

# ASP project. Problematics of dissolved oxygen. Theory and practice

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**Abstract.** The article presents the latest results of studies of dissolved oxygen affect on efficiency of ASP flooding project being executed by Salym Petroleum Development N.V. Company. Pilot project associated with experimental injection of solutions of anionic surface-active substance (surfactant), soda and polymer into formation for enhanced oil recovery (ASP project) has been implemented since 2016. Stability of one of ASP components – polymer strongly depends upon presence of iron, hardness cations and dissolved oxygen in water. As long as polymer is used for injection on two project stages, which are the main and the most extended, at the stage of ASP unit design a complex of polymer protection from negative factors was considered, particularly impact of oxygen, which causes not only oxygen corrosion, but also irreversible destruction of polymer chains. The article describes studies of polymer solution stability, contains analysis of viscosity loss in the course of time in presence of iron and oxygen for polymer solutions. It justifies selection of chemical deoxygenation method for dissolved oxygen control. The article describes program of ASP laboratory studies and analytical instrumentation applied. It provides ASP process scheme and recommendations for technology implementation.

**Keywords:** ASP flooding, EOR, ASP Pilot project, West Salym oil field, dissolved oxygen, free-radical degradation mechanism, chemical oxygen scavenging, nitrogen blanketing, amperometrical method, polymer stability

**Recommended citation:** Bondar M.Y., Shuster M.Y., Karpan V.M., Kostina M.Y., Azamatov M.A. (2018). ASP project. Problematics of dissolved oxygen. Theory and practice. *Georesursy = Georesources*, 20(1), pp. 32-38. DOI: <https://doi.org/10.18599/grs.2018.1.32-38>

## Introduction

Salym Petroleum Development N.V. is executing Pilot project for experimental injection of solutions of anionic surface-active substance (surfactant), soda and polymer into formation for enhanced oil recovery. Active injection of ASP solution (anionic surfactant, soda, polymer) started in July 2016, the next phase – polymer flooding – was implemented in 2017. The project will be completed by the end of Q1, 2018. Stability of one of ASP components, polymer, strongly depends upon presence of iron, hardness cations and dissolved oxygen in water. In order to remove potential negative effect of dissolved oxygen ASP project includes a number of actions focused both on removal of dissolved oxygen from water and restriction of its ingress from atmosphere or chemicals in the process of operation. Detailed description of ASP project and aspects of oxygen management: from determination of its negative effects to methods of its removal and control over its concentration for future

ASP projects are reviewed in the paper (Erke, Kostina, Bondar et al., 2018).

This article presents the latest results of studies of dissolved oxygen effect on efficiency of ASP flooding.

## Effect of dissolved oxygen in ASP project

Polymer applied in ASP project is partially hydrolyzed polyacrylamide – in conditions of chemical flooding it is exposed to be impacted by high temperature, pressure, shear stress, hardness salts dissolved in water, iron and dissolved oxygen, which affects its stability (Erke, Kostina, Bondar et al., 2018).

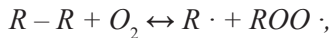
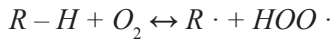
As long as polymer is used for injection on two stages of the project (at first injection of ASP solutions and then injection of polymer solution), which are the main and the most extended, on the stage of ASP unit design we developed a complex of polymer protection from the above-mentioned negative factors, especially from oxygen impact, which causes not only oxygen corrosion but also irreversible destruction of polymer chains (Isabel Vega et al., 2015; Seright, Skjevrak, 2014; Wellington, 1983), also due to free radical mechanism, which results in irreversible loss of solution viscosity meaning loss of its target properties (Erke, Kostina, Bondar et al., 2018).

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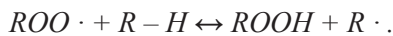
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Partially, such chemical sensitivity of polymer is used when polyacrylamides are applied for treatment of wells, when solutions of strong oxidizers – peroxides (so-called polymer breaker) being sources of radicals (due to unstable -O-O- connection) are used for their destruction.

