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Validation and optimization of a new instrument for measuring the performances of ultrafiltration membranes

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ABSTRACT

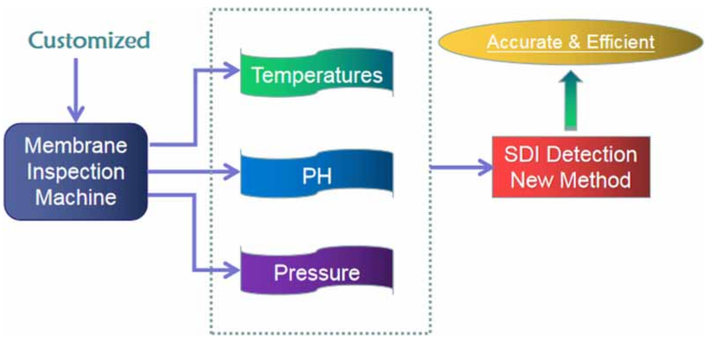
In this study, we introduce the current situation of the membrane inspection industry. We have developed a new integrated rapid membrane inspection machine (MIM) to test the performances of nanofiltration or ultrafiltration membranes by measuring the silt density index (SDI) value (i.e., the key performance indicator) of the water filtered. This study optimizes many key conditions include temperature, pH value and pressures, then establishes a new SDI detection method of ultrafiltration membranes by using MIM. We set the temperature of the test liquid to 25 °C, pH in the range of 7–8, and pressure to approximately 0.10 MPa. The results reflect that MIM can be used to effectively determine the SDI of ultrafiltration membranes using the new method and the results of the method validation are satisfactory.

Key words: membrane inspection machine (MIM), optimization, performance testing, silt density index (SDI), ultrafiltration membrane (UF), validation

HIGHLIGHTS

- A new integrated rapid membrane inspection machine (MIM) is developed.
- The performances are reported of ultrafiltration membranes by measuring silt density index (SDI) value.
- By optimizing the conditions, we established a new SDI detection method of ultrafiltration membranes by using MIM.
- The new method is efficient and accurate.
- MIM can be used in ultrafiltration or nanofiltration in the future, and can also be used to determine the water flux and desalination rate of membranes.

GRAPHICAL ABSTRACT



1. INTRODUCTION

At present, water treatment is of more and more concern. With the continuous development of new materials (Beshkar *et al.* 2015; Moshtaghi *et al.* 2016; Heidari-Asil *et al.* 2020), membrane treatment technology is more and more widely used in water treatment. There are many disposal methods and treatment technologies. Panagopoulos outlined the Zero Liquid Discharge (ZLD) approach (Panagopoulos *et al.* 2019), and many mitigation measures (Panagopoulos & Haralambous 2020).

Ultrafiltration is widely used in many fields including industrial wastewater treatment, domestic sewage reuse, domestic water purification, and seawater desalination pretreatment because of its advantages of high separation efficiency, no phase transition during the separation process, and green energy savings (Wang *et al.* 2013a; Zhao *et al.* 2014). Ultrafiltration is used for brine treatment in desalination industries and other industries (Panagopoulos 2021). However, presently, the market contains many ultrafiltration membrane products, whose qualities are mixed. The focus of the membrane inspection industry is on accurately evaluating the performance of ultrafiltration membranes. Notably, the quality of the filtered water is often the most direct reflection of the membrane quality. In a study, a bench-scale hollow fiber ultrafiltration system was designed and built to assess the impact of operational parameters on membrane performance and fouling (Waterman *et al.* 2016).

The silt density index (SDI) is an important index for evaluating the quality of the water filtered by an ultrafiltration system, and it can effectively represent the content of particle matter, colloidal material, and suspended matter in the water (Wolf *et al.* 2005; Wang *et al.* 2014). In real-world scenarios, SDI is usually taken as an effective operation-monitoring parameter of ultrafiltration membrane water treatment systems. The current operating experience with these systems shows that only when the SDI is less than 3 can strict requirements for water quality be met (Vrouwenvelder *et al.* 2003; Halpern *et al.* 2005; Wang *et al.* 2013b, 2013c). With reference to the EPEC standard (2018), *Immersion Hollow Fiber Ultrafiltration Membrane Module*, coded EQE481502004S, there is a clear requirement for the SDI value of the water quality in the film product related performance testing. Therefore, the ability to accurately test the SDI of the water filtered by an ultrafiltration system can be used to effectively evaluate the quality of ultrafiltration water production. Thus, the SDI of the water filtered by an ultrafiltration system is a significant tool for guiding ultrafiltration pretreatment technology, efficient management and maintenance of membrane water treatment systems, and improving the treatment effect of ultrafiltration water.

