Methods of pollution control and waste management

Experiment 14

Membrane methods in wastewater treatment

Manual

Department of Chemical Technology

The aim of this experiment is to get knowledge on membrane methods and to test commercial microfiltration membrane for separation of anionic surfactants from emulsified oily wastewater. Oily wastewaters are generated in many industrial processes, such as petroleum refining, petrochemical, food, leather and metal finishing.

The separation process is carried out using an asymmetric membrane Micro CARBOSEP 40 M14 with carbon support with pores size d=0.14 μm in diameter and selective active layer made of zirconia (ZrO_2) in tubular membrane module with length 40 cm and size D=0.687 cm in diameter with active surface 80 cm^2 produced by Tech Separation Millipore. General scheme of the apparatus using in the experiment is presented below. Anionic surfactants (AS) are separated from the mixture of the products of sulfonation of alkylbenzenes and their further neutralization with potassium hydroxide solution.

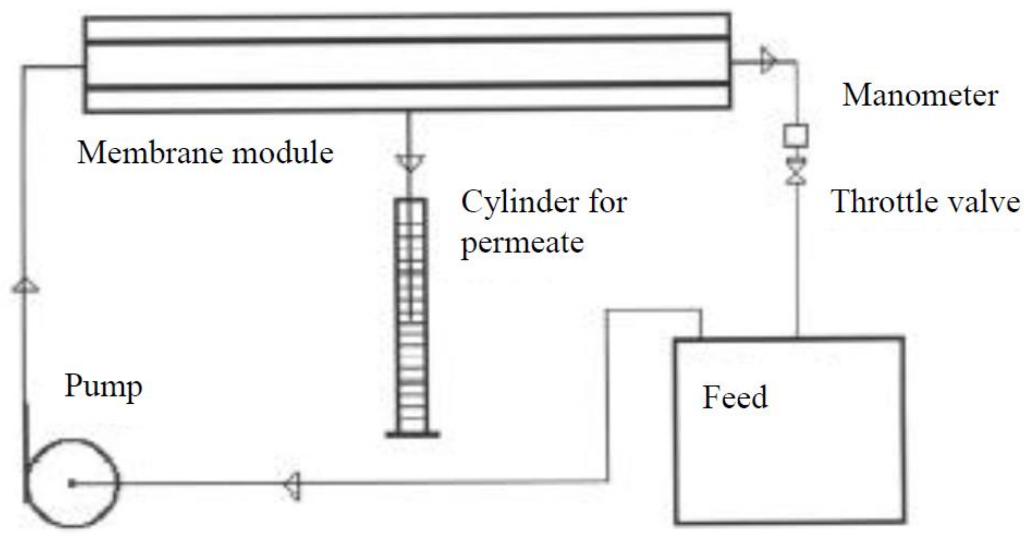

Figure 1. Apparatus scheme.

Equipment/Materials: spectrophotometer UV-vis (200-800 nm), buffers solutions, methylene blue solutions (acidic and basic), laboratory glassware: separatory funnels, pipettes, volumetric flasks, conical flasks and beakers.

Procedure:

1. Adjust pumping speed to achieve an appropriate transmembrane pressure

3. Measure the permeate and the retentate flux (at five transmembrane pressure given by instructor).

4. Perform analysis of anionic surfactants concentration in the selected permeates

4. Measure absorbance of chloroform solutions of complexes of anionic surfactants with methylene blue at 680 nm

Analysis of anionic surfactants concentration is carried out according to Minister of Environmental Protection standard from 6th of November 2002.

Analytical procedure for permeate analysis

1. Pipette 20 cm^3 of permeate and place in volumetric flask with capacity 200 ml, fill with distilled water (1:10 dilution)

2. Measure pH of solution using universal indicator paper (pH should be close to 7). Add 0.1 M KOH solution if necessary.

3. Measure out 100 ml of solution using volumetric pipette and place in separator funnel, add 10 ml of buffer, 5 ml of neutral solution of methylene blue and 15 ml of chloroform (basic solution).

