CHAPTER IV

EXPERIMENTS

This chapter will be focused on apparatus and experimental procedures of microfiltration with and without ultrasound.

4.1 Apparatus

- Analog oscilloscope model SS-7802 of IWATSU, Japan.
- Power amplifier model P-602 of A-TECHNIC, Thailand.
- Signal generator model AFG-310 of Tektronix, U.S.A.
- Piezoelectric ceramic model SONOX-P4 of Bruel & Kjaer (UK) Ltd, England.
- Ceramic membrane of British Berkefeld, England.
- Particle size analyzer model LS230 of COULTER CORPORATION, U.S.A.
- Microscope model B071 of Olympus, Japan.
- Centrifugal pump model PKm 60-1 of PEDROLLO, Italy.

4.2 Microfiltration Unit

The microfiltration unit and the ceramic membrane used in this thesis are shown in Figure 4-1 and Figure 4-2, respectively. The filtration was carried out using a tubular module. A tubular ceramic membrane with an effective area of 223.2 cm^2 and the pore size of 0.9 microns was installed in a tubular housing, which had a capacity of 2,050 cm^3. Two of identical composite transducers were attached to the outer surface of the housing to supply the ultrasound in case of ultrasonic microfiltration.

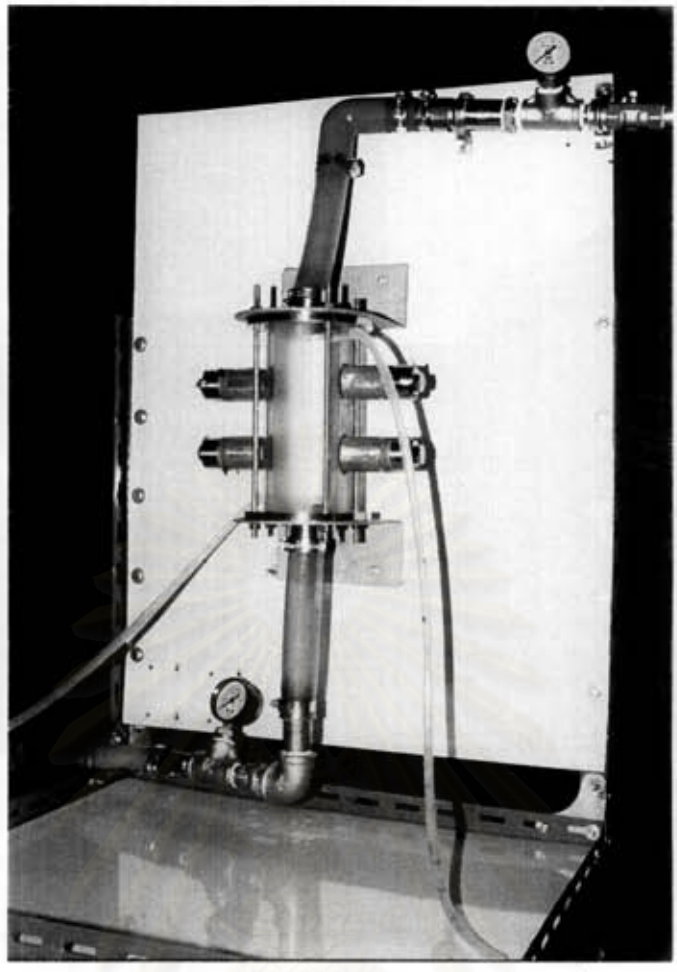

Figure 4-1: Photograph of microfiltration unit

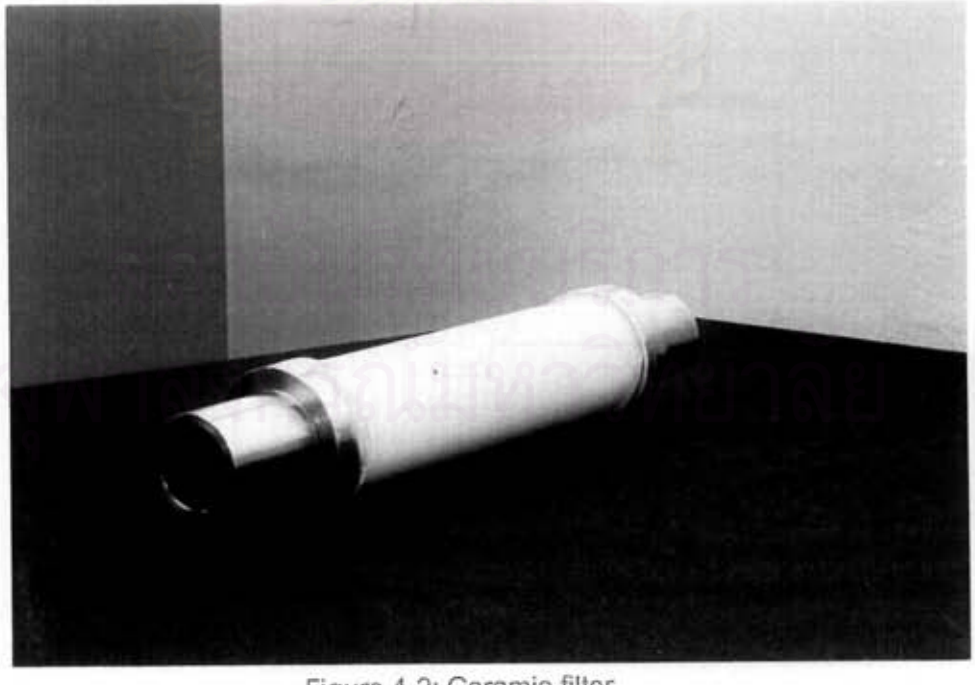

Figure 4-2: Ceramic filter

Figure 4-3: Acoustic power supplied system

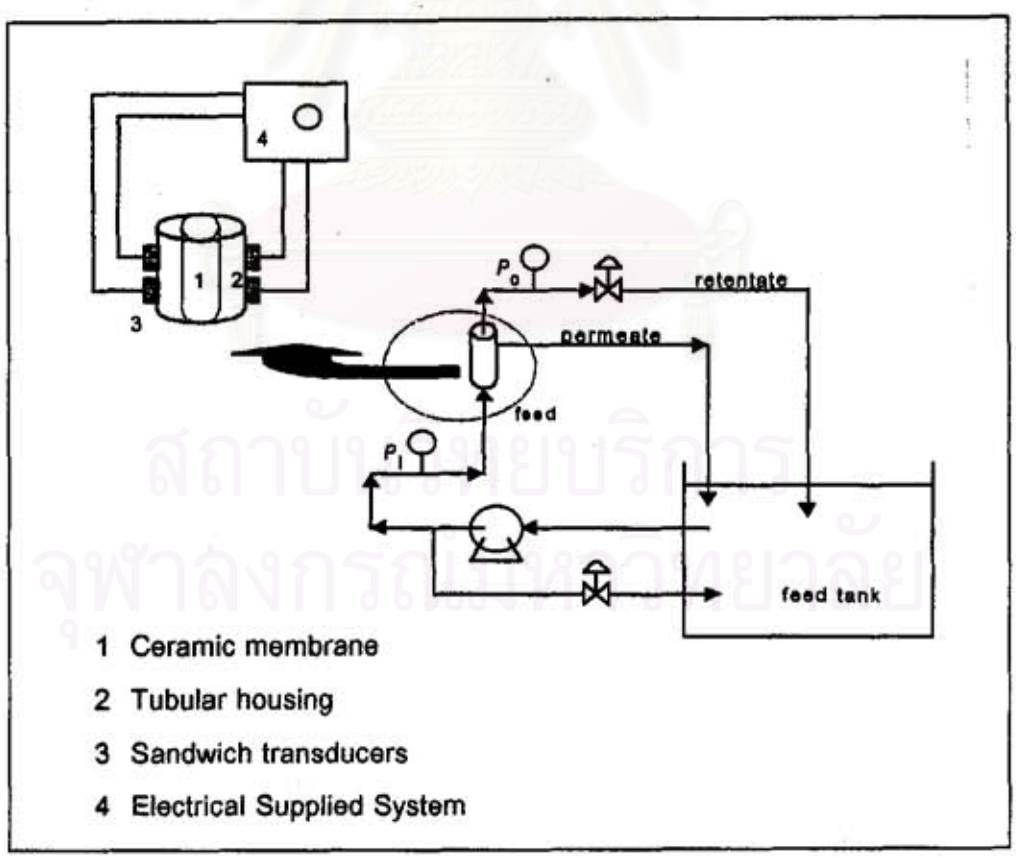

Figure 4-4: The schematic of microfiltration system

4.3 Study of microfiltration characteristics

The objective of our experiment was to elucidate the optimum operating condition for microfiltration of yeast suspension both with and without ultrasound. As much as the microfiltration theory is concerned, there are four parameters that affect the filtration process. Effects of applied pressure, feed flow velocity and feed concentration were thus studied at various operating conditions as illustrated in Table 4-1. To avoid the effect of temperature, it was then controlled at 30 °C throughout the process.