The current SDI test is based on the ASTM D4189-07 standard (2007) of the American Society for Testing and Materials. It mainly specifies methods to test the SDI of water. However, it does not specify a uniform condition for testing the water filtered by an ultrafiltration system. Although the standard describes the performance requirements of the filter membrane used for SDI testing, the SDI test membrane performance is different in the market. SDI and modified fouling index characterization methods are well known for evaluating the membrane fouling potentials of the dispersed particulate matter (e.g., suspended solids and colloids) in a feed (Brauns *et al.* 2002).

We have manufactured a new instrument for measuring the performance (MIM) of ultrafiltration membranes. Different than the common film detectors on the market, MIM has double channels and can be used in single or double channels (see Figure 1(a) and 1(b)). Moreover, MIM is highly economical and replicable, and it can test the key performances of nanofiltration or ultrafiltration membranes. If it tests the performance of a membrane in two channels, it is more efficient and more time-saving than in a single channel.

Therefore, following the ASTM D4189-07 standard, we comprehensively analyzed the influence of the properties of the filter membrane and feed liquid on the test results of the SDI value of the water filtered by an ultrafiltration system, following which we validated and optimized conditions. We then realized the accurate and efficient detection and analysis of SDI.

2. MATERIALS AND METHODS

2.1 Terminology

2.1.1. 5 NTU deionized water

Standardized deionized water with turbidity of 5 NTU.

2.1.2. SDI_5, SDI_{10}, or SDI_{15}

The ultrafiltration water sample taken from the initial filtration 500 mL and the time of filtering 500 mL of the same water sample after 5, 10, and 15 min. The SDI of the water sample was calculated as follows:

$$SDI_\tau = \left(1 - \frac{\tau_0}{\tau_1}\right) \times 100/\tau \tag{1}$$

where

SDI_τ denotes the SDI value at time τ;

