OILFIELD PRODUCED WATER - CHEMICAL COMPOSITION AND ASSESSMENT OF ITS IMPACT ON THE ENVIRONMENT

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ABSTRACT. Waste water from crude oil production is often polluted by organic compounds, suspended solids, heavy metals and microorganisms. On one side discharge of oilfield produced water into natural water bodies could lead to environmental pollution and on the other side the contaminants hamper reuse of the water. The article presents the results of analysis of produced water from an oilfield in the Republic of Bulgaria - in terms of chemical composition, physicochemical and microbiological parameters. The data obtained show that produced water meets the legislation requirements and it cannot be considered as a source of environmental pollution. Field observations and analytical results revealed that most probably the microbial activity causes clogging and corrosion of the enterprise pipelines.

Keywords: oilfield produced water, oily wastewater

ОТПАДЪЧНА ВОДА ОТ НЕФТОДОБИВ – ХИМИЧЕН СЪСТАВ И ОЦЕНКА НА ВЪЗДЕЙСТВИЕТО Й ВЪРХУ ОКОЛНАТА СРЕДА

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РЕЗЮМЕ. Отпадъчната вода от добив на суров нефт често е замърсена с нефтопродукти, неразтворени вещества, феноли, тежки метали и микроорганизми. Заустването й в естествени водни тела би могло да доведе до екологични проблеми. Замърсяването затруднява оборотното използване на водата. В статията са представени резултатите от анализ на проби от реална производствена отпадъчна вода от предприятие за добив на нефт на територията на Р България – по отношение на химичен състав, физикохимични и микробиологични показатели. Получените данни показват, че отпадъчната вода отговаря на изискванията на законодателството и не може да бъде евентуален източник на замърсяване на околната среда. Въз основа на теренни наблюдения и резултатите от анализите, може да се заключи, че микробната активност е основна причина за намаляване на полезно сечение на тръбопроводите на предприятието.

Ключови думи: отпадъчна вода от добив на суров нефт, отпадъчна вода, замърсена с нефтопродукти

Introduction

Despite the development of renewable energy sources and their implementation, nowadays industry and everyday life still needs increasing amounts of crude oil. Oil well production fluid usually consists of oil and water that is generally separated by physical techniques. The water stream is referred to "oilfield produced water" or in broader sense - oily wastewater.

The composition of produced water depends mainly on the oilfield geological conditions, the recovery method and the age of the production wells. Although concentrations of different pollutants can vary by order of magnitude, the contaminants of produced water can be divided into the following groups: dissolved and dispersed oils, dissolved formation minerals, production chemicals, production solids, and dissolved gases (Fakhru'l-Razi et al., 2009). Oil is a natural mixture of hydrocarbons, such as benzene, toluene, ethylbenzene and xylenes (BTEX), naphthalene, phenantherene, dibenzothiophene (NPD), polyaromatic hydrocarbons (PAHs) and phenols. Water-soluble are the polar constituents, distributed between the low and medium carbon ranges. BTEX and phenols are the most soluble compounds in produced water. Organic acids (formic and propionic) are typically found in produced water. Aliphatic hydrocarbons, phenols, carboxylic acid and low molecular weight aromatic compounds are most often included as soluble oil compounds in produced water. Usually PAHs and some of the heavier alkyl phenols (C6–C9 alkylated phenols) are less soluble in produced water and present as dispersed oil. Dissolution of formation minerals leads to availability of inorganic anions and cations (including heavy metals - cadmium, chromium, copper, lead, mercury, nickel, silver, and zinc), and radioactive materials in produced water. Salt concentration can vary from a few parts per million (ppm) to about 300,000 ppm. Sulphate concentration in produced water is generally lower than in seawater. Radium-226 and radium-228 are the most abundant naturally occurring radioactive materials in produced water. Most often they co-precipitate with barium sulfate. Production chemicals (such as scale and corrosion inhibitors, biocides, emulsion breakers, antifoam, water treatment chemicals, etc.) are added to treat or prevent operational problems. A wide range of polar and charged molecules are used (such as alkylbenzen sulfonate (LAS), alkyldimethylbenzenylammonium compounds, 2-alkyl-1-