Mechanism of degradation (loss of target viscosity properties) under impact of oxygen can be described with the following reactions:



where R-R – Section of polymer chain. When started, the reaction occurs by chain mechanism:

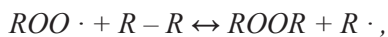
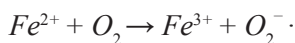


In the presence of active ions or molecules, for example, strong oxidizers or reducers, or ions of transition metals, such as iron, generation of free radicals becomes stronger, which accelerates the process of polymer chain destruction.

The Company performed own studies of stability of polymer solutions, one of results is provided in Figure 1, which is aligned with general principles (Seright, Skjevraak, 2014,).

Presence of iron ions accelerates the process of free radicals release with involvement of oxygen (Isabel Vega et al., 2015; Wellington, 1983), but in deoxygenated medium the impact of iron is insignificant, which is also described in (Isabel Vega et al., 2015; Wellington, 1983) and in our stability tests.

In presence of iron (or cation of other transition metal or oxidizer) and oxygen, mechanism of free radicals release occurs according to the following reactions:



where R-R – Section of polymer chain.

In the above reactions it is important that simultaneous presence of iron and oxygen in water causes the biggest

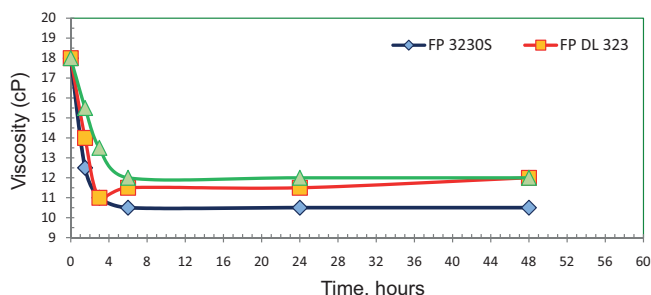


Figure 1. Diagram of viscosity loss in the course of time in presence of iron and oxygen for polymer solutions of Flopaam series (FP 3230S – applied in the project) in solution with polymer concentration of 2500 mg/l, iron – 5 mg/l, oxygen 10 mg/l, at temperature 83 °C

impact on length of polymer chain, and therefore on polymer viscosity. The other side of presence of trivalent metal ions is cohesion of polymer chains between each other, which will abruptly increase solution viscosity above the required value. This issue was raised in (Seright, Skjevraak, 2014), however in ASP project there were no preconditions for iron level increase, even taking into account corrosion of extensive sections of pipelines, to the level when cohesion of polymer chains in solution starts; iron was present in water in amounts directly activating chain radical mechanisms.

As long as pipeline electrochemical corrosion (and therefore presence of ions of iron and other heavy metals in water) cannot be fully excluded, even after water treatment including desalting on reverse osmosis membranes, it was decided to focus all efforts on removal of dissolved oxygen (deoxygenation) with process mode norm of not more than 10 mkg/l and protection of the process from atmospheric oxygen (Erke, Kostina, Bondar et al., 2018) and oxygen coming into the system along with ASP components.

Mechanism of viscosity reduction partially has free radical nature, and that means that reaction can progress not only by oxidizing principle – with involvement of oxidizers, but also with involvement of reducers, which also should be taken into account.

### Oxygen management methods applied in Pilot project

Review of dissolved oxygen management methods and their application in ASP flooding pilot project are described in the paper (Erke, Kostina, Bondar et al., 2018). The project is being executed at several infrastructure facilities; the main facility of the project process scheme is unit for treatment and injection of ASP chemical solutions. The main sources of oxygen ingress into the process in this process scheme are: artesian wells, bulk chemicals granules of which contain voids with atmospheric air, surface active substances (surfactants) and isobutyl alcohol (IBA). Atmospheric oxygen can dissolve in case of contact with water in vessels and tanks (balanced concentration of oxygen in real conditions may reach 9-10 mg/l). Therefore oxygen management is simultaneously performed in several areas (Erke, Kostina, Bondar et al., 2018):

- Creation of nitrogen blanketing in all vessels and tanks in order to exclude contact of fluids with atmospheric oxygen. Nitrogen is generated in nitrogen-air station;
- Chemical scavenging of dissolved oxygen by dosing reducer immediately after water treatment taking into account oxygen excess coming in downstream of dosing point (with chemicals, etc.);
- Control over water-chemical mode of chemical scavenging of oxygen (pH, alkalinity, temperature, etc.);

- Continuous analytical control over oxygen concentration in various process streams for timely identification of oxygen ingress sources and its removal, control is performed both with use of instrumentation and by various laboratory methods.

