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A Study of Rotational Ultrafiltration System for Fructose Recovery from Glucose Fermentation Process

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Abstract

The technical challenge of recovery fructose from fermentation broth is high viscosity of fermentation liquid and high content of nano-size particles. In this study, an innovative rotational circular-plate ultrafiltration membrane filter with a scraping device was employed to purify fructose and remove fouling cake attached to the membrane surface, achieving fructose filtrate flux of above 40 L/m^2/h. By the Darcy's Law, the operation parameters for optimum flux such as temperature, pressure, aeration rate could be found. The optimal fructose recovery from fermentation broth was over 90%. The permeate of ultrafiltration membrane could meet the requirements for commercial fructose in terms of chromaticity, turbidity and sugar content. The traditional complex filtration process can be replaced by the novel ultrafiltration device which has an advantage of waste reduction.

Keywords: Rotational ultrafiltration; Fructose; Flux, Fermentation broth; Sugar content

Highlight

The separation efficiency of novel ultrafiltration system was evaluated.

Fructose could be physically separated from fermentation broths with high viscosity.

The optimum recovery ratio of fructose from fermentation broths was over 90%.

Introduction

Fructose is a natural six-carbon sugar presenting in fruit, vegetables and honey. It has high sweetness, but is relatively expensive. However, with advancements in the food industry, fructose syrup has replaced natural fructose. Fructose syrup is obtained by refining corn with enzyme treatments and converting corn sugar into fructose syrup. With its low cost and ease of combining with various foods, many food and beverage production processes now use fructose syrup for flavoring.

Fructose is typically created by passing purified glucose through a column of glucose isomerase, causing a portion of the glucose to undergo isomerization into fructose, and resulting in syrup composed mainly of glucose and fructose. This syrup is then passed through activated carbon and ionic exchange resin for purification, and finally condensed into a colorless fructose syrup product. For the vital process of removing fermented impurities and eliminating color created during the production process, Oliver filters coated with diatomaceous earth are often used in the past, with activated charcoal combined in a plate and frame filter press to further separate and remove any fermentation agents, fermentation impurities and activated charcoal. After production, the diatomaceous earth and sugar laden activated charcoal are disposed of as regular waste.

In this study, a rotary ultrafiltration system was used instead of conventional filter press to remove byproducts and impurities, namely fermented substances and activated carbon powders, to reduce the amount of wastes, and to meet the food quality standards in the fructose syrup production processes. The increase in the efficiency of fructose purification by using the novel filtration system was discussed. Financial analysis to determine the economic feasibility of the membrane filtration system will also be performed to verify that the result of this study is practical.

Literature Review

Fructose ($C_6H_{12}O_6$) is a simple sugar monomer [1-3]. It is readily soluble in water, and can be produced from starch hydrolysis, which includes processing the starch through liquefaction and saccharification, yielding glucose syrup. The glucose syrup is then treated to isomerase to cause isomerization of glucose into fructose. A filtration system, containing an Oliver filter coated with diatomaceous earth and a filter press, was often used for removing fermentation agents and impurities and activated charcoal. Those important production steps are illustrated in Figure 1.

One key feature of this process is culturing Streptomyces albus to produce isomerase [4]. The actinomycete can directly catalyze glucose, allowing 50% concentration glucose into the isomerization reaction vessel. Mixing at pH 6.8~7.2 and 65~70°C for about 70 hours yields syrup consisting of 40% glucose and 35% fructose and 75% solid matters. Not only is the sweetness of this product high, the overhead costs are much lower than that of granulated sugar when corn starch is used.

In the fructose production process described above, filtration and color adjustment are very important elements in the downstream of the process, affecting the purity of the final product and its appearance. Current traditional production facilities use vacuum filters, named Oliver filters, which utilize negative pressure to suck fermentation broths through a packed bed of diatomaceous earth [5]. Because the apertures in diatomaceous earth beds are always larger than 1μm, it is not easy to improve filtration efficiency in this phase. As a result, an additional filter press is required for achieving the requirements of fructose purity. However, the yielded filtrate is slightly yellowish in