The schematic of microfiltration system is shown in Figure 4-4. The experimental procedures were set up as follows,

Demineralized water was employed for filtration at selected conditions in order to obtain the reference water flux which was used to check the cleanness of the regenerated membrane and to measure the resistance of membrane, R_m.

Dry baker's yeast suspended in tap water was used as the feed solution. Feed was pumped from the feed tank into the filtration module in the cross-flow mode at the selected applied pressure and feed flow velocity. Both retentate and permeate were recirculated to the feed tank. The permeate flux, J, was calculated by measuring the volume of the permeate overflowing from the filtration module every two minutes until it reached the steady-state value which was termed as there was no change of the permeate volume with time for twenty minutes. Permeate was sampled every ten minutes in order to measure the concentration by cell counting method as explained in section 4.6.

After permeate flux reached the steady-state value, the feed of yeast suspension was replaced by demineralized water so that the total permeation resistance, R_t, was measured. Then, the cake formation over the membrane surface was removed by a sponge and the filtration of demineralized water was carried out again in order to measure the resistance of plugging in the pore, R_p.

After each operation, the ceramic membrane was cleaned by being immersed in 0.5 N of NaOH solution for 1.5 to 2 hours in order to remove the particles trapped inside the pore of the membrane. The regenerated membrane should have the same water flux at the same condition as that before the operation otherwise the cleaning process would be repeated.

In case of ultrasonic microfiltration, ultrasound was continuously applied to the system since the beginning of operation at frequency and intensity of 23.8 kHz and 2.2 W/cm^2, respectively.

Table 4-1: Operating conditions of microfiltration system for the study of the effects of pressure, feed flow velocity and feed concentration on permeation performance

feed concentration (g/l)	feed flow velocity (m/s)	applied pressure (kPa)	ultrasonic system
5	0.29	11.27, 16.66, 26.46, 36.26, 46.06	off
5	0.29	11.27, 16.66, 26.46, 36.26, 46.06	on
5	0.02, 0.17, 0.29, 0.48	16.66	off
5	0.02, 0.17, 0.29, 0.48	16.66	on
5, 10, 20	0.29	26.46	off
5, 10, 20	0.29	26.46	on

4.3.1 Study of the effect of the sound frequency

The purpose of this experiment was to study the effect of the sound frequency on permeate flux. Sound frequency was varied as 19.8, 21.5, 23.8 and 30.0

kHz, respectively whereas the input power was constantly applied at 25 W. Applied pressure and feed flow velocity were kept constant at 26.46 kPa and 0.29 m/s, respectively.

4.3.2 Study of the effect of the sound intensity

The objective of this experiment was to study the effect of the sound intensity on the permeation performance. Sound intensity was varied as 0.91, 1.77, 2.19, 2.68 and 3.53 W/cm^2. Applied pressure and feed flow velocity were held constant at 26.46 kPa and 0.29 m/s, respectively.

4.3.3 Study of the effect of the irradiation time

The objectives of this experiment were to obtain effect of the irradiation time on the permeate flux.