**Selection of dissolved oxygen management methods**

Two methods of oxygen management were initially reviewed during development of ASP project:

- Membrane deoxygenation on membrane contactors (Klaassen, Feron, Jansen, 2005) operating in nitrogen-vacuum scheme, because nitrogen station is applied in the project regardless of selected oxygen management method and is able generate nitrogen of required quality – not lower than 99.8%; principle of operation and appearance are shown in Figure 2.

- Chemical deoxygenation (scavenging of dissolved oxygen with reducer).

Traditional deaerators widely applied in energy industry were not reviewed due to equipment size and cost.

Economic calculation by total costs for project life cycle (about 1-1.5 years) showed efficiency of the second option, in addition contactors would have complicated already complex process scheme of water treatment (Erke, Kostina, Bondar et al., 2018). But for future projects this method can be applied, if, for example, dissolved oxygen and dissolved carbon dioxide should be removed simultaneously.

For implementation of the second method screening of different oxygen reducers-scavengers based on

organic and inorganic sulfite-containing substances are performed, including laboratory testing of selected chemical (Table 1).

Despite a number of obvious disadvantages, such as generation of sulfates in the process of reaction with oxygen, limited operating range of pH and temperature, sulfite-containing (sulfites, bisulfites, metabisulfites) scavengers were selected for further processing mainly because they are cheap and easily accessible, and also due to easy involvement into operating process as well as due to operation mode of dosing equipment. In addition, sulfites can generate protective film (as stated by the manufacturer) on equipment walls, which contributes into reduction of oxygen corrosion rate (Erke, Kostina, Bondar et al., 2018). Herein sulfites mean sodium bisulfite, sodium sulfite and sodium metabisulfite, including with catalyst.

During laboratory testing a process of oxygen scavenging with AMINAT™ KO-2 (The chemical safety passport AMINAT™ KO-2 according to TS 2149-098-17965 829-03 of the company CJSC “ECOS-1”) chemical was simulated and impact of various factors on kinetics of reaction (pH, impact time, temperature, etc.) was estimated. Description and results of experiments for assessment of AMINAT™ KO-2 efficiency during deoxygenation of distilled water are provided in (Erke, Kostina, Bondar et al., 2018).

Basing on results, in order to provide conditions for chemical deoxygenation process in distilled water it is required to additionally perform dosing of alkaline chemicals. Experience in deoxygenation of distilled water with alkalization to pH = 6.7 showed high