ethylamine-2-imidazolines, 2-alkyl-1-[N-ethylalkylamide]-2-imidazolines, and di-[alkyldimethyl-ammonium-ethyl]ether), however their concentration in produced water usually is as low as 0.1 ppm (Veil et al., 2004). Production solids are a wide range of materials (formation solids, corrosion and scale products, bacteria, waxes, and asphaltenes). In anoxic produced water, sulfides (polysulfides and hydrogen sulfide) are generated by sulfate reducing bacteria (Neff, 2002). Since different toxic chemicals are available in produced water, few microorganisms can survive. Some analyses show that there are 50–100 cells of microorganisms per mL, in which the majority of microorganisms are aerobic Gram-positive bacteria (Weidong et al., 2001). Bacteria can cause corrosion of equipment and pipelines or clog those (Veil et al., 2004). Some inorganic crystalline substances (SiO_2, Fe_2O_3, Fe_3O_4, and $BaSO_4$) are found in the suspended solids (SS) in produced water (Shubo et al., 2009). Usually dissolved CO_2, O_2 and H_2S may be found in it (Fakhru'l-Razi et al., 2009). Produced water is the largest waste stream generated in oil industry (Hosny et al., 2015).

Table 1 summarizes the range of produced water characteristics in different oilfields in the world (Tibbetts et al., 1992; Bessa et al, 2001; Li et al, 2006; Lu et al., 2006; Ahmadun et al., 2009; Li et al, 2010; Hosny et al., 2015).

Table 1.
Range of produced water characteristics in different oilfields in the world

Parameter	Value	Parameter	Value
pH	4.3 – 10.0[1]	Phenols, mg/L	0.009-23
COD, mg/L	274-2517	Chlorides, mg/L	274-2517
BOD_5, mg/L	11-21[2]	Sulfates, mg/L	2-1650
TOC, mg/L	0-1500	Bicarbonates, mg/L	77-3990
TSS, mg/L	1.2-1000	Sulfides, mg/L	0.14[4]
TDS, mg/L	675-141522	Sodium, mg/L	132-97300
O&G, mg/L	31-275	Potassium, mg/L	24-4300
TPH[3], mg/L	49-64	Calcium, mg/L	13-25800
Total polar, mg/L	9.7-600	Magnesium, mg/L	8-6000
Higher acids, mg/L	<1-63	Barium, mg/L	0.25-650
Volatile, BTEX, mg/L	0.39-35	Strontium, mg/L	0.02-1000
Total oil (IR), mg/L	2-565	Heavy metals[5], mg/L	0.068-48.38

[1] Most often: 6.7 – 7.4
[2] Only Li et al., 2010
[3] Total petroleum hydrocarbons
[4] Only Lu et al., 2006
[5] Considered elements in this case are: Sb, As, Be, Cd, Cr, Cu, Pb, Hg, Ni, Ag, V, and Zn

Produced water is considered an oilfield waste which has to be managed in the following hierarchy: a) minimization of produced water production; b) reuse and recycling, and c) disposal - as the last desired option. Reuse and recycling of produced water comprise underground injection applied for increasing the oil production; use for irrigation, livestock or wildlife watering and habitats, and various industrial uses (such as dust control, vehicle washing, power plant makeup water, and fire control) (Veil et al., 2004). Reuse in oil operation can be applied after treating the produced water to meet the quality required to prevent / reduce formation damage. When produced water is used for irrigation and restoration, or for cattle and animal consumption it should be treated to meet the corresponding standards.

When discharge is the applied option, the produced water has to be treated to meet the environmental standards. Discharge of non-treated or non-sufficiently treated oily wastewater can affect the environment in the following aspects: (1) polluting drinking water and groundwater resources, endangering aquatic life; (2) endangering human health; (3) atmospheric pollution; (4) affecting crop production; (5) destructing the natural landscape. Even safety issues may arise due to coalescence (Wenhu et al., 2013).

Salinity is considered as a major contributor of toxicity of the oil produced water (Neff et al., 2002). Dispersed oil and droplets increase the biochemical oxygen demand (BOD) of the affected water. They rise to the surface of water, where volatile and/or toxic compounds evaporate. Nonpolar hydrocarbons and hydrogen sulfide in produced water are toxic. Some production chemicals can increase partitioning of oil compounds into the aqueous phase at high concentrations (Henderson et al., 1999). Heavy metals toxicity is less than that of nonpolar organics in produced water (Elias-Samlalsingh and Agard, 2003).

The environmentally acceptable disposal of oily wastewater is a current challenge to the petroleum industry. As it can be seen from Table 1, the composition of produced water significantly differs for different oilfields. In order to implement a proper management of the produced water and to protect the environment, the parameters of the specific wastewater have to be investigated and monitored on regular basis. That is why the present work is concentrated on studying the characteristics of produced water from an oil field in Bulgaria, as well as to assess the environmental impact of water discharge.