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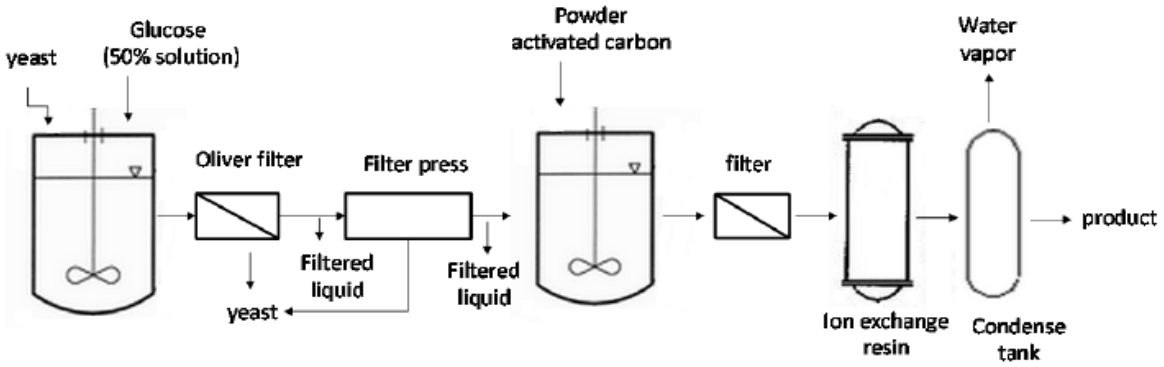


Figure 1: Flowchart illustrating the process of using microbial enzymes to convert glucose into fructose through isomerization.

color. To have products acceptable to consumers requires additional steps for adjusting color. This process mostly involves mixing powdered activated carbon to remove color, and then further refining through an ion exchange resin column [6].

In recent years, rapid advances in the membrane industry have also been applied to process and break down starch and other polymer matrices [7]. Using an ultrafilter reactor loaded with an enzyme (e.g., α-amylase) [4] and continuously-fed starch solution, a steady supply of glucose can be obtained. This is because while enzymes (proteins), starches and polymers are too large to pass through the ultrafilter membrane, smaller molecules such as glucose can cross. This not only greatly improves product purity, but also eliminates bacteria contamination. Thus, this glucose filtrate can be directly used for production in glucose products, isomerization into fructose, or as fermentation material.

Ultrafilter is easy to operate, and always has clear cut-off according to molecular weights. Simply changing the membrane pore diameter, temperature, duration and other conditions of the substrate can control the type of product yielded in the resulting filtrate [7]. Removing fermentation yeast and residues by filtration is essential in fructose manufacturing. Lee and Rose [8] and Lewandowicz et al. [9] used 0.2 μm film membrane distillation to separate and remove yeast, indicating that the size of yeast should be greater than 0.2 μm. Other studies by Sung et al. [10] and Ino and Hirano [11] used 0.1 μm and 0.45 μm pore size ceramic and PVDF membrane to separate fermented residue. Youn et al. [12] used 0.01 μm pore size membranes with molecular weight cut-off greater than 30,000 to filter reconstituted apple juice, yielding a filtrate containing 99% sugar, showing that fructose molecules are smaller than 10 nm. This study will use a rotating flatbed PVDF ultra filter membrane module instead of the traditional diatomaceous earth filter filtration process to shorten the fructose production process and reduce the diatomaceous earth filtration and waste disposal costs.

Materials and Methods

This experiment simulates fructose production process, and will directly connect the fermenter through the solid-liquid separation module array, adding fructose fermentation broth and powdered activated carbon to initiate fructose purification. This experiment can be roughly divided into two main phases.

Prepare at least 100 L of fructose fermentation broth, as well as 105 g of activated carbon to mix into the fermentation broth to functionally simulate the color removal process. First, heat the test liquid to 50-60°C and inject into the experimental membrane unit in batch mode. Record changes in effuse through the rotational ultrafiltration membrane while adjusting the different operating conditions including temperature (10~50°C), pressure (-20 kPa~-60 kPa), aeration (0~8 m^3/min) and fixed membrane rotational frequencies (12 revs/min). Analyze the resulting filtrate for turbidity, apparent color, sweetness, and viscosity to confirm compliance with fructose food product quality requirement.

Turbidity: Standard Methods for the Examination of Water and Wastewater, 20th ed. Method 2130B.

Apparent color: Standard Methods for the Examination of Water and Wastewater, 20th ed., Method 2120A and B.

Sweetness: The high fructose corn syrup (HFCS) is measured and expressed in Brix degree (°). Sugar concentration can be measured with a refractometer that is capable of converting the measured experimental data automatically into Brix degrees.

Viscosity: It was measured using Rheometer (type MCR51), which was created by the Anton Paar Company (Germany). If the fermentation fluid is Newtonian fluids, then the viscosity will be constant.