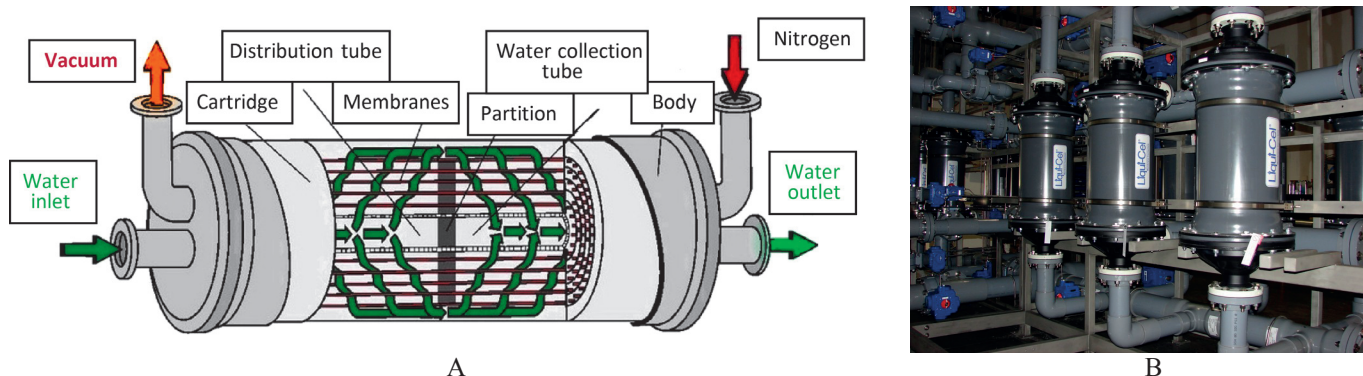


Figure 2. Principle of operation (A) and appearance (B) of membrane contactors for deoxygenation

| Oxygen scavenger       | Passivating properties | Increase of salt concentration | Toxicity            | Stoichiometric consumption for 1 mg of O <sub>2</sub> | Commodity form   |
|------------------------|------------------------|--------------------------------|---------------------|---|------------------|
| Sodium sulfites        | no                     | yes                            | No data             | 7.9   | Powder, solution |
| Hydrazine              | yes                    | no                             | Toxic, carcinogenic | 1.0   | Liquid           |
| Carbohydrazine         | yes                    | no                             | No data             | 1.4   | Liquid           |
| Methyl-ethyl-ketoxime  | yes                    | no                             | No data             | 5.4   | Liquid           |
| Hydroquinone           | yes                    | no                             | No data             | 6.9   | Liquid           |
| Di-ethyl-hydroxylamine | yes                    | no                             | Safe                | 1.2   | Liquid           |

Table 1. Chemicals review– oxygen scavengers ((Erke, Kostina, Bondar et al., 2018) with supplements)

efficiency of chemical even at low temperature. Almost full oxygen scavenging occurred even during the first minutes of the experiment, and after ten minutes oxygen concentration decreased to required values of 10-15 mkg/ dm<sup>3</sup> (Erke, Kostina, Bondar et al., 2018).

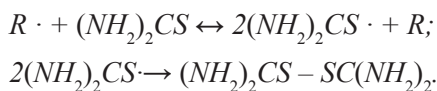
Basing on performed studies for assessment of reverse osmosis permeate chemical deoxygenation with use of AMINAT™ KO-2 chemical efficiency it is possible to make the following conclusions (Erke, Kostina, Bondar et al., 2018):

1) Efficient chemical scavenging of oxygen in the process of AMINAT™ KO-2 dosing occurs in case of long alkalization, thus providing pH value of treated water above 6.2-6.3;

2) Duration of permeate residence in accumulating tank after dosing of AMINAT™ KO-2 chemical does not affect the efficiency of deoxygenation during further water heating;

3) Recommend to perform permeate alkalization upstream and downstream of accumulating tank with correction of dosing by pH value, use more alkalized forms of chemicals, for example, sodium sulfites or sodium bisulfites.

Also, it should be noted that there is an alternative to the above-mentioned methods to remove negative impact of oxygen – use of polymers with protective additives such as urea and thiurea. Molecules of urea interact with radicals and connect into stable molecules:



Such or similar additives are often used by manufacturers of polymers when it is required to compensate negative interaction, including oxygen, hydrogen sulfide, etc. For the Pilot project it was decided to take the selected way (chemical scavenging of oxygen) due to high price for special protected polymers.

### Dissolved oxygen management during operation. Water-chemical mode

In process of operation pH change in clean water tank was identified where reverse osmosis permeate is supplied (having reduced pH value due to carbonic acid). The pH factor was decreased below 5.0 units due to hydrolysis of metabisulfite, which reduced chemical efficiency. By results of studies it was decided to increase pH value only in clean water tank by dosing soda ash solution (Erke, Kostina, Bondar et al., 2018).

The dynamics of oxygen concentration in various streams was also analyzed. In case of shutdown of nitrogen blanketing of volumetric equipment or contact with atmosphere (for example, in process of pre-commissioning operations or shutdowns) abrupt increase of oxygen concentration was observed, which was the result of water system tendency to come to balanced concentration of dissolved oxygen in specific conditions (Rubin Battino et al., 1983; Kai Fischer, Michael Wilken, 2001) (Figure 3).