Methods and materials

Six samples (noted as No 1, 2, 3, 4, 5, and 6) were collected from six discharge points of a Bulgarian oil extracting company. Temperature, pH value, ΔpH value (index of water saturation with respect to $CaCO_3$), Eh value, and specific conductance were measured at sampling points by using electrometric methods (with combined electrodes and WTW Multi 340i/SET device). Collected samples were preserved following the standard procedures and were transported in a cooling bag to the laboratories where other parameters were measured.

In the course of sampling the pipes clogging and corrosion were observed.

Dissolved oxygen was determined by using Winkler method. Hardness, respectively concentration of calcium and magnesium ions, was found by titration with EDTA. Concentration of chloride and bicarbonate ions were determined by titrimetric methods - titration against silver nitrate (Mohr's method) and hydrochloric acid solution (methyl orange method), correspondingly. Turbidimetric method was applied to determine the concentration of sulfates. Concentration of metals, selenium and arsenic were determined by ICP-AES. Oil hydrocarbons concentrations were determined by spectrophotometric method in UV range, after their separation by extraction with CCl_4. Total suspended solids (TSS) were found by weight method as samples were dried at 103-105 ^oC for 2 hours (ASTM, 1979; EPA, 1983; BSS, 1989; APHA, 1992; Krawczyk, 1996; UNEP/WHO, 1996).

Oil and grease (O&G) was measured following EPA Method 1664, by extraction in n-Hexane (EPA, 2010). Only two samples were analysed – number 2 and 6.

Total organic carbon (TOC) was determined by catalytic combustion method (Pt catalyst, 680 ^oC) using a TOC Analyzer SHUMATZU model TOC-VCSH.

Phenol amount was determined spectrophotometrically by using Spectroquant NOVA 60 and Spectroquant phenol test. The method is analogous to EPA 420.1, APHA 5530 C+D and ASTM D 1783-01.

Suspended solids mineralogical composition was determined by X-ray diffractometer (Dron - UM1, Russia) under the following conditions: 2 Theta / 5 \ + (0) -85 \ + (0); Cu / Ni; 30 kV / 20 mA; 0.09 / 1 s. Suspended solids of sample No 2 were analyzed.

Chemical oxygen demand (COD) was determined spectrophotometrically by using Spectroquant NOVA 60, a Merck COD cell test, after chloride depletion and sample digestion in a Spectroquant TR420 device. The method corresponds to DIN38409-41-2, DIN ISO 1575 and is analogous to EPA410.4, APHA 5220 D, and ASTM D 1252-06 B. Biological oxygen demand, 5 days, 20 ^oC (BOD_5) was determined following widely accepted standard procedures (Young et al., 2003).

Count of viable microbial cells was made by the plate or liquid media count methods (Parks and Roland, 1997). Aerobic heterotrophic bacteria were counted by plating on agar, as three replicates were made for each dilution. A three-tube most-probable number technique was applied for estimation of the number of anaerobic heterotrophic bacteria, bacteria fermenting sugars with gas production, denitrifying bacteria and sulphate-reducing bacteria.

Results and discussion

Data on integral physicochemical parameters of the studied samples are presented in Table 2. The values of concentration of macro-components and micro-components respectively are summarized in Table 3 and Table 4.

By comparing values presented in Tables 1 and 2, it may be stated that the pH value of studied water is in the range of the typical values. Having in mind the existing relation between salts concentration and specific conductance, it is clear that studied produced water is practically in the low edge of the worldwide reported salt concentrations of produced water. Measured Eh values are indicative for anaerobic conditions, in accord with very low concentrations of dissolved oxygen in most samples (Table 2). The fast ΔpH test showed that waters are oversaturated with respect to calcium carbonate and pipes corrosion, due to the impact of CO_2 forms in water, is not expected.

As it is typical for produced oilfield water, Na^+ is the major cation and Cl^- is the major anion found in studied waters (Table 3). Amounts of all water macro-components are in the low edge of the worldwide reported concentrations in produced water (Tables 1 and 3).

