International Standard

ISO 20304-4

Fine bubble technology — Water treatment applications —

Part 4: Test method for evaluating the antifouling performance of fine bubble water in crossflow membrane filtration systems

Technologie des fines bulles — Traitement de l'eau —

Partie 4: Méthode d'essai pour l'évaluation des performances anti-encrassement de l'eau à fines bulles dans les systèmes de filtration tangentielle sur membrane

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Foreword

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This document was prepared by Technical Committee ISO/TC *281*, *Fine bubble technology*.

A list of all parts in the ISO 20304 series can be found on the ISO website.

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Introduction

Membrane separation processes exhibit high efficiency in removing various impurities from water. Hence, they have been widely employed for pure-water production, reclaimed-water treatment, and industrial wastewater treatment. However, the application of membrane separation technologies is limited by membrane fouling, which directly affects the economic feasibility and sustainability of these technologies. Conventional membrane antifouling techniques include introducing pre-filters, enhancing membrane hydrophilicity, applying surface coatings, and frequent physical and chemical cleaning. However, these approaches often lead to increased operational costs, environmental concerns, and limited effectiveness. In recent years, fine bubble (FB) and ultrafine bubble (UFB) technologies have been demonstrated to considerably reduce membrane fouling. However, there are currently no effective methods to evaluate the efficiency of these technologies for reducing membrane fouling.

The silt density index (SDI), introduced by the American Society for Testing and Materials (ASTM), is a widely used standard for measuring the fouling potential caused by colloidal and suspended particles. However, the SDI has several limitations including a lack of temperature correction, insensitivity to membrane resistance variations, and poor correlation with colloidal or suspended particle concentrations. These shortcomings influence the accuracy and reproducibility, raising concerns about its predictive value. To address these issues, ASTM has proposed a fouling index model based on microfiltration membranes (MFI-0,45), which improves the accuracy of membrane fouling assessments under various operational conditions by considering the filtration mechanism of the cake layer as well as temperature, viscosity, and membrane area.

However, this method employs dead-end filtration, which is unsuitable for water containing FBs. FBs adhere and accumulate on the membrane surface, decreasing the permeate flux, increasing the filtration resistance, and reducing the effective contact area between test water and membranes, causing considerable fluctuations in the assessment results and failure in accurately reflecting the true extent of membrane fouling. Therefore, neither the SDI nor the fouling index model based on dead-end filtration can accurately evaluate the effects of FBs and UFBs on reducing membrane fouling.

In practical reverse osmosis (RO) operations, membranes are typically operated under crossflow conditions, which better mimic real-world operating scenarios and are more suitable than dead-end filtration for evaluating the influence of FBs on reducing membrane fouling. Many researchers have adopted crossflow filtration setups to improve the measurement of fouling indices, such as the membrane fouling index (MFI), thereby demonstrating the feasibility and maturity of the crossflow approach. Building on these advancements, this document improves on traditional approaches by moving away from using fixed reference conditions, such as 0,45 µm membranes and 200 kPa pressure. Instead, it compares the fouling indices under conditions with and without FBs to specifically evaluate the role of bubbles in fouling mitigation.

Using the crossflow filtration mode, the permeate flow through the membrane is quantitatively measured over time, and a crossflow membrane fouling index (CMFI) is established. This testing condition is in good agreement with the actual operation mode of membrane filtration, resulting in a more realistic and accurate reflection of membrane fouling and enhancing the reliability and performance of membrane fouling assessment. Therefore, CMFI is not only suitable for evaluating the effectiveness of FB technology in reducing membrane fouling but also provides valuable guidance for the design and optimization of industrial processes that integrate FB technology with membrane treatment applications.

Fine bubble technology — Water treatment applications —

Part 4: Test method for evaluating the antifouling performance of fine bubble water in crossflow membrane filtration systems

1 Scope

This document specifies a method for assessing the impact of ultrafine bubbles (UFBs) on membrane antifouling using the crossflow filtration system as the evaluation setup. The evaluation compares the membrane fouling behaviour of the original wastewater (control) with that of the same wastewater after UFB generation under specified operating conditions. This document is applicable to dispersions containing UFBs, including dispersions in which microbubbles (MBs) are present in combination with UFBs.

The method employs the crossflow membrane fouling index (CMFI), measured under constant transmembrane pressure during crossflow filtration as the evaluation criterion.

This method is applicable to various membrane treatment systems including ultrafiltration, nanofiltration, and reverse osmosis and can be used for different types of wastewater containing particulates, colloids, and soluble organic matter.