In process of pre-commissioning and startup of ASP project when injection of surfactant solution, its co-solvent, isobutyl alcohol (IBA), soda, salt and polymer into the reservoir was started, in final mixed

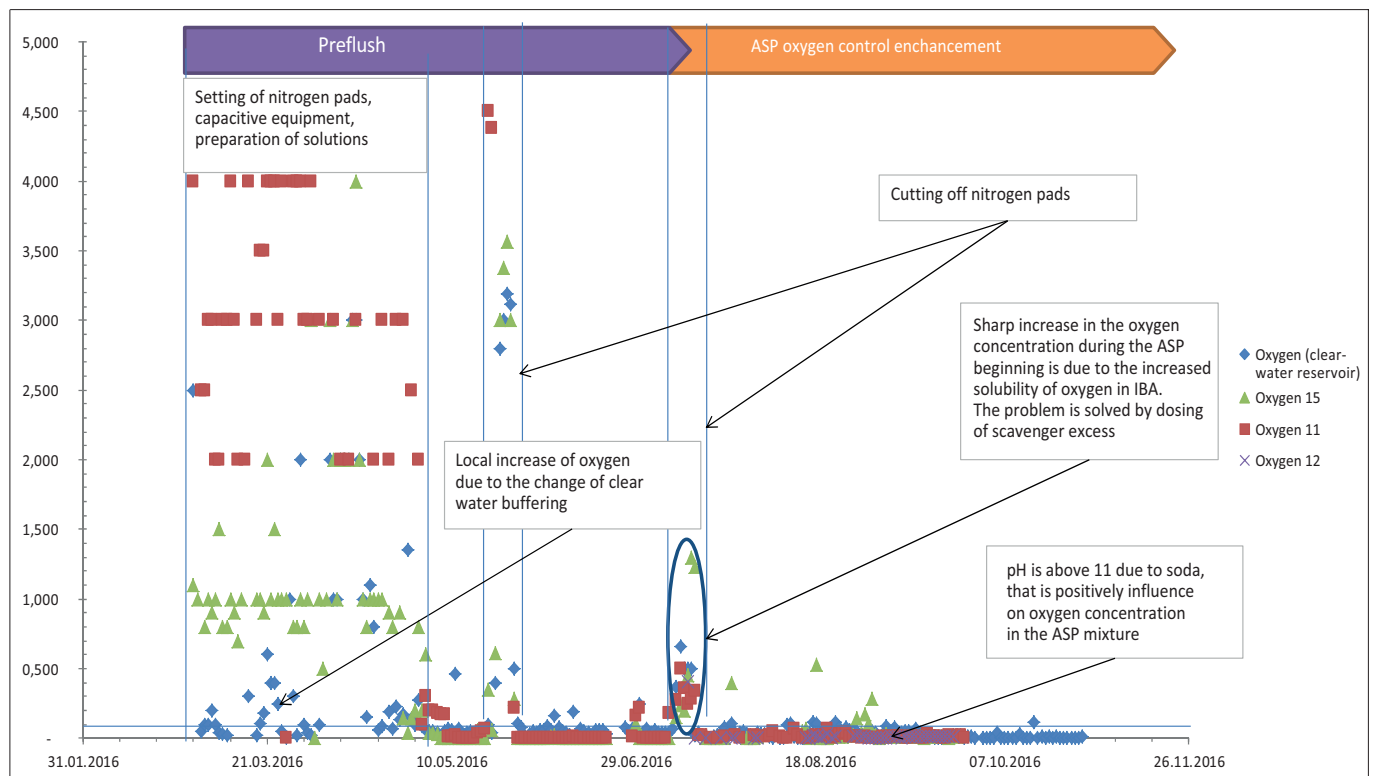


Figure 3. Dynamics of oxygen concentration in various flows during the implementation of the ASP Pilot project (Erke, Kostina, Bondar et al., 2018)

