

MEASUREMENT OF CAKE THICKNESS ON MEMBRANE FOR MICROFILTRATION OF YEAST USING ULTRASONIC POLYMER CONCAVE TRANSDUCER

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An ultrasonic measurement method was applied to obtain information about the properties of cake formed on the membrane in microfiltration of suspensions of yeast. The microfiltration was carried out at low applied pressure in order to assess that the cake formed was incompressible. A spherically concave transducer made of piezoelectric films of a vinylidene fluoride-trifluoroethylene copolymer was adopted. The surface shape of the cake was found not to be flat by the reflection technique. An on-line method of measuring cake thickness was developed by using the transmission technique, and the results showed good agreement with data reported elsewhere.

Introduction

Ultrasonic measurement is a very useful method for obtaining information about the process conditions and consistent product properties from outside the process boundary without perturbing the process. There are many industrial applications for ultrasonic measurement such as flowmetry, thermometry and interface sensing. However, published work concerning its use in chemical engineering is relatively limited.^{1,6)}

Membrane separation has become a common operation in the chemical and biochemical industries recently. In this operation, knowledge of the cake properties is significant because the permeation rate is limited by the formation of cake. But this problem has rarely been investigated, due to difficulty of measurement.

In this work, an ultrasonic method of measuring cake thickness on microfiltration membrane was developed. The microfiltration of a suspension of yeast was conducted at low applied pressure so that no compression of cake occurred.

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1. Ultrasonic Transducer

One of the present authors (K.K.) and his co-workers have obtained a wide-band ultrasonic transducer made of piezoelectric films of a ferroelectric polymer, vinylidene fluoride (VDF) trifluoroethylene (TrFE) copolymer with a comonomer ratio of 78/22.⁴⁾ This polymer film transducer is a flexible electroacoustic transducer in which the electroacoustic action occurs by a piezoelectric mechanism.

1.1 Concave spherical transducer³⁾

The concave spherical transducer used is shown in Fig. 1. Its radius of curvature was 4.5 mm. A brass rod was embedded in a polymer cylinder. The top end of the rod was polished into a spherical concavity. The rod then acted as one of the electrodes of the transducer.

Procedures for preparing the film were as follows. A solution of polymer was dropped onto the concave electrode. A centrifuge was used to spread the fluid film. The solution was dried, and the resultant film was annealed at about 145°C. The film was then polarized under an electric field. Concave transducers with large aperture angles are easily fabricated by this process.

2. Measurement of Cake Thickness by Reflection Technique

The acoustic impedance of cake is supposed to be similar to that of the membrane because of their comparable densities. Therefore, it seems that the sensing of the interface between cake and membrane is difficult. For ease of measurement and high sensitivity, the reflection technique was adopted to detect the echo from the interface.

2.1 Experimental

1) Sound speed in cake To convert the measured time of the round-trip interval for an impulse to thickness, the speed of sound in several media must be known. These values were determined by measuring the time required for sound to travel a given distance between two transducers, in a space filled with the medium, by a digital oscilloscope. The cake formed under the same conditions as the experiment for measuring the thickness was scratch out of the membrane surface and provided as a sample. Figure 2 shows the measurement results. It can be seen from the figure that the speed of sound is almost constant in spite of differences in frequency. The average value of the speed of sound in cake is shown in Table 1 along with those in water and suspensions of yeast.

2) Experimental set-up A block diagram of the measuring system is shown in Fig. 3. The impulse is generated with a pulse generator (Picosecond Pulse

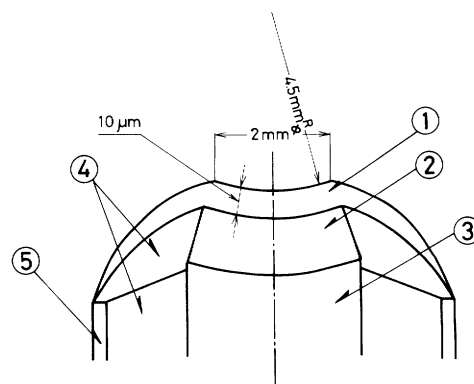


Fig. 1. Construction of P(VDF-TrFE) concave transducer
①, Al electrode; ②, P(VDF-TrFE); ③, metal rod; ④, polymer; ⑤, metal cylinder