Analyze pre-processing and post-processing broth samples for particle size, electron microscopy.

Particle size: It was measured using Malvern MRK528-01 Zetasizer Nano System. The suspension temperature was set at 20°C and continuously stirred for 0.5 h to reach a steady state.

Electron microscopy: This system combined the 900 times slide and power image analysis system to measure the particle size.

Because this test mainly focuses on the efficiency of using membrane for removing impurities from fructose fermentation broth, analyzing the effect of important contributing factors can be analyzed through Darcy's Law and equations using experimental data.

$J=\Delta P/\mu R_m$

J: filter flux

ΔP: transmembrane pressure

μ: viscosity of filtered liquid

R_m: resistance coefficient of membrane and fouling

This experiment uses a rotational ultrafiltration membrane device (Figure 2) which was the product of New Century Membrane Co., LTD (Taiwan)., including a filter tank and circular-plate ultrafiltration modules installed in the film slot (PVDF, hydrophilic material, average

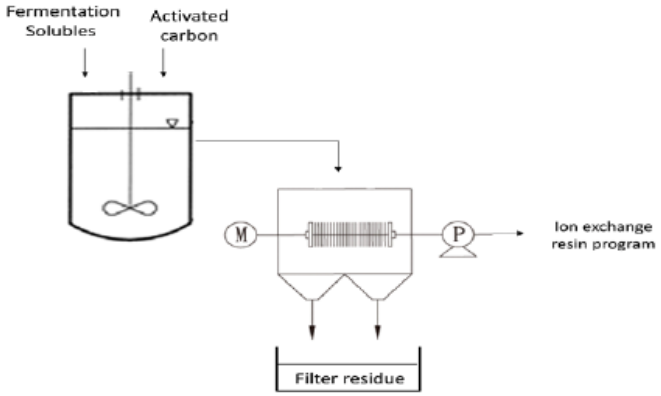


Figure 2: Schematic of the filtration system for fructose production.

pore diameter 30 nm, total membrane area 0.7 m^2). The ultrafiltration module is connected to a rotary drive mechanism and pump. When the pump is activated, negative pressure draws the fermentation broth through the membrane filters, yielding liquid fructose for processing towards the final quality requirements. The membrane filter assembly includes a guide assembly (Figure 3), in which the plastic guide rod is sandwiched between two filtration membranes, and remains in contact with the film. This allows scrape removal of accumulated material along the filtration membranes, extending the filtration membrane's optimal operation lifespan.

Results

Utilizing rotational ultrafiltration membrane device successfully separated large impurities and activated carbon from the fructose fermentation broth. Figures 4 and 5 display the particle sizes before and after filtration. Photographs of the broth under the electron microscope before and after filtration (Figure 6) also indicate that the particle sizes were drastically different.

Operating temperature directly affects the slope of the shear stress vs. shear rate. Figures 7 and 8 portray the linear relationship between shear stress and shear rate at temperatures 50°C and 60°C. The slope indicates broth viscosity, suggesting that viscosity is lower at 60°C than at 50°C.

As shown in Table 1, aeration increases filtration flux by at least 10% compared to non-aerated conditions but under no rotation. Table 2 shows the effect of aeration intensity on the filtration flux under the rotation rate of 12 revs/min. Although the aeration creates a net increase in flux compared to non-aerated conditions, aeration intensity and filtration flux do not necessarily have a positive correlation with each other. Considering operation costs, the process with the most stable filtrate flux and lowest energy consumption was chosen.

Figure 8 illustrates film throughput and operating temperature have a direct relationship; a higher operating temperature results in greater flux. Figure 8 also displays a direct relationship between transmembrane pressure and filtration flux; a higher operating pressure results in greater flux over 60 L/m^2/hr.

The flux over time under operation conditions is shown in Figure 9. Even though aeration mitigates the membrane fouling, backwashing of the membrane is still required to maintain filtration flux. Over time, the backwash time decreases. The system in this experiment should have a full clean time to recover the membranes, with the optimal operation period between cleanings as 3 hours to keep the production rate.

From the sampling and analysis of the filtrate, color chromaticity analysis of the filtrate demonstrated that aeration was beneficial for both filtrate color and turbidity. The Table 3 shows the optimum aeration of 4 m^3/min yielded the best color.