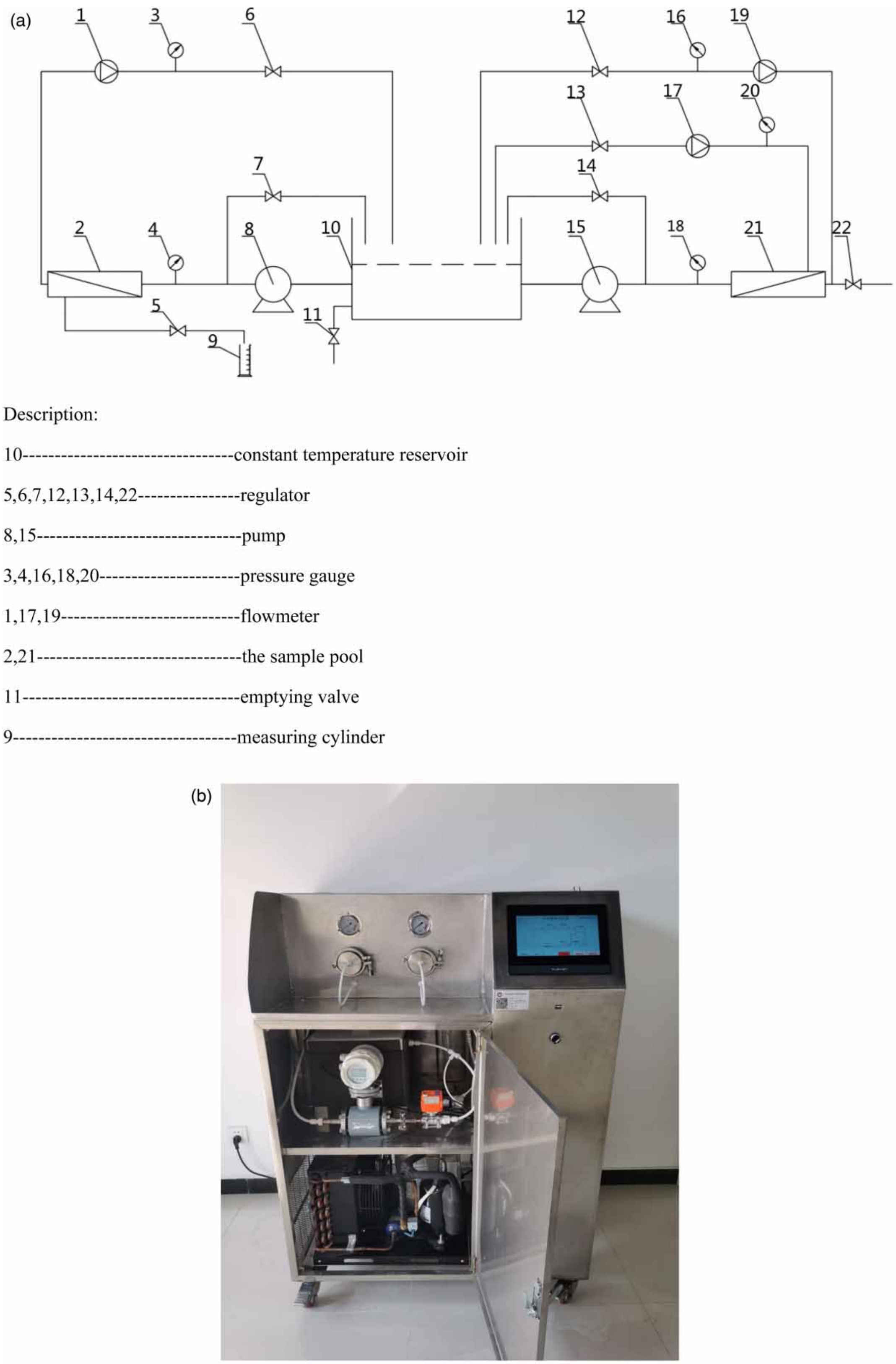


Figure 1 | (a) Diagram of the integrated membrane performance testing device. (b) Image of the integrated membrane performance testing device.

τ denotes the interval between the collection of two water samples; it can be 5, 10, or 15 min;
τ_0 denotes the initial collection time of 500 mL of filtered water, s;
τ_1 denotes the time spent in collecting 500 mL of filtered water after time, s.

According to the ASTM D4189-1995 *Standard Test Method for Silt Density Index (SDI) of Water*, the experimental interval time can be 5 minutes, 10 minutes or 15 minutes, but SDI_{15} is still the most commonly used in many standards and in practice.

2.2. Instrument and reagents

- 2.2.1. New instrument for measuring the performance of the membrane.
- 2.2.2. A stopwatch, scale (Mettler Toledo).
- 2.2.3. Reagents: hydrochloric acid (analytically pure), sodium hydroxide (analytically pure), polyethylene glycol (weight-averaged molecular weights of 6,000, 10,000, and 20,000), and deionized water (conductivity less than 10 μS/cm).
- 2.2.4. Ultrafiltration membrane: randomly select ten kinds of different products from different enterprises.

2.3. Experimental steps

- 2.3.1. The MIM was connected, as shown in Figure 1.
- 2.3.2. Four samples of the same membrane were prepared to be tested.
- 2.3.3. The samples were washed with deionized water.
- 2.3.4. Two of the prepared samples in 2.3.1 were selected and placed in the sample pools (Figure 1(a) parts 2 and 21) for testing.
- 2.3.5. The inlet pressure was slowly adjusted to 0.150 ± 0.005 MPa, and the pre-pressure-treatment was for 30 min.
- 2.3.6. All other parameters were fixed to optimize the temperature conditions. The test water temperature was optimized from 15 °C to 45 °C, in order to determine the optimal experimental temperature.
- 2.3.7. Under the optimal experimental temperature, other experimental conditions were fixed to optimize the pH value. The test pH value was then optimized from 6 to 11, in order to determine the optimal experimental pH value.
- 2.3.8. Under the optimal experimental temperature and pH value, other experimental conditions were fixed to optimize the pressure. The test pressure was optimized from 0.09 to 0.105 MPa and the pressure was stabilized for 10 min.
- 2.3.9. A total of 500 mL filtered water was collected using a measuring cylinder, and the corresponding time was recorded as the collection time with a stopwatch.
- 2.3.10. Another two samples were taken and simultaneously tested according to the above steps. That means two replications were performed for the experiments.

2.4. Experimental content

- 2.4.1. Condition optimization: temperatures, pH, and pressure.
- 2.4.2. On the basis of 2.4.1, we tested ten ultrafiltration membranes. Simultaneously, the test results were compared with those obtained by the research group of the Zhuhai Branch of Beijing Institute of Technology.

3. RESULTS

In Figure 1, the feed liquid in the constant temperature liquid storage tank is put into the sample pool at the same time to collect the filtrate in the corresponding produced water collection and measurement container of the sample pool in unit time, and the volume of filtrate and the SDI value of the filtrate are measured respectively.

3.1. Optimization

3.1.1. Temperature optimization

Deionized water of 5 NTU was used for the test. The result of temperature optimization is listed in Table 1.

It can be seen from Table 1 that the SDI value increases with the increase of temperature. The higher the temperature is, the more easily the secondary pollution will occur, resulting in the increase of SDI. However, there is little difference between the 15 and 25 °C data, and a little lower temperature has little effect. So we choose 25 °C as close to normal temperature for the method temperature.

Table 1 | SDI of water filtered by the UF at different temperatures

	15 °C	25 °C	35 °C	45 °C
SDI_5	2.01	1.87	2.29	2.58
SDI_{10}	1.49	1.46	1.52	1.71
SDI_{15}	1.39	1.32	1.40	1.48

3.1.2. pH optimization

The solution pH was adjusted by using hydrochloric acid and sodium hydroxide solutions. The filtration degrees of polyethylene glycol solutions (PEG) of different molecular weights can reflect the filtration performance of the membrane. The result of pH optimization is seen in Table 2.