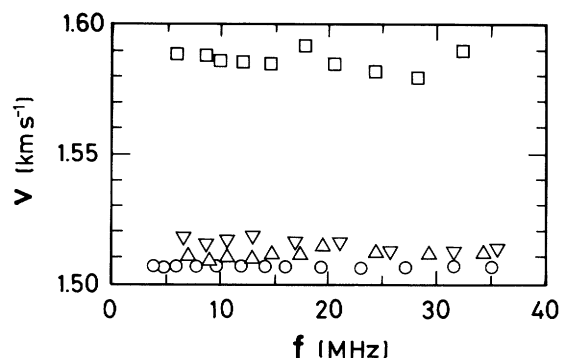


Fig. 2. Speed of sound in distilled water, suspensions and cake of yeast

○, distilled water; △, suspension of yeast (5 g/l); ▽, suspension of yeast (10 g/l); □, cake of yeast

Table 1. Average value of speed of sound in distilled water and in suspensions and cake of yeast

Sample	Sound speed [$\text{m} \cdot \text{s}^{-1}$]
Distilled water	1506
Suspension of yeast (5 g/l)	1511
Suspension of yeast (10 g/l)	1514
Cake of yeast	1593

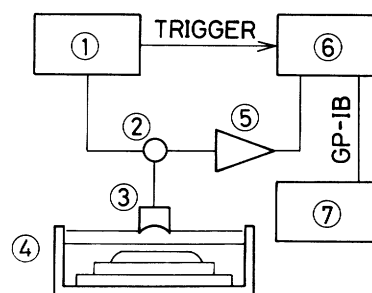


Fig. 3. Block diagram for measurement of cake thickness by reflection technique

①, pulse function generator; ②, directional coupler; ③, transducer; ④, sample; ⑤, amplifier; ⑥, digital oscilloscope; ⑦, personal computer

Labs., model-1000B) and introduced to a transducer. The ultrasonic sound is transmitted to a sample. An echo from the sample is received by the transducer again and stored temporarily in a digital memory (LeCroy 9400). The data are then sent to a computer via GPIB. By scanning in a horizontal direction, a clear two-dimensional image can be obtained.

3) **Sample** The sample was prepared as follows. A suspension of bread yeast of 2.0 g/l concentration was microfiltered in an unstirred batch cell of 110 ml volume and with a membrane area of 12.4 cm², as described in the previous paper⁵. The low applied pressure of 100 kPa used in this work shows that the cake formed is incompressible⁵.

The membrane on which the cake of yeast formed was detached from the cell and then coated with jelly to prevent diffusion into water. The membrane used was Millipore HA of catalogued mean pore size 0.45 μm.

2.2 Results and discussion

The echo of the impulse at 40 MHz is displayed in Fig. 4. Four echoes can be seen from the figures, corresponding respectively to that from the cake surface, from the cake/membrane interface, from the bottom of the membrane and from the metal supporting membrane. The gap observed between membrane and metal may be penetrated by jelly. The signal-to-noise ratios (S/N) for these echoes range from 5 to 20 dB. The value for the echo from cake/membrane interface was low but good enough to detect. The two-dimensional image is shown in Fig. 5. It was obtained by scanning the transducer in the direction perpendicular to the membrane surface at every 0.02 mm. The detailed surface shape of the cake was obtained.

The cake thickness determined by timing the round-trip interval and the speed of sound was 0.54 mm, which was close to the thickness of 0.55 mm measured by using the micrometer with a needle linked to the device for electrical resistance measurement⁵. This means that ultrasonic measurement is applicable for determination of cake thickness on a microfiltration membrane. However, the reflection technique is not suitable for on-line measurement.