This method is primarily intended for dispersions containing UFBs (see Annexes A to C and E); however, it is equally applicable, by analogy, to dispersions containing MBs (see Annex D).

This document can guide the design and optimization of industrial processes that integrate FB technologies for enhanced membrane antifouling.

NOTE 1 This method is intended for waters that contain a measurable amount of either particulate matter or colloidal matter, or both. It is not applicable to essentially particle-free waters, such as permeate from reverse osmosis or ultrafiltration systems.

NOTE 2 Users can record supplementary information, e.g., turbidity, total suspended solids, and particle-size distribution; however, such data are not required in this document.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Terms

3.1.1
crossflow
filtration method wherein the feed stream flows tangentially to the membrane surface rather than perpendicular to it

Note 1 to entry: The flow orientation ensures that the fluid on the upstream side of the membrane moves parallel to the membrane surface rather than directly through it.

3.1.2
crossflow membrane fouling index
CMFI
index calculated from the development of filtration velocity through a membrane filter under a crossflow mode

3.1.3
concentrate
stream containing rejected particles and solutes discharged from a membrane module during crossflow filtration

3.1.4
dead-end filtration
filtration method wherein the only exit for the upstream fluid is through the membrane

Note 1 to entry: In this configuration, the feed stream flows perpendicularly to the membrane surface and all the fluid must pass through the membrane to exit the system

3.1.5
flux
throughput of a membrane, typically quantified as the permeate volume produced per unit time and per unit membrane surface area

Note 1 to entry: Flux is commonly expressed in litres per square meter per hour ($L/m^2/h$), and measurements are often standardized at a specific temperature (typically 20 °C).

3.1.6
membrane fouling
undesirable accumulation of substances on a membrane surface or within its pores during filtration processes

Note 1 to entry: This buildup hinders the membrane's performance by reducing its permeability or flux, increasing the system operating pressure and decreasing the membrane separation efficiency.

3.1.7
pore size
size of openings of a porous membrane expressed using either a nominal (average) or an absolute (maximum) value

Note 1 to entry: For microfiltration membranes, the pore size is usually expressed in µm, whereas for ultrafiltration, nanofiltration and reverse-osmosis membranes, it is typically expressed in nm.

3.1.8
transmembrane pressure difference
pressure difference across a membrane, which is the driving force for fluids passing through it

Note 1 to entry: This transmembrane pressure difference allows the membrane to selectively exclude or permit certain substances based on its pore sizes or other specific properties.

3.1.9
feed pressure
pressure applied to the feed side of a membrane in a membrane separation system

3.1.10
pure-water flow rate
volume of pure water passing through a membrane per unit time under specific conditions

3.1.11
permeate flux
volume of permeate produced per unit membrane area per unit time

Note 1 to entry: Permeate flux is typically expressed in L/m^2·h.

4 Abbreviations

ASTM the American Society for Testing and Materials

CMFI crossflow membrane fouling index

FB fine bubble

MB microbubble

MFI modified fouling index

RO reverse osmosis

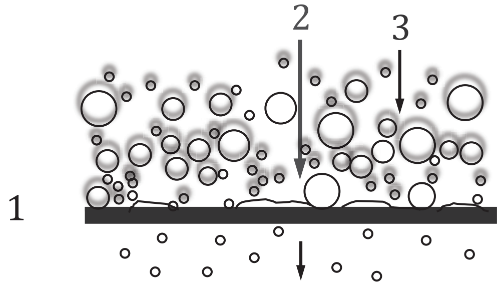
UFB ultrafine bubble

5 Principle

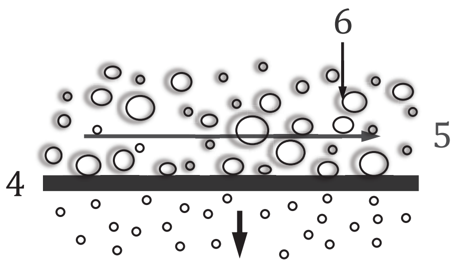
5.1 General

In this method, the CMFI is used to assess the effect of FBs on membrane fouling. The CMFI offers an approach that is different from the one offered by the modified fouling index (MFI) established by the ASTM, which determines the fouling potential of the feed water of membrane systems based on a similar principle that involves measuring fluid flow through the membrane filter as a function of time. MFI uses dead-end filtration, as illustrated in Figure 1(A), which is not suitable for evaluating water containing FBs because hydrophobic bubbles accumulate on the membrane surface and adhere to it, reducing its effective filtration area. This leads to inaccurate results; thus, the beneficial effect of UFBs on membrane fouling mitigation cannot be accurately assessed using this method.