High or low pH of water affects the existing form of the substances present in the water, increasing the SDI of the water filtered by the ultrafiltration membrane. When pH >8, the soluble ions (Ca^{2+}, Mg^{2+}, etc.), iron ions, and aluminum ions in the water will form solids or colloids with increase in pH, and will then be deposited at the bottom. When pH < 7, it is advantageous only for the samples containing iron ions, but has little effect on common sewage and seawater, thereby impacting the SDI test. Therefore, the pH is optimized in the range of 7–8.

3.1.3. Pressure optimization

Deionized water of 5 NTU was used for the test. The results of pressure optimization are listed in Table 3.

The operating pressure of UF is a very important factor when it is used. Generally, in order to improve the membrane flux, the operating pressure will be higher. Higher membrane flux leads to higher filtration efficiency. For the pressure with little difference, we choose 0.1 MPa, which is easier to operate as the experimental pressure.

3.2. Testing

We opted for a temperature of 25 °C, pH of 7.5, and pressure of 0.10 MPa for the next test.

Deionized water with a turbidity of 5 NTU was used to test the ultrafiltration membranes of ten different brands, and the test data were compared with those of other inspection institutions, as shown in Table 4. That is, reference to the statistics shows a good agreement between the results obtained using this instrument and those achieved by other institutions using their own methods. According to the standard DL/T 588-2015, the absolute SDI_{15} value of the difference detected by the two mechanisms is not greater than 0.45, which is the reproducibility. It can be seen that the relative standard deviation of test results between different laboratories is satisfactory.

4. CONCLUSIONS

To effectively test the filtration performance of an ultrafiltration membrane, we set the temperature of the test liquid to 25 °C, pH in the range of 7–8, and pressure to approximately 0.10 MPa. Using the MIM instrument, we could quickly detect the SDI

Table 2 | SDI of water filtered by the UF at different pH values

pH	6	7	8	9	10	11
PEG-10,000 – 500 mg/L – SDI_{15}	1.98	1.82	1.63	2.59	4.14	4.59
PEG-20,000 – 1,000 mg/L – SDI_{15}	3.65	3.22	3.33	4.21	4.39	4.55

Table 3 | SDI of water filtered by the UF at different pressures

Pressure, MPa	0.09	0.095	0.10	0.105
SDI_{15}	1.93	1.79	1.63	1.64

Table 4 | Comparison of ten samples between two institutions

	Our tested-SDI_{15}	Third party tested-SDI_{15}	Reproducibility
Sample 1	2.12	2.25	0.13
Sample 2	3.15	3.41	0.26
Sample 3	1.91	1.94	0.03
Sample 4	1.65	1.52	0.13
Sample 5	1.78	1.84	0.06
Sample 6	1.52	1.40	0.12
Sample 7	4.63	4.72	0.09
Sample 8	1.43	1.52	0.09
Sample 9	1.65	1.55	0.10
Sample 10	1.78	1.84	0.06

value of the water filtered by the ultrafiltration membrane. The ultrafiltration water samples were taken from the initial filtration 500 mL and the time of filtering 500 mL of the same water sample after 15 min. The results reflect that MIM can be used to effectively determine the SDI of UF. Through the two sample pools of MIM, the time of collecting 500 mL filtrate will be shortened by nearly half, which greatly improves the efficiency. Otherwise, the new method and the results of the method validation are satisfactory. For the future, perhaps these studies about the MIM instrument could be used for measuring other parameters, including the desalination rate and water flux. The MIM instrument may fulfil the need for realizing quality control in water treatment facilities. In the future, with the application of MIM-related research results, we plan to formulate relevant methods and standards, and make contributions to unifying the national ultrafiltration membrane water treatment and detection industry.

ETHICAL APPROVAL

No ethical approval was obtained because this study did not involve a prospective evaluation, and did not involve laboratory animals.

CONSENT TO PARTICIPATE

Not applicable.

CONSENT TO PUBLISH

Yes.

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COMPETING INTERESTS

This manuscript has not been published or presented elsewhere in part or in entirety and is not under consideration by another journal. All study participants provided informed consent, and the study design was approved by the appropriate ethics review board. We have read and understood your journal's policies, and we believe that neither the manuscript nor the study violates any of these. There are no conflicts of interest to declare.

AUTHOR CONTRIBUTION STATEMENT

LYG worked the concept and design, analyzed the data and made important revisions to the paper. YR collected data, transplanted and made important revisions to the paper. QD collected data and made important revisions to the paper. JML collected data, interpretation of data and made important revisions to the paper. SCF collected data and made important revisions to the paper. DJ* designed the work and substantively revised it. All authors approved of final paper to be published.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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