The refractive index results are listed in Table 4, with 1.3288 representing pure water. The fermentation broth refractive indices

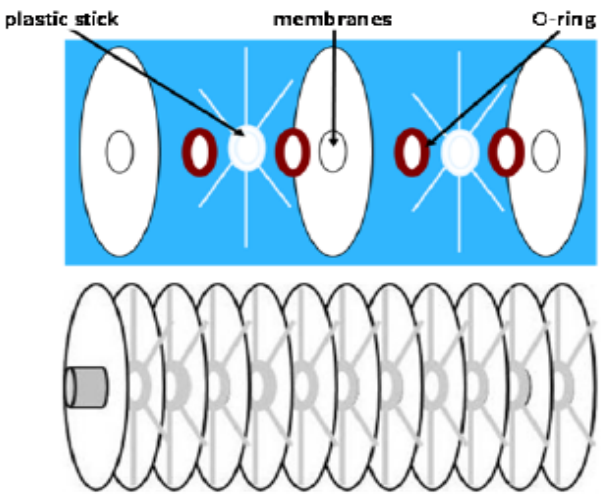


Figure 3: Interior schematic of the ultrafiltration membrane module.

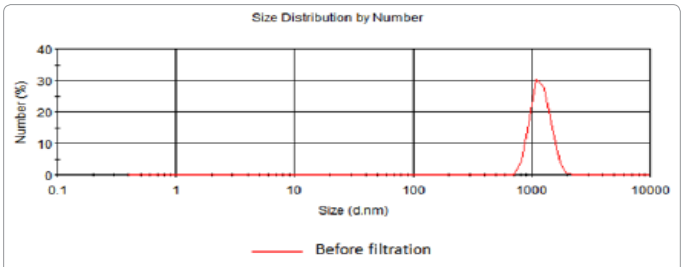


Figure 4: Particle size distribution before filtration.

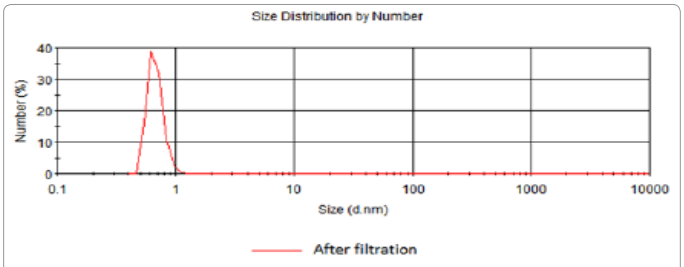
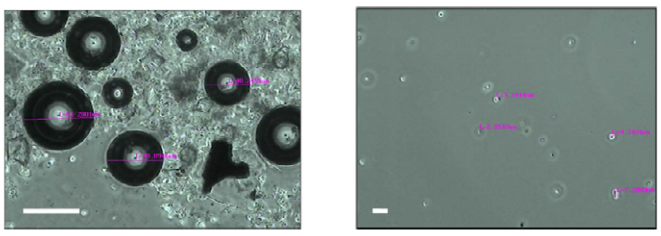


Figure 5: Particle size distribution after filtration.



Re: bar is 50 μm Re: bar is 10 μm

Figure 6: Electron microscopy of unfiltered (left) and filtered (right) substrate.

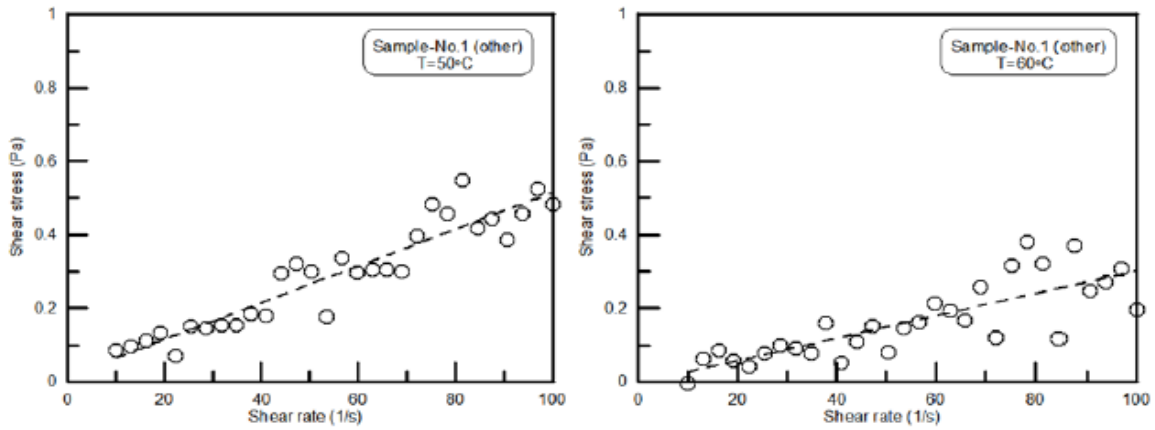


Figure 7: Relationship between fermentation liquid temperature/viscosity.