Table 2.
Integral physicochemical parameters of the studied water samples

No	t, ^oC	pH	Eh, mV	χ, mS/cm	ΔpH	Diss.O_2 mg/L
1	28.0	7.86	+164	3.28	+0.40	0.42
2	26.5	7.59	-52	3.38	+0.08	<0.08
3	26.0	7.78	-40	3.92	+0.27	<0.08
4	27.8	7.61	-92	4.05	+0.13	<0.08
5	28.4	7.73	+176	4.28	+0.15	0.51
6	28.2	7.55	-104	6.08	+0.13	<0.08

Table 3.
*Macro-components of the studied sample, mg/L**

No	1	2	3	4	5	6
Ca^{2+}	84	85	88	85	86	110
Mg^{2+}	32	32	40	40	40	52
Na^+	605	681	770	823	805	1273
K^+	206	204	198	202	210	220
Cl^-	1064	1165	1413	1314	1387	2133
HCO_3^-	480	480	520	527	488	615
SO_4^{2-}	20	50	5	20	32	21
Hardness	3.4	3.4	3.8	3.8	3.8	4.9

*Hardness – in mmol/L

Table 4.
Micro-components of the studied water samples, mg/L

No	1	2	3	4	5	6
As	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Be	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cd	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cr	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cu	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Pb	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Hg	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Ni	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Se	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Ag	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Tl	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
V	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Zn	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05

Studied waste water can be classified as hard water. Attention is drawn to the relatively low concentrations of SO_4^{2-}. Future work has to be carried out in order to be elucidated whether this fact is due to precipitation of sulfates (mainly calcium sulfate) or to sulfate reduction either to sulfur or to sulfide ion, the latter followed by precipitation of sulfides. The hypothesis for possibility of heavy metals sulfides precipitation is supported also by the data on the concentration of heavy metals in studied waters (Table 4). Slightly alkaline pH values also may facilitate the precipitation of heavy metals.

Table 5 summarizes data on some integral chemical parameters of the analyzed water.

Table 5.
Integral parameters of the studied water samples, mg/L

No	COD	BOD_5	Oil hydro-carbons	Phenols	TSS	TOC
1	13	5.91	2.4	0.020	5	8.76
2	44	36.18	10.7	0.079	40	24.38
3	26	7.86	3.9	0.004	5	10.86
4	27	11.96	3.9	0.006	2	18.76
5	20	5.16	3.4	0.017	170	9.37
6	41	19.16	5.2	0.019	20	12.14

Measured COD values are below those reported for non-treated produced water by other authors (compare with Table 1). Data for COD point out that all studied samples show low concentration of chemically oxidizable (by dichromate) ions and compounds. Total suspended solids also were practically at the low edge of concentrations cited by other works (see Table 1).

Determined concentrations of oil hydrocarbons and phenols are below the limits of the Bulgarian legislation. Described results, combined with the found correlation between the concentrations of determined phenols and hydrocarbons (coefficient of correlation r = 0.912), hinted to the idea that probably heavy organic compounds present in the waste water as tiny droplets. They might be determined as total amount of oil and grease (O&G) by extraction in hexane. That is why two samples were analyzed for O&G content. O&G found in sample No 2 was 32 mg/L and in sample No 6 – 135 mg/L. These values are in the range of concentrations, pointed by other authors (cf. with Table 1). Obviously the heavy organic compounds are more soluble in hexane than in tetrachloromethane and the concentration of O&G, especially for sample 6, is significantly higher than that of oil hydrocarbons. The finding is consistent with the high density of the crude oil reported as 0.9383 g/cm^3 by Balinov (Balinov, 1980). We have determined an average value of 0.952 g/cm^3. The oil can be classified as heavy crude oil - with density in the range of 0.88 – 1.00 g/cm^3 (Fingas, 2015). It is supported also by the brief gas chromatographic measurement that shows not only typical low weight mass hydrocarbons but also compounds having molar mass higher than 300 g/mol and corresponding to polycyclic aromatic hydrocarbons (Marvin, 1999). According to Fingas (2015), heavy crude oil is rich in tri- to pentacyclic terpanes and aromatic steranes.

The X-ray powder diffraction of TSS of sample No 2 revealed the presence of the following solid materials: sulfur (S^o, S_6, S_8); iron sulfides (FeS, FeS_2); heavy metal sulfides (Cu_2S, CuS, MnS, MnS_2); iron oxides (Fe_2O_3, Fe_3O_4); sulfates and chlorides [$Fe(NH_4)_2(SO_4)_2(H_2O)_6$ and $NH_4MgCl_3.6H_2O$], silicates [($Ca_2(MgFe^{2+})_5Si_8O_{22}(OH)_2$ and $NaAl_2(OH)_2AlSi_3O_{10}$] and carbonates ($CaCO_3.MgCO_3$). Although we obtained X-ray data for only one sample, we may propose that most likely the problems with pipes clogging and corrosion are due to electrochemical corrosion. Initially the cathodic depolizer is the dissolved oxygen and in the later stage – microbially produced sulfide ions, which precipitate metal sulfides, such as iron sulfide, manganese sulfide, and copper sulfide.