In practice, crossflow filtration, as shown in Figure 1(B), is typically used in wastewater treatment to control membrane fouling. Therefore, to accurately evaluate the effect of FBs on fouling mitigation, the assessment method should employ crossflow filtration. The CMFI used in the proposed method addresses this problem and provides a more accurate assessment of membrane fouling.



(a)



(b)

Key

1	membrane
2	feed flow
3	applied pressure
4	membrane
5	feed flow
6	applied pressure

Figure 1 — Schematic of filtration modes (a) Dead-end filtration with indications of water flow and pressure directions; (b) Crossflow filtration with indications of water flow and pressure directions

In the CMFI-based method, FBs pass through membranes in a crossflow manner during which changes in the filtration rate are continuously monitored. CMFI is calculated based on the collected data regarding the change in the flow rate over time. The FB contribution to membrane fouling prevention can be effectively determined by comparing the CMFI values for the same test sample with and without FBs.

5.2 Crossflow membrane fouling index

To determine the CMFI, the fluid flow through the membrane filter is measured as a function of time. Thus, the relationship between the flux and cumulative permeate volume is expressed as Formula (1)

$$J(t) = \frac{1}{A} \frac{dV(t)}{dt} \tag{1}$$

where

$V(t)$	accumulated permeate volume (L),
A	membrane area (m^2),
$J(t)$	flux ($L/m^2/h$),
t	filtration time (s),
$\frac{dV(t)}{dt}$	derivative of V with respect to time ($L \cdot s^{-1}$).

Darcy’s law, describing fluid flow through a porous medium (the membrane), is given as follows (Formula (2)):

$$J(t) = \frac{\Delta P}{\eta R_t} \tag{2}$$

where

ΔP	applied transmembrane pressure (Pa),
η	water viscosity (Pa s),
R_t	total resistance, including membrane resistance and fouling resistance (s/m).

Combining Darcy's law with the flux relationship gives (Formula (3)):

$$\frac{dV(t)}{dt} = \frac{\Delta PA}{\eta R_t} \tag{3}$$

The total resistance R_t is the sum of the membrane resistance and fouling resistance, which is given as follows (Formula (4)):

$$R_t = R_m + R_f \tag{4}$$

where

R_m	membrane resistance (s/m),
R_f	fouling resistance (s/m).

The fouling resistance is linearly related to the cumulative permeate volume, as given by (Formula (5)):

$$R_f = \frac{IV(t)}{A} \tag{5}$$

where I represents fouling potential index (m^{-2}).

Substituting Formulas (3), (4), and (5) into Darcy's law gives Formula (6):

$$\frac{dV(t)}{dt} = \frac{\Delta PA}{\eta\left(R_m + \frac{IV(t)}{A}\right)} \tag{6}$$

Separating the variables $V(t)$ and t gives Formula (7):

$$dt = \frac{\eta\left(R_m + \frac{IV(t)}{A}\right)}{\Delta PA} dV \tag{7}$$

Integrating both sides from 0 to $V(t)$ gives Formula (8):

$$t = \frac{\eta}{\Delta PA}\left[R_m V(T) + \frac{IV(t)^2}{2A}\right] \tag{8}$$

During the cake filtration period, resistance and cumulative permeate volume $V(t)$ are linearly related, and the slope (b) represents the fouling tendency of the driven permeate, which is shown in the following formulas (Formulas (9) and (10)):

$$\frac{t}{V(t)} = \frac{1}{Q_{avg}} = \frac{\eta R_m}{\Delta PA} + \frac{\eta I}{2\Delta PA^2} V(t) \tag{9}$$

$$b = \frac{\eta I}{2\Delta PA^2} = \frac{d}{dV(t)}\left(\frac{t}{V(t)}\right) \tag{10}$$

where Q_{avg} average flow rate (L/s).

The CMFI is calculated based on the slope of the t/V vs. V curve and is given by Formula (11). The minimum value of the slope (tanα) is equal to CMFI. The CMFI is then corrected using η, A_0, and ΔP_0, as expressed in

Formula (12). Therefore, it is independent of water viscosity, membrane area, and applied transmembrane pressure.