Time (mins)	No aeration flux (L/m²/hr)	Under aeration flux (4 L/m²/hr)
10	38.9	41.2
30	15.7	20.3
60	8.6	10.5

Table 1: Effect of aeration on the filtration flux during an ultra-filtration under no rotation.

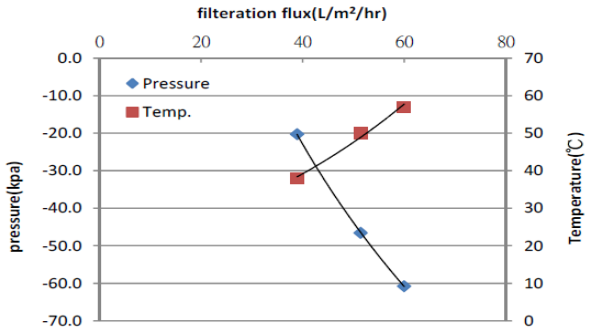


Figure 8: Relationships between membrane filtration system temperature, pressure and flux.

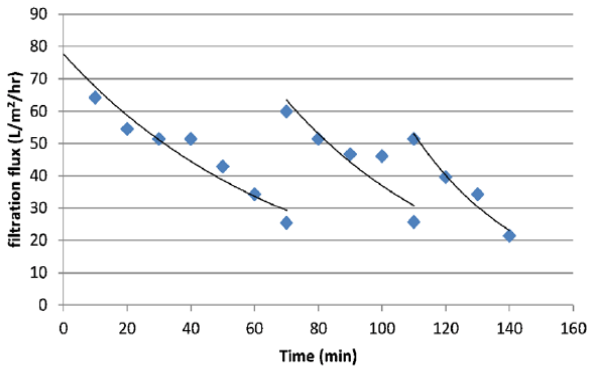


Figure 9: Time course of filtration flux under 4m³/min aeration.

before and after filtration were kept at high values, ranging from 1.3861-1.3900 with an average of 1.3866, showing that there are other substances dissolved in water. Also, the fermentation broth had a sweetness content of about 34.5˚ before filtration. It remained above 33.2˚ after filtration, with a loss rate of less than 4% (Table 5).

Discussion

Particle size analysis

As displayed by Figure 4, before filtration, 105 g of activated carbon was mixed into the fructose fermentation broth and particle size analysis was performed. Particle size before filtration ranged from 0.6 to 5 μm. After filtration, particle size ranged from 0.5 nm to 2 nm, as shown in Figure 5. Because the mean pore size of the ultrafiltration membrane used in this experiment was 30 nm, the yeast could be removed, conforming to Lee [5] and Lewandowicz's [6] removal of 0.2 μm yeast. Larger particles such as powdered activated charcoal (20~40 μm) and other impurities were removed. This was also reflected under electron microscope as those shown in Figure 6. The ultra filter was confirmed to have removed impurities including activated carbon and yeast from the fermentation broth.

Effects of temperature and transmembrane pressure on filtration flux

Bases on the formula, viscosity=(shear stress)/(shear rate), the shear stress is proportional to shear rate with a viscosity constant. Viscosity is the slope of the graph displaying shear stress over shear rate; viscosity decreases as temperature increases when viscosity decreases, the shear stress of the liquid decreases, and shear rate increases, causing filtrate flux to increase. The Darcy's Law indicates that filtrate flux and operating pressure have a directly proportional relationship. The results of this experiment show that temperature directly affects the broth viscosity because of the lower slope of linear curve. Higher viscosity results in higher filtration resistance as well as lower filtration flux. Also, film throughput and operating temperature show a direct relationship. A higher operating temperature results in greater flux. The direct relationship between transmembrane pressure and filtration flux can be displayed by Figure 8; a higher operating pressure results in greater flux over 60 L/m²/hr.