BOD_5 parameter varies between 5.16 and 36.18 for the series of samples and those values are similar to reported by other authors (Li at al., 2010). On the other side these are relatively low values found in wastewaters and meet national requirements. A good correlation between BOD_5 values and the concentrations of determined hydrocarbons (coefficient of correlation r = 0.974) was found. This implies that probably natural microorganisms in the produced water are living on energy from bio-oxidation of some of the available hydrocarbons.

The numbers of different groups of microorganisms in the studied waters are presented in Table 6.

Table 6.
Microbiological analysis of the studied water samples

Groups of micro-organisms	Cells/mL					
	1	2	3	4	5	6
Aerobic heterotrophic bacteria	1.5×10^2	4.4×10^3	1.4×10^2	1.1×10^2	5.1×10^2	4.8×10^3
Anaerobic heterotrophic bacteria	2.5×10^1	4.0×10^3	4.5×10^2	3.0×10^2	7.5×10^2	9.5×10^3
Bacteria fermenting sugars with gas production	$<10^1$	$<10^1$	$<10^1$	$<10^1$	$<10^1$	$<10^1$
Denitrifying bacteria	$<10^1$	7.5×10^2	$<10^1$	$<10^1$	6.5×10^2	4.5×10^3
Sulphate-reducing bacteria	$<10^1$	6.5×10^2	9.5×10^2	2.5×10^1	$<10^1$	6.5×10^2

Data, presented in Table 6 show that the amount of microflora is directly dependent on the content of biodegradable organic substances in water. The highest number of aerobic and anaerobic heterotrophic bacteria were found in the water samples No 2 and No 6, in which the values of BOD_5 are higher. The lack of bacteria fermenting sugars with gas production indicates that the organic compounds in water samples possibly are hydrocarbons and their derivates - mainly organic acids and alcohols.

Sulfate-reducing bacteria (SRB) were found in the samples, which are characterized by low Eh value. Despite that the concentrations of the sulfates in the water samples were low (in the range of 5 to 50 mg / L) the number of these bacteria was about 10^2 cells / mL. The presence of SRB in some of the samples is related to their ability to use a variety of oil hydrocarbons and phenols as source of carbon and energy. Since hydrogen sulfide is the product of the sulfate-reduction process, the corrosion problems of the pipes may be due to the microbial activity of these bacteria.

The presence of denitrifying bacteria in some of the samples indicated the possible presence of their final electron acceptor – nitrate.

Requirements toward the oil-drilling companies for discharging produced water or re-injected water are given by the Decree 6 (Bulgarian Council of Ministers, 2000). The main parameters of wastewater that must be followed and the limits which must not be exceeded are given in Table 7.

Table 7.
Maximum permissible concentrations / levels of water pollutants subjected to observation in oilfield production water intended for discharge (Bulgarian Council of Ministers, 2000)

Parameter	Threshold value
pH	6 - 9
TSS	50 mg/dm^3
BOD_5	50 mg/dm^3
Oil hydro-carbons	20 (40[1]) mg/dm^3
Phenols	1.0 mg/dm^3
Sulfides	1.0 mg/dm^3
Heavy metals (totally)[2]	5.0 mg/dm^3

[1] - at production capacity less than 10000 t/day
[2] - Sb, As, Be, Cd, Cr, Cu, Pb, Hg, Ni, Se, Ag, Tl, V, and Zn.

The comparison between the upper limits given in Table 7 and the values found and presented in Tables 2, 4 and 5 shows that all water samples meet the legislation criteria (except for sample 5, TSS) and they cannot be a source of environmental pollution. Many oil and gas companies around the world are working towards the implementation of "zero-discharge" of contaminants in produced water in accordance with the EU water framework directive (EU, 2000). Since then, the environmental requirements have been strengthened with respect to oil hydrocarbons (up to 5 mg/dm^3 in some countries). Therefore, in the future produced water from stations 2 and 6 could pose some problems.

Conclusions

A wide range of parameters of oilfield produced water were determined and analyzed.

Studied produced water samples meet the legislation and cannot be considered as a source of environmental pollution.

Based on field observations and analytical results, most probably, the microbial activity is the main reason for clogging and corrosion of the enterprise pipelines.

Further studies are needed to reveal the mechanism of the microbial effect and the eventual impact of heavy oil hydrocarbons on the company pipes and equipment and on the environment.

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