtained during the process of purification of the OPWM from the OSWM using the initial gasoline fraction as an extractant, a 5% solution of $Fe_2(SO_4)_3$ salt as a coagulant, and the separated OPWM contains an extractant. Therefore, during the next purification of the OSWM the possibility of using of the IWW containing $Fe(OH)_3$ instead of a coagulant up to 15% of the slurry mixture has been determined. At the same time, in the next process of purification of the OSWM research work was carried out on the possibility of using of the OPWM containing gasoline fraction instead of an extractant up to 5% of the purified slurry mixture. The obtained results were consistent with the results of the previous research work.

Scientific explanations about the reason for the occurrence of the coagulation process are widely interpreted in the literature (Bayramov, et al., 2018; Bayramov, et al., 2019; Osmano, 2020, Osmanov, 2022). Based on these explanations, it can be noted that when a 5% solution of $Fe_2(SO_4)_3$ salt is used as a coagulant, this substance is hydrolyzed in the aquatic environment in a short period of time and $Fe(OH)_3$ is obtained.

$Fe_2(SO_4)_3 + 6H_2O \leftrightarrow 2Fe(OH)_3 + 3H_2SO_4$

$SO4^{-2}$ anions react with carbonates in the environment and various sulfate salts are obtained. The compound $Fe(OH)_3$, formed in a short period of time, collapses at the bottom part due to the attracting solid particles to itself like $Al(OH)_3$ and increasing of gravity force. When the concentration of $Fe_2(SO_4)_3$ coagulant is too high, the amount of $Fe(OH)_3$ multiplies. Therefore, $Fe(OH)_3$ precipitates attracting the sludge particles containing OPWM, to itself. In this case, regardless of the amount of the extractant, the speed of coagulation process decreases and the separation of the OSWM from the OPWM also decreases.

Based on the results of the research, it can be noted that in the oil refining industry using of a 5% solution of $Fe_2(SO_4)_3$ salt as a coagulant in the way mentioned above is more preferable according to the financial and environmental indicators than the facilities that carry out the processes of separation of OSWM from OPWM, refining and are currently in operation.

That is why, in accordance with the research work carried out by us, it can be considered scientifically based that the purification of the OSWM formed in the oil refining industry from OPWM using $Fe_2(SO_4)_3$ has great economic and environmental importance.

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