solution abrupt increase of oxygen concentration was observed. It was defined that jumps in oxygen concentration change recorded with inline analyzer were aligned with readings from sensors of IBA instant flow rate. It is associated with high oxygen solubility in alcohols, including isobutyl alcohol. Thus, IBA filling in truck tanks occurs not in fully sealed and isolated conditions, oxygen dissolved in IBA was coming into ASP solution and the sensor was recording that (Erke, Kostina, Bondar et al., 2018). It is noted that balanced concentration of oxygen in alcohol may reach dozens of mg/l (Kai Fischer, Michael Wilken, 2001; W. Rodgers Baird, Robert T. Foley, 1972). Attempts to perform measurement of oxygen content resulted in wide range, from several mg/l to 50-80 mg/l.

In order to guarantee removal of oxygen coming from IBA or other source it was decided to increase excess of sulfites with further reduction to 10-15 mg/l, as a result of which oxygen concentration reduced within several days. Then it was decided to reduce excess of sulfites and increase it only in cases of unexpected deviations from process mode norms (shutdown or insufficiency of nitrogen blanketing, loss of containment of tank equipment, etc.).

Recommendations on use of sulfites were also developed, because their use can have a number of side negative effects in West Salym as well as on other fields: sulfates as products of oxygen scavenging are processed by sulfate-reducing bacteria (SRB) producing hydrogen sulfate which leads to active corrosion of equipment and pipelines. Also barium in formation waters will produce barite (form of barium sulfate), sediments of which are extremely difficult to remove even with strong acids.

Sulfites as strong reducers, can also participate in radical reactions of polymer degradation, which means necessity of control over concentration of residual sulfites in water, but most sulfites after reaction with oxygen produce sulfates, anions inert in relation to free radical mechanisms, and residual sulfites in produced

concentrations do not significantly affect viscosity. This was taken into account during development of program for control of water-chemical mode.

### Analytical control program

Program of laboratory studies of ASP projects contains over 38 analytical indicators and over 45 applied methods, both standard (regulatory documentation, RD, GOSTs, ASTM) and specially developed for the project. Frequently several methods were simultaneously applied for key indicators, which is associated with the fact that the same indicators in different media, streams and conditions require the use different measurement methods.

Measurement of dissolved oxygen concentration in laboratories is performed by Winckler’s method or method of iodometric titration (Guidance document 52.24.419-2005. Mass concentration of dissolved oxygen in water. Method of performing measurements by iodometric method). Despite the fact that Winckler’s method is considered to be standard chemical method for solutions analysis with its multiple modifications simplifying studies, it is not possible to use it for operational express-analysis in field conditions (Erke, Kostina, Bondar et al., 2018). Therefore for operational measurement of dissolved oxygen concentration in water during the project analyzers of dissolved oxygen or oxymeters were used (Figure 4 A, C), as well as special test-kits for express determination of dissolved oxygen (visual colorimetric method, Figure 4 B).

After analysis of analytical instruments and consultations with manufacturers it was decided to use only devices based on more neutral method – amperometric method.

The principle of amperometric analyzer for dissolved oxygen is based on electrochemical method for measurement of oxygen concentration.

Number of portable devices and two types of analytical instruments are used in the project for different ranges of measurement (from close to

