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# Atomic force microscopy and IR spectrometry application in detecting the type and nature of contaminants on reverse osmosis membrane elements

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**Abstract.** The main, the most acute problem of barometric technology today is reagent provision. For its stable and trouble-free operation, a complex of expensive and unique chemical reagents (biocides, antiscalses, washing compositions) is required. Each manufacturer develops his own chemical composition for a certain membrane type. This leads to a significant cost rise of reagents, as well as creates a dependence of the consumer technology from a particular supplier. However, these reagents do not always meet the quality requirements - despite the guarantees provided by manufacturers, the equipment fails before the due service life as a result of deposits in the pores and on the membrane surfaces. To develop a more effective washing composition, it is required to determine the type and consist of these deposits. The report illustrates the methods for determining the consist and type of deposits on reverse osmosis membranes. The solution of the problem of reagent provision for the example of the wastewater treatment plant at the Ashalchinsky field of the extra-viscous oil of PJSC "Tatneft" is presented.

## Introduction

The experience of the vapor gravitational technology application obtained by PJSC "Tatneft" at the Ashalchinskoye field of super viscous oil showed that the use of steam for its extraction poses a problem both in preparation and disposal of a water large amount [1].

By the end of 2014, the volume of wastewater chemical water treatment (CWT) of the Ashalchinsky field was about 8,000 m<sup>3</sup> per day.

The problem solving of the region's environmental tension was achieved through the a sewage treatment plant (STP) commissioning based on baromembrane technologies. However, during the STP operation, a number of problems arose: firstly, the washing composition declared by the manufacturer did not cope with its main task - washing the membranes away from salt deposits - as a result premature failure of the reverse osmosis unit STP membrane elements. Occurs secondly, the composition claimed by the manufacturer contains components other than the components of the CWT boiler plant compositions at the Ashalchinsky and North Ashalchinsky fields, which causes the customer to purchase components from various sources.

In the third, the washing-out composition of the CWT has unique reagents in its composition, because of which the consumer is forced to apply for reagents strictly to the manufacturer.

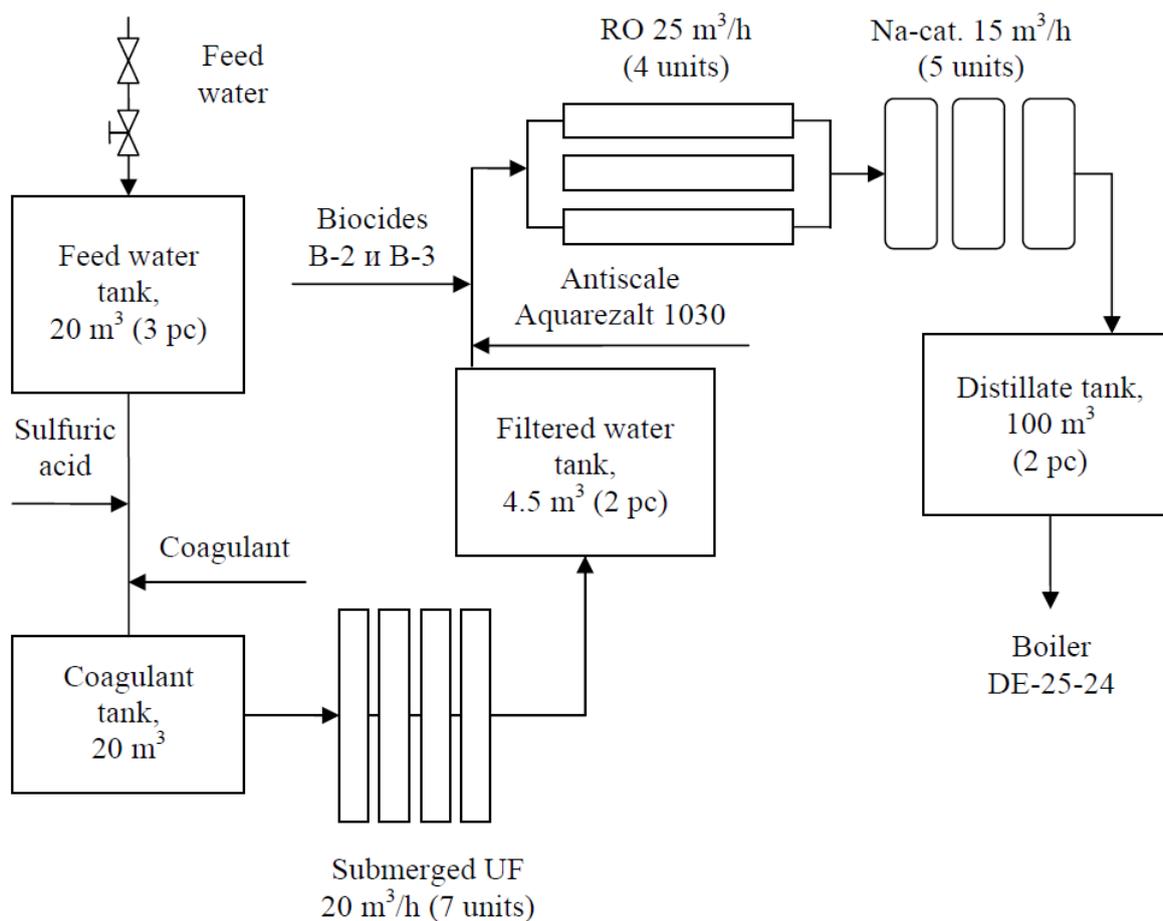
The development of a unified composition of a more efficient washing composition, consisting of available chemical reagents, would solve the above problems [2].