Applied pressure and feed flow velocity were controlled at 26.46 kPa and 0.29 m/s, respectively. Ultrasound was irradiated to the system for 4 minutes at different time, i.e. at 2^{nd}, 6^{th}, 10^{th} and 20^{th} minute of filtration. Input power was controlled at 25 W. Permeate volume was measured every 15 seconds.

Besides the previous experiments, effects of the ultrasonic irradiation on the membrane and the yeast were also studied.

4.4 Study of the effect of the ultrasonic irradiation on the membrane

4.4.1 Scanning Electron Microscope (SEM) test

The analyzing of Scanning Electron Microscope (SEM) was employed to investigate the change of the pore size and the inside structure of those membranes.

SEM photographs were taken from the samples of ceramic membrane before being used (after being reduced the thickness) and those after being filtered together with ultrasonic irradiation for some period of time. Selected positions to be photographed were at the position directly irradiated in the side view (cross-section).

4.4.2 Mercury Porosimetry

Measurement of the membrane pore size was also studied using the method of Mercury Intrusion Test. The membranes before and after being sonicated were taken to measure their pore sizes.

4.5 Study of the effect of the ultrasonic irradiation on the yeast

The purpose of this experiment was to study the effect of the ultrasonic irradiation on the yeast. Three experiments were carried out to study the change of the cells' morphology by the observation in the microscope, the particle size distribution using the Pore Size Analyzer and the growth capability of the sonicated cells.

4.5.1 Microscope observation

The filtration was taken at the power of 40 W, which was the highest input power given by the made transducers. Yeast broth in feed tank was sampling to analyze the change in morphology in microscope observation.

4.5.2 Particle Size Analyzer

Three samples, illustrated in Table 4-2, were taken to examine if there was any change in particle size distribution of the yeast cells during the process of microfiltration with ultrasonic in our experiments.

Table 4-2: Samples taken to study the particle size distribution of yeast cells

Sample No.	Sample ID*	pump	acoustic intensity (W/cm^2)	temperature (°C)
1	Sample11	off	0	30
2	Sample8	off	3.6	30
3	Sample4	on	0	30

Note: * = Sample ID represents in the experimental data shown in Appendix C

- All samples were irradiated using ultrasonic probe without the microfiltration process.

These samples were taken at 30th, 60th, 90th minute to measure the particle size distribution using the Particle Analyzer.

4.5.3 Cultivation in agar-based medium

The experiments were divided into two parts to investigate the effect of filtration coupling with ultrasound and the effect of ultrasound only on the yeast's cultivation capability.

In the first part, yeast broth in the feed tank after being filtrated through the module under ultrasonic irradiation of intensity 3.6 W/cm^2 for 30, 60 and 90 minutes were sampled to be cultivated in agar-based culture medium to examine the change in cultivation capability after being filtration coupling with ultrasound.

In the second part, in order to investigate the effect of ultrasonic irradiation on the growth capability, five samples, illustrated in Table 4-3, were chosen. Since the use of high ultrasound generally causes the higher temperature of the system, the experiments were thus carried out to investigate the effect of cavitation and temperature separately.

Table 4-3: Samples taken to study the cultivation capability of yeast cells

Sample No.	acoustic intensity (W/cm^2)	temperature of yeast suspension (°C)
1	0	30
2	3.6	30
3	100	30
4	100	73
5	0	73

Each sample was taken for 1 cm^3, and then was diluted in 0.86% saline solution to various dilution rates (10^{-1} to 10^{-7}). Each dilution was taken for 0.1 cm^3 to cultivate in the agar-based medium. The observation of numbers of cells that could be cultivated was investigated in the Petri dish which contained 30-300 colonies.

4.6 Cell concentration analysis

Cell concentration could be measured using the measurement of cell number. Feed and permeate were sampled to measure the number of cells using a microscope by counting cells placed in a hemacytometer which has a special square grid marked on the surface of the glass slide. A ridge on each side of the grid holds a cover slid off the grid by a known distance so that the volume of a square is precisely known. A sample of cell suspension to be counted is allowed to flow under the cover slip and to fill the counting chamber. Then the number of cells per unit area of grid can be counted under the microscope. Very dense concentration can be counted if they are diluted appropriately [10]. Each concentration was repeatedly measured three times.