3. On-line Measurement of Cake Thickness by Transmission Technique

For on-line measurement the transmission technique was adopted.

3.1 Experimental

1) **Attenuation coefficient of cake** To measure the cake thickness by the transmission technique, the attenuation coefficient of the cake must be known in advance. The attenuation coefficient α is given by the following equation².

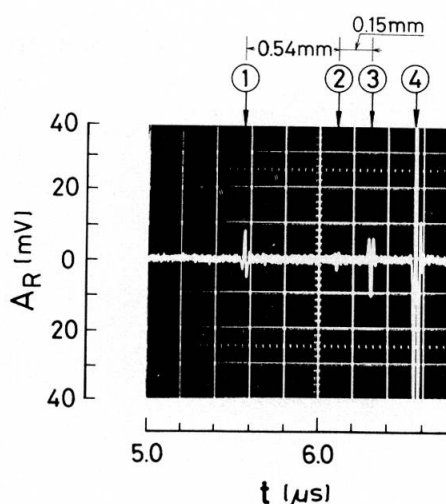


Fig. 4. Reflected echo signals (2V/div., 0.2 μs/div.)

①, echo from cake surface; ②, echo from cake/membrane interface; ③, echo from bottom of membrane; ④, echo from metal supporting membrane

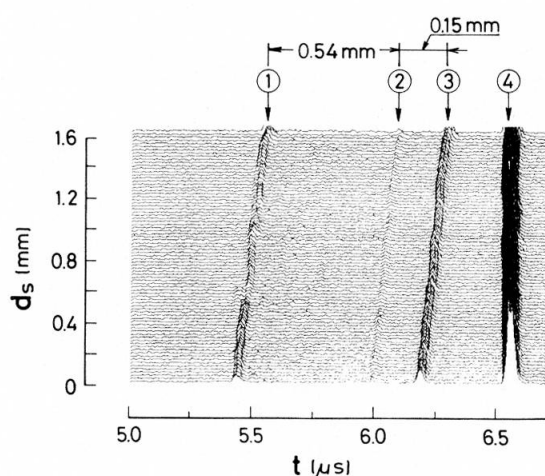


Fig. 5. Image of cake on membrane in horizontal direction

①, cake surface; ②, cake/membrane interface; ③, bottom of membrane; ④, metal supporting membrane

$$\frac{\alpha}{f^2} = \frac{20 \log(A_1/A_0)}{df^2} \quad (1)$$

where d is a given path length, A_1 or A_0 is the amplitude of a wave at an initial point or that at a point where the wave has propagated over the distance d . Figure 6 shows the result of the attenuation coefficients for the cake, suspensions of yeast and distilled water. The cake was prepared by the same procedure used for measuring the speed of sound in the cake. This figure indicates that the attenuation coefficients decrease with increase in the frequency, but that above 30 MHz they become constant. In this work, we used ultrasound of frequency 40 MHz for the on-line measurement of cake thickness. The values of the attenuation coefficients at this frequency are summarized in Table 2. The value for the cake is more than 20 times those for distilled water and suspen-

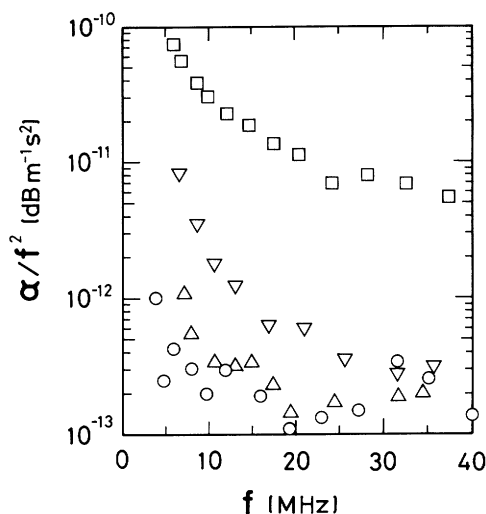


Fig. 6. Attenuation coefficients for distilled water, suspensions and cake of yeast