4. Hold the funnel firmly but gently in both hands so that it can be turned from the vertical to horizontal direction, shake mixture not vigorously. Keep the stopcock tightly seated with the fingers of the other hand in such a way that the fingers can open and close the stopcock quickly to release the pressure that may be built up from solvent vapor or evolved gases. Wait for phase separation and after this transfer chloroform phase to the other separator funnel, add 110 ml of distilled water and 5 ml of acidic solution of methylene blue (acidic solution). Shake mixture during 1 minute, transfer chloroform phase to volumetric flask with capacity 50 ml.

5. Extract twice of water phases (basic and acidic) with 10 ml of chloroform. All chloroform phases transfer into the same volumetric flask.

6. Filtrate the chloroform extracts using fluted filter paper and then transfer the chloroform solution into a volumetric flask with capacity 50 ml and fill with chloroform.

7. Measure the absorbance of the solution from point 6 at 680 nm with chloroform as reference. Determine concentration of anionic surfactants per MBAS using a calibration curve (Figure 2).

The report of the experiment should include:

1. Purpose of the experiment.
2. Description of the experiment
3. Calculations of velocity of feed flow into linear velocity V_L [m^3 s^{-1}] taking into account active surface of membrane 80 cm^2.
4. Calculations of permeate flux J_V given in [m^3 s^{-1} m^{-2}] taking into account active surface of membrane 80 cm^2.

5. Figure which illustrates the relationship between linear velocity of feed and transmembrane pressure: V_L = f (TMP)
6. Figure which illustrates the relationship between flux of permeate and transmembrane pressure: J_V = f (TMP).
7. Concentrations of anionic surfactants per sodium dodecylbenzene sulfonate given in mg/cm^3 (determined using calibration curve and spectrophotometric measurements results).
8. Optimal conditions to achieve high process yield and effectiveness (discussed on the basis of experimental results).
9. Conclusions. In particular:
   advantages and disadvantages of membrane methods, economic side of the membrane process, comment on whether the aims of the experiment have been achieved.

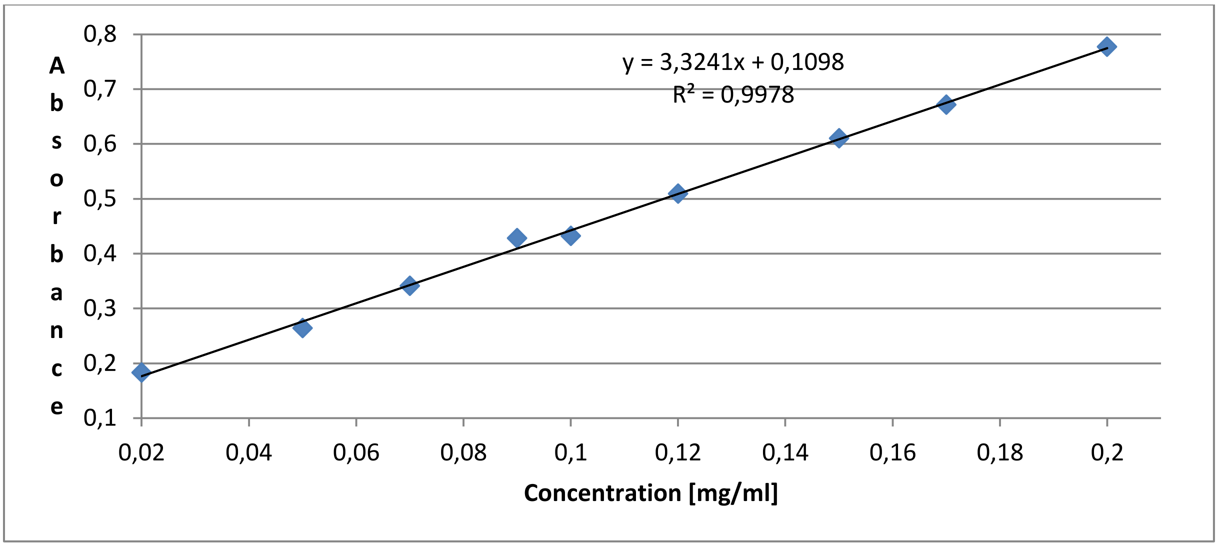

Figure 2. Calibration curve for absorbance of complex anionic surfactants with methylene blue (recorded at 680 nm in chloroform).