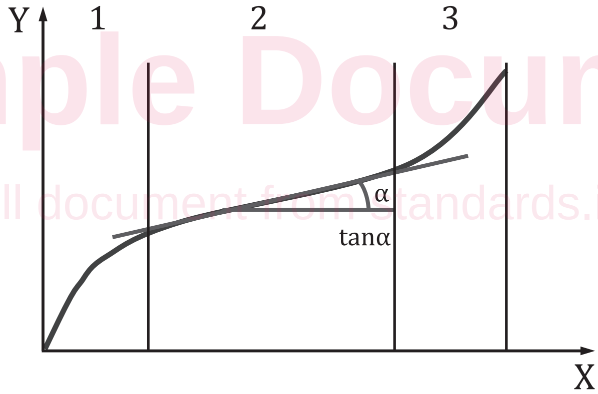
$$\tan\alpha = \frac{\eta \cdot I}{2 \cdot \Delta P A^2}, \quad (11)$$

$$C = \tan\alpha \times \frac{\eta_{20}}{\eta} \times \frac{\Delta P}{\Delta P_0} \times \left(\frac{A}{A_0}\right)^2 \quad (12)$$

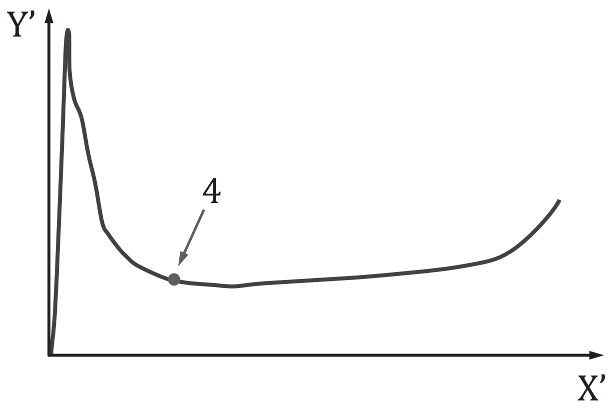
where

η_{20}	water viscosity at 293,15 K (Pa s),
C	is crossflow membrane fouling index (CMFI),
A_0	membrane area used in the control group during the crossflow membrane fouling index (CMFI) measurement (m^2),
ΔP_0	transmembrane pressure applied in the control group during the crossflow membrane fouling index (CMFI) measurement (Pa).

The minimum value of tan α is equal to the CMFI because the filtration mechanism frequently blocking filtration at the start, resulting in a high slope. Next, cake filtration begins and gradually becomes the governing mechanism until cake compression is initiated, which increases the slope. Figure 2(A) shows the tan α calculated based on the t/V vs. V curve. Figure 2(B) shows that tan α is dependent on time and the minimum value of tan α is equal to the CMFI.



(A)



(B)

Key

X	$V(t)$ (L)
Y	$t/V(t)$ (s·L^{-1})
X'	t (s)
Y'	tanα (s·L^{-2})
1	blocking filtration stage
2	cake filtration stage
3	cake compression stage
4	CMFI

Figure 2 — Analysis of tan α during different filtration phases. (A) tan α calculated based on the *t*/*V* vs. *V* curves during blocking filtration, cake filtration, and cake compression phases. (B) Relation between tan α and filtration time

6 Apparatus

6.1 General

The apparatus required for conducting the CMFI-based membrane fouling evaluation is specified in 6.2 to 6.13.

6.2 Water pump

A water pump shall generate pressure in the range of 0,1–0,6 MPa to ensure the passing of untreated wastewater through the membrane module.

6.3 Membrane module

Suitable membrane materials shall include ceramics, organic materials, metals, nonpolar materials, and composites. Several membrane structures, such as single-channel tubular, multichannel tubular, hollow fibre, and flat sheet membranes, can be used. The membrane shall include an inlet for feed water, a circulation outlet, and a permeate outlet. The pore size and surface area of the membrane shall be within the ranges of 1–2 000 nm and 0,005–30 m^2, respectively.

6.4 Measuring device

A device shall be capable of measuring the mass or flow rate of the permeate at a scale interval not greater than 0,01 g, and it shall support continuous data recording and transmission.

6.5 Data recording and storage device

A device with a recording interval of 0,01–10 s shall be used for real-time data logging and storage.

6.6 Pressure sensor installed on the membrane loop

A sensor shall be used for monitoring pressure on the circulating side of the membrane.

6.7 Pre-membrane pressure sensor

A sensor shall be used for measuring the feed pressure of the fluid entering the membrane.