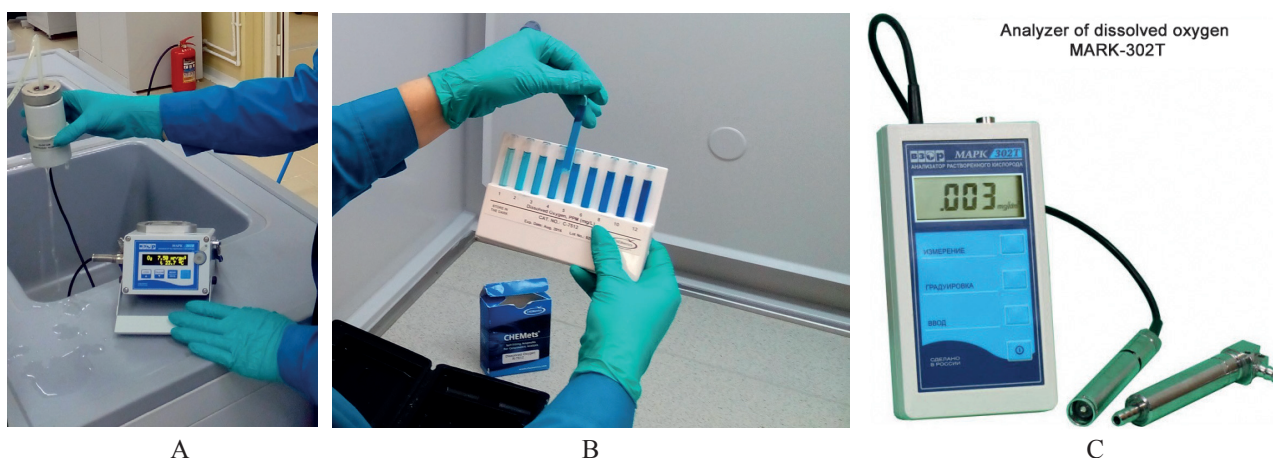


Figure 4. Laboratory methods for measurement of oxygen concentration. A – Portable flow-type analyzer MARK-3010, B – test-kits Chemetrics, C – portable flow-type analyzer MARK-302T.

balanced concentration values to insignificant values of several mkg/l).

Selected express-methods for measurement of oxygen by visual colorimetric methods are also used for verification: Indigo blue carminic for values of at least 1 mg/l and Rodhazine D for values in mkg/l (ASTM D 888-87, Dissolved Oxygen in Water; ASTM D5543-09, Standard Test Methods for Low-Level Dissolved Oxygen in Water) (Figure 4).

For complete control over oxygen concentration at various stages of ASP process scheme a number of sampling points were designed and arranged, as well as flow-type analytical measuring instruments.

Green boxes in simplified diagram (Figure 5) of the process show sampling points, and yellow boxes – spots of oxygen control with flow-type analytical instruments, red arrow indicates spot of oxygen scavenger supply.

Such number of control points of oxygen concentration allows to promptly identify reasons of concentration increase (oxygen sources), and use of three approaches to determination (portable instrument – test-kit – instrumentation) minimizes probability of wrong determination of oxygen concentration. In addition, special focus is made on determination of excess of sulfite-ions in various media, as indication of processes occurring in them. Absence of sulfites can become a signal for quick increase of oxygen level (Erke, Kostina, Bondar et al., 2018). It is particularly important to have an excess of chemical before mixing with isobutyl alcohol or other liquid, in which oxygen solubility can be significantly higher than in water.

Also, implementation of scheme with nitrogen blanketing of tank equipment allows to make a conclusion that process media should be protected after introduction of chemical scavenger or introduction of polymer into the process. That means that there is no need to make nitrogen blanketing in source water tanks, intermediate tanks of water treatment unit, etc.; which will allow to reduce operational expenditures.

### Conclusions and recommendations

During implementation of ASP technology where one of the key roles is played by control over oxygen dissolved in water and solutions, it is important to take into account a number of factors and implement the following recommendations (Erke, Kostina, Bondar et al., 2018):

- At design stage it is required to exclude contact with atmospheric air by means of maintenance of nitrogen blanketing in tank equipment, especially for deoxygenated media, because the system will quickly return to balanced concentration of oxygen in water or water solution, which is mg/l units. There is no need to make nitrogen blanketing in tanks with source water, intermediate tanks of water treatment unit, etc.; this will allow to reduce OPEX.