○, distilled water; △, suspension of yeast (5 g/l); ▽, suspension of yeast (10 g/l); □, cake of yeast

Table 2. Average value of attenuation coefficient of distilled water and in suspensions and cake of yeast above frequency of 30 MHz

Sample	Attenuation coefficient [$\text{dB} \cdot \text{m}^{-1} \cdot \text{s}^2$]
Distilled water	1.212×10^{-13}
Suspension of yeast (5 g/l)	1.714×10^{-13}
Suspension of yeast (10 g/l)	2.606×10^{-13}
Cake of yeast	5.936×10^{-12}

sions of yeast. Therefore, it may be concluded that almost all the attenuation of sound waves occurs in the cake and thus the cake thickness can be measured by the transmission technique.

2) **Experimental apparatus** The block diagram of the experimental set-up is shown in Fig. 7, which is essentially similar to that shown in Fig. 3 except for having two concave transducers. The transmitting transducer was fixed inside the unstirred batch cell and the receiving one was mounted on a precision stage assembly outside the cell to set the transducer in a suitable position to receive the sound to traverse the cake. The position of the transmitting transducer was 5 mm above the membrane surface in order to take a focus. Because the ultrasonic frequency was 40 MHz, the response time of the transducer was 25 μs . The cell had the same geometry as that described in the previous section. The applied pressure was 100 kPa and the concentration of bulk suspension was 2.0 g/l. Therefore, we could neglect the compression of the cake⁵⁾.

3.2 Results and discussion

The change in amplitude of the propagated wave divided by the initial one with elapsed time is shown in Fig. 8. The value of the S/N ratio was more than

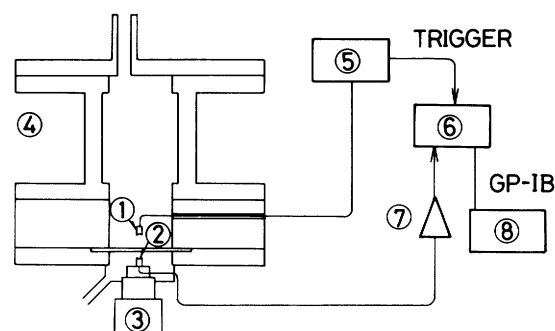


Fig. 7. Block diagram for on-line measurement of cake thickness by transmission technique

①, transmitting transducer; ②, receiving transducer; ③, precision stage assembly; ④, unstirred batch cell; ⑤, pulse function generator; ⑥, digital oscilloscope; ⑦, amplifier; ⑧, personal computer

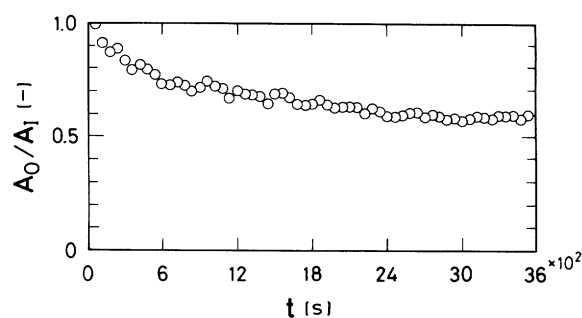


Fig. 8. Change of amplitude of propagated wave

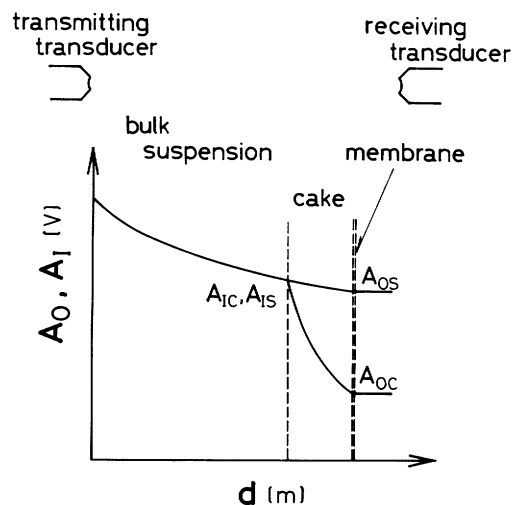


Fig. 9. Notation for determination of cake thickness by transmission technique

40 dB, which means that the experimental error is less than 1%. It can be seen from the figure that the transmission ratio decreases but the rate of decrease becomes slow with increase in time.