6.8 Pressure-reducing valve

A valve shall be used to maintain the feed pressure of wastewater entering the membrane module at a constant pressure.

6.9 Temperature sensor

A sensor shall measure the temperature of entering wastewater (precision = 0,1 °C).

6.10 Water tanks

Two tanks shall be used, one for the water sample to be tested and the other for the permeate.

6.11 Concentrate container

A container shall be used for storing wastewater that has passed over the membrane surface during crossflow filtration.

6.12 UFB generator

A UFB generator shall be based on any one method or a combination of the thirteen methods described in ISO 20480-3:2021.

6.13 Precision electronic balance

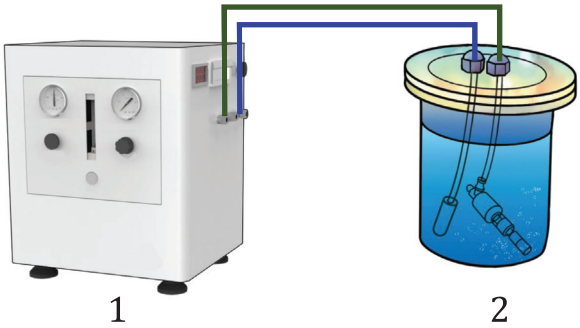
A precision electronic balance shall have an accuracy of at least 0,01 g. It shall be capable of real-time data transmission to a computer or a host system for recording the weight in real time.

7 Procedure

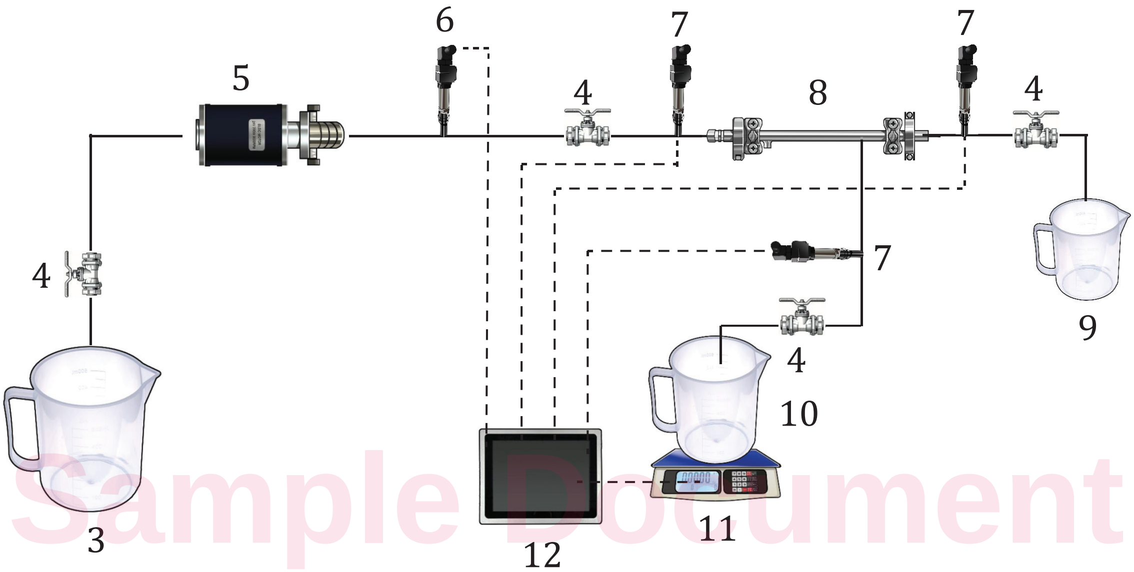
The procedure involves three steps: equipment setup, data collection, and CMFI calculation.

7.1 Equipment setup

The connections and structure of the equipment are shown in Figure 3.



a) UFB Generator



b) Representative apparatus for measuring the CMFI at a constant pressure using a crossflow filtration model

Key

1	UFB generator
2	reactor
3	inlet reservoir
4	pressure-reducing valve
5	pump assembly
6	temperature sensor
7	gauge pressure sensor
8	membrane module
9	concentrate container
10	outlet reservoir
11	precision electronic balance
12	data recording and storage device

Figure 3 — Apparatus for UFB generation and CMFI measurement using a crossflow filtration model.

7.2 Data collection

Data collection is performed as follows:

Two sets of experiments are performed sequentially: (1) in the control group, the membrane module is directly filled with wastewater for membrane filtration experiments; (2) in the UFB-treated group, a UFB