Wastewater treatment unit boiler "Ashalchi" Ashalchinsky extra-viscous oil fields of PJSC



"Tatneft" STP boiler "Ashalchi" is designed to process the wastewater treatment the CWT boiler at Ashalchinsky field and re-use them for the CWT boiler North Ashalchinsky field.

The STP scheme includes a submerged ultrafiltration unit, a reverse osmosis unit, and a sodium cationing unit (figure 1).



**Figure 1.** Scheme CWT STP.

The reverse osmosis consists of four reverse osmosis (RO) units "Sharya MP-25-RO", three of which are workers, one is a backup one. The capacity of each installation is permeate 25 m<sup>3</sup>/h. The estimated flow rate for the initial water is 38.5 m<sup>3</sup>/h. Work on manufacturing and installation of "Sharya MP-25-RO" was carried out by the enterprise "BIOTECH-PROGRESS" in 2015. The installation itself was commissioned in May 2016. In the summer of 2017, the roll elements were replaced because of their premature failure. One of these elements samples was used for research.

### Selection and preparation of reverse osmosis membrane material samples

The initial material for the investigation of the deposit composition was the spent cellulose acetate membranes of the reverse osmotic roll filtration element NanoRO K 8040-C of the Sharya MP-25-RO unit for processing of the Ashalchi boiler plant effluents.

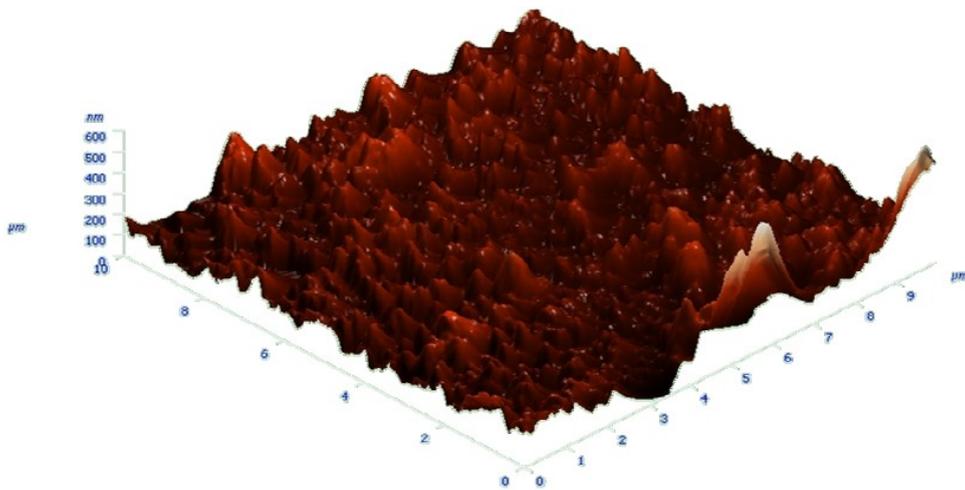
RO NanoRO K 8040-C consists of 21 cellulose acetate membranes and a perforated tube. Samples were selected from 5 points along the length and 4 points along the widths 1, 8, 15 and 21 of the membrane layer. A total of 80 samples. The dimensions of the samples are 30 × 40 mm.