- It is required to maintain excess of oxygen scavenger at acceptable level. Lack of excess is a signal of problem and increase of oxygen level in near future, big excess also can be the cause of polymer degradation;

- It is required to comply with water-chemical mode for application of chemical scavenger which means maintenance of optimal temperature, pH and residence time;

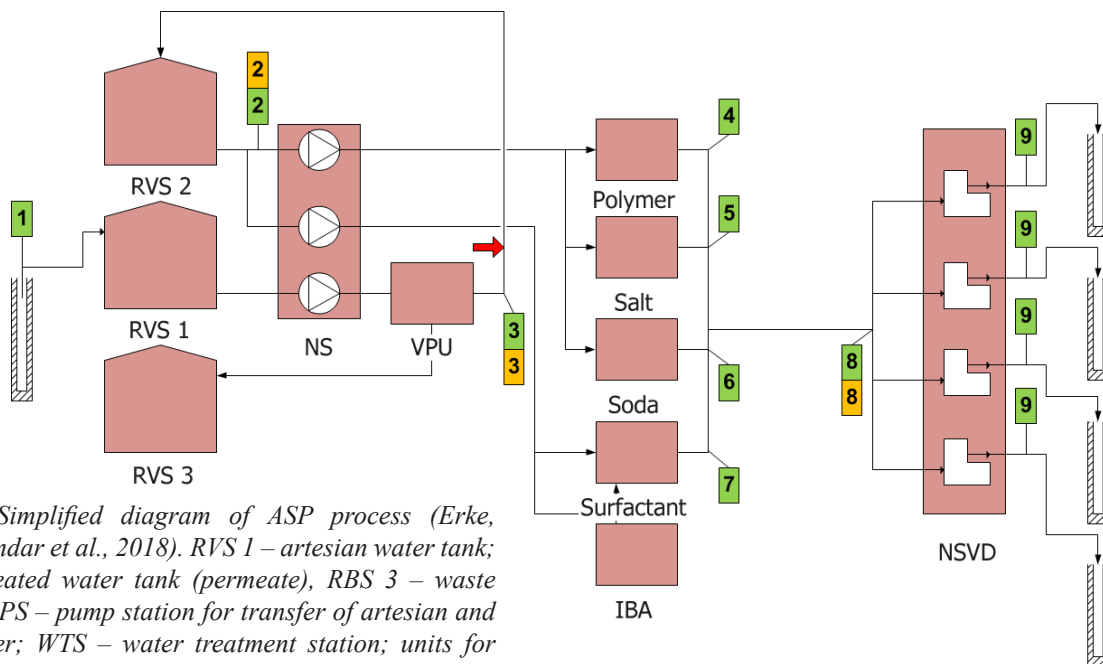


Figure 5. Simplified diagram of ASP process (Erke, Kostina, Bondar et al., 2018). RVS 1 – artesian water tank; RVS 2 – treated water tank (permeate), RVS 3 – waste water tank; PS – pump station for transfer of artesian and treated water; WTS – water treatment station; units for preparation of mother solutions of chemicals (polymer, salt, soda, surfactant, isobutyl alcohol); HPPS – high pressure pump station for injection of ASP solutions into the reservoir. Flows of media in which analysis of dissolved oxygen concentration is performed are marked with figures: 1 – Artesian water; 2 – Treated water (permeate) downstream of RVS tank; 3 – Treated water (permeate) upstream of RVS tank; 4 – Polymer mother solution; 5 – Salt mother solution; 6 – Soda calcinated mother solution; 7 – Surfactant and isobutyl alcohol mother solution; 8 – ASP mixture at inlet point of injection pump station after mixing; 9 – Sampling points on injection line of high pressure pumps.

- It is required to take into account oxygen ingress with ASP components, especially those in which oxygen solubility is abnormally high;
- It is important to understand what methods of oxygen concentration measurement are applicable and optimal for different media, especially multi-component media, for this purpose it is required to consider side physical and chemical processes which may result in wrong measurement of oxygen concentration. Also, the use of different alternative methods will be useful for verification;
- Special focus should be made on development of sampling points system and their arrangement in order to exclude contact with atmosphere in process of sampling and compliance with sampling rules;
- When selecting a chemical method it is important to select the chemical, which application will not lead to additional actions for implementation of optimal water-chemical modes.

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Manuscript received 30 November 2017;  
Accepted 2 March 2018; Published 30 March 2018