To determine the cake thickness, two conditions, with and without cake, as shown in Fig. 9, must be considered. Under no-cake condition, the wave is attenuated only by the suspension of yeast. Once the cake forms, it also weakens the wave in the cake layer.

For each condition, the wave attenuation in the distance δ_c , which corresponds to the cake thickness, can be expressed as follows.

$$\left(\frac{\alpha}{f^2}\right)_{\text{cake}} = \frac{20 \log(A_{IC}/A_{OC})}{\delta_c f^2} \quad (2)$$

or

$$\left(\frac{\alpha}{f^2}\right)_{\text{sus}} = \frac{20 \log(A_{IS}/A_{OS})}{\delta_c f^2} \quad (3)$$

where A_{IC} or A_{IS} is the amplitude of a wave at the position of δ_c distant from the membrane surface and A_{OC} or A_{OS} is that at the membrane surface. The subscript C or S denotes the condition with or without cake formation. Because the region above δ_c is filled with bulk suspension of yeast for each case, A_{IC} should equal A_{IS} . From these equations, we can eliminate A_{IC} , that is, A_{IS} , and obtain the following equation for δ_c .

$$\delta_c = \frac{20 \log(A_{OC}/A_{OS})}{\{(\alpha/f^2)_{\text{sus}} - (\alpha/f^2)_{\text{cake}}\} f^2} \quad (4)$$

Exactly speaking, A_{OC} or A_{OS} differs from the amplitude of a wave at the receiving transducer because attenuation occurs by permeated water in the region between the membrane surface and the transducer. However, the attenuation by water is negligibly small compared with that by cake as shown in Fig. 6. Therefore, the value of the cake thickness could be calculated by using Eq. (4). The results are shown in Fig. 10 along with data measured by the method using the modified micrometer⁵⁾, which is unusable for on-line measurement. The agreement between them is satisfactory.

Conclusion

To measure cake thickness in microfiltration, which is the one of the most important parameters for determining the permeation rate, an ultrasonic measurement technique was developed. The detailed surface shape of the cake was obtained by the reflection technique and the change in cake thickness with elapsed time was measured successfully by the

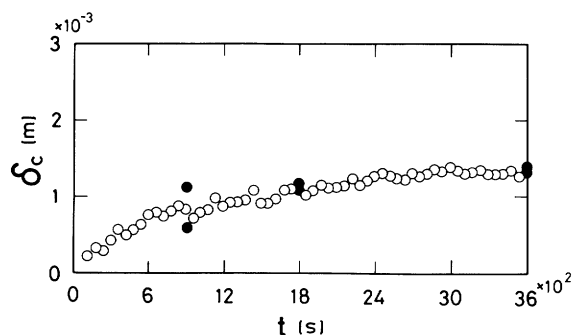


Fig. 10. Calculated result of cake thickness
○, measured by ultrasonic method; ●, measured by modified micrometer⁵⁾

transmission technique.

Nomenclature

A_{I, A_O}	= amplitude of a wave at an initial point or that at a point where the wave has propagated	[V]
$A_{IC}, A_{OC}, A_{IS}, A_{OS}$	= amplitude of a wave as designated in Fig. 9	[V]
A_R	= amplitude of a reflected wave	[V]
d	= distance from transmitting transducer	[m]
d_s	= scanning distance of transducer	[m]
f	= frequency of a wave	[Hz]
t	= time	[s]
v	= sound speed	[m · s ⁻¹]
α	= attenuation coefficient	[dB · m ⁻¹]
δ_c	= cake thickness	[m]

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