Further sample preparation was carried out:

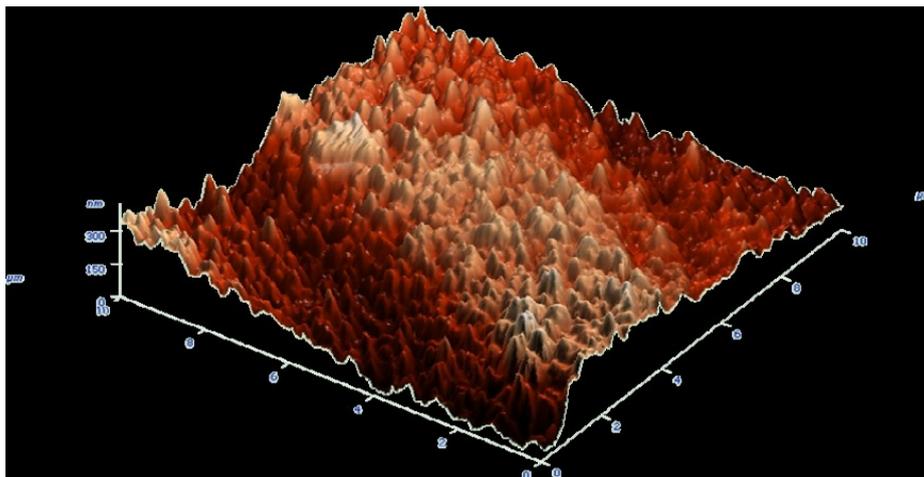
- 2 samples (at the membrane unit inlet and outlet, 21 layers) were soaked in a distillate - for subsequent examination with a scanning electron microscope;
  - 5 samples (along the length of the filter, 15 layer) were soaked in 4% citric acid solution and 1% acetic acid solution for 15 hours to obtain acidic extracts (for UF spectrometry);
  - 5 samples (along the length of the filter, 15 layer) were soaked in a 1% solution of sodium chloride for 15 hours to obtain salt extracts (for UF spectrometry).
- The remaining samples were examined in dry form.

### Atomic force microscopy of samples

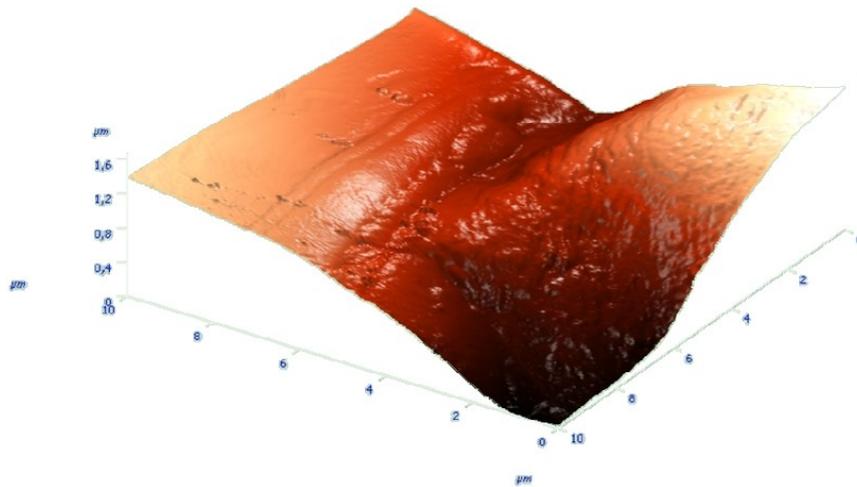
Investigation of the morphology of the spent reverse osmosis acetate cellulose membrane surface was performed on a scanning probe microscope Solver HV using atomic force microscopy. Surface images are presented in figure 2-4.



**Figure 2.** 3D surface of first sample (at the entrance to the filter). The zone size is 10  $\mu\text{m}$ .



**Figure 3.** 3D surface of third sample. The zone size is 10  $\mu\text{m}$ .



**Figure 4.** 3D surface of fifth sample (at the out to the filter). The zone size is 10  $\mu\text{m}$ .

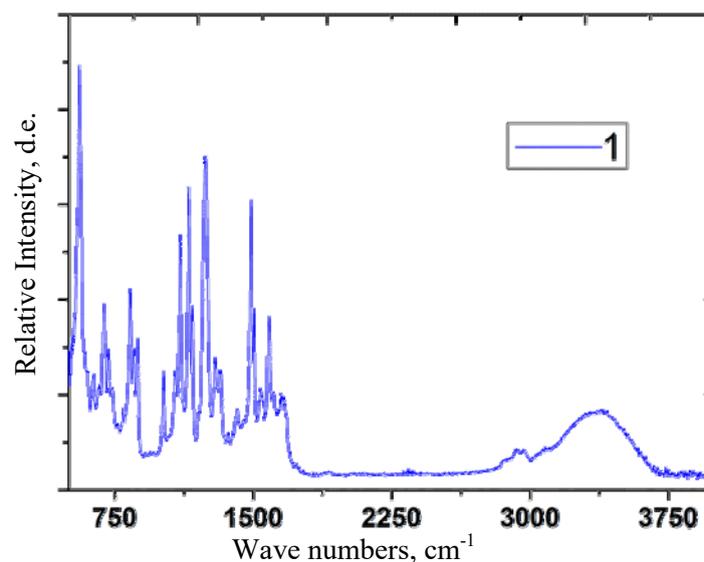
A total of 5 dry samples were scanned (along the length of the filter, 21 layers).

Analyzing the pictures, you can see that the closer the sample is to the exit from the device, the smoother the profile of its surface becomes.

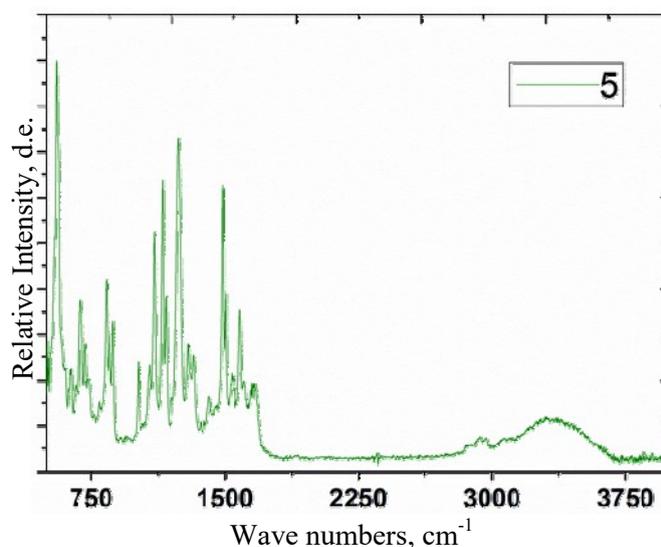
#### **Investigation of reverse-osmotic acetate cellulose membrane surfaces by IR spectrometry**

Two dry samples were used for the studies: at the inlet and outlet of the filter, 8 membrane layer.

Investigation of infrared spectra was carried out using the IR spectrometer ALPHA (ALPHA-E module) by Bruker Optics (Germany) using the ATR (attenuated total reflection) method. Infrared spectra (figure 5, 6) are taken in the middle infrared range using the OPUS software. The natural surface of samples on a ZnSe crystal is studied. The area of the sample contact with the crystal is 19.6  $\text{mm}^2$ .



**Figure 5.** IR spectrum of the membrane sample at the inlet to the filter.



**Figure 6.** IR spectrum of the membrane sample at the outlet of the filter.

Measurement mode:

- Resolution - 2  $\text{cm}^{-1}$ .
- Sample scan time - 50 scans.
- Background scan time - 50 scans.
- Measuring range - 500 ... 4000  $\text{cm}^{-1}$ .

The intensity peaks presented on figures 5, 6 correspond to a certain set of chemical components, of which the membranes are deposited. Interpretation of atomic force microscopy results and infrared spectrometry of membrane samples allowed to determine the consist and character of the deposits, which, in turn, made it possible to select the optimal consist of the more efficient washing composition for the previously described effluent treatment unit. The composition components were unified, generally available chemical reagents.

### Conclusion

On the basis of information of the feed water parameters, the washing composition consist, and the operating equipment a regime map for the management of water-chemical regime of the baromembrane unit for medium-pressure boiler was developed.

### Acknowledgments

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### References

- [1] Sakhabutdinov R Z, Gubaidulin F R, Loyko A V, Itskov S V and Buslaev E S 2014 Development of technology that envisages processing of waste waters after chemical-water treatment of boilers to produce steam at Ashalchinsky field *Equipment and technologies for oil and gas industry* 5 35-39
- [2] Chichirova N D *et al* 2017 *J. Phys.: Conf. Series* 891 012276