Effect of aeration on the filtration flux

Aeration is used to increase the shear stress on the membrane surface to mitigate particle deposition. Proper amounts of aeration effectively prevent membrane fouling and increase filtrate flux. However, excessive amounts of aeration cause difficulties in increasing flux. This could be that the shear force of aeration bubble through membrane surface disrupted membrane fouling, affected the ability of fouling withdraw, and caused the flux deduction, while maintaining

Time (mins)	Flux under no aeration	flux (L/m²/hr) under 4m³/min aeration	flux (L/m²/hr) under 8m³/min aeration
10	74.1	75.2	73.4
20	60	60.5	60.1
30	46.2	51.4	42.8
60	25.7	34.2	34.2

Table 2: Comparison of 3 levels of aeration on filtration flux.

Time (min)	Flux (L/m²/hr) under no aeration		Flux (L/m²/hr) under 4m³/min aeration		Flux (L/m²/hr) under 8m³/min aeration	
	Chromaticity	Turbidity (NTU)	Chromaticity	Turbidity (NTU)	Chromaticity	Turbidity (NTU)
10	330	144.1	235	0.3	207	ND
20	227	2.2	119	ND	197	ND
30	200	2.9	111	ND	189	ND
60	194	1.2	103	ND	173	ND

Table 3: Color chromaticity and turbidity under three levels of aeration.

Item	Refractive Index
Water	1.3288
Fermentation Broth Before Filtration (1)	1.3866
Fermentation Broth After Filtration (1)	1.3861
Fermentation Broth Before Filtration (2)	1.39
Fermentation Broth After Filtration (2)	1.3889

Table 4: Refractive indices of fermentation broth before and after filtration.

Time (mins)	Flux under no aeration	Flux (l/m²/hr) under 4m³/min aeration	Flux (L/m²/hr) under 8m³/min aeration
30	34.5	33.5	33.9
60	33.5	33.6	33.9
90	33.4	33.8	33.5
120	32.7	33.9	33.6
150	33.5	33.9	33.3
180	33.2	33.3	33.2

Table 5: Sweetness (Brix°) of fermentation broth before and after filtration.

flux above 30 L/m²/hr. The optimal amount of aeration is shown to be about 4m³/min in this experiment.

Relationships between film flux, operating pressure, and operating temperature

Increases in operating temperature decrease the fructose broth viscosity, thereby reducing the resistance of the broth travelling through the film and increasing film flux. Similarly, increases in operating pressure also increase film flux, conforming to Darcy's Law. Therefore, temperature and pressure are important factors that need to be considered because they affect the stability of production. The optimal operating conditions of this experiment exist near the intersection in Figure 8. Operating temperature should be at least 42°C, pressure should be about -30 kpa, and optimum filtrate flux is 40 L/m²/hr.

Description of film clean process

In this experiment, permeate flux is maintained through aeration and backwash. Complete cleaning is required to maintain the film after 3 hours of operation. Since the fructose broth contains living organisms, there may be problems with biofouling. Therefore, this experiment used plastic scraping devices to effectively remove biofouling materials. The film is further cleaned through backwashing. Backwash entails rinsing with 30 seconds of clean water; the fouling materials would be removed from the membrane surface because of the membrane inflation. The full clean process should also include 6 extra minutes of aeration. This process restores the flux to almost 100%.

Filtrate color and turbidity analysis

The optimum aeration rate in this experiment was determined through assessing the color and turbidity of the filtrate under various aeration conditions. Results suggest that aeration of 4 m³/min yielded the best color. Even though turbidity was lowest at 8 m³/min aeration, there was much less color removal. This was because too much aeration causes materials on the film surface to damage the film, allowing smaller particles to pass through the film. The resulting product would therefore be low in quality and have higher turbidity. Based on the results of this experiment, the optimal aeration was selected as 4 m³/min.

Overall quality analysis

Product quality is usually based on sugar content. Sugar content was determined from the results of the refractive index experiment. Results of this experiment indicate that filtration caused 4% decrease in sugar content. The decrease in sugar content was because the fermentation broth was filtered using physical means rather than chemical ones, and 4% of the sugar was absorbed by the materials that were filtered out. This also proves that in terms of fructose content, the filtered fermentation broth meets the requirements for fructose food products.

Economic comparison of fructose filtration production processes

This study simplified the process of fructose filtration using an ultrafiltration instead of traditional diatomaceous earth filtration, filter

press and additional color removal filtration, and performed both activated carbon and impurity removal in one unit operation. This has advantages of time saving, easy operation and decreasing initial capital investment that less than half price of traditional machine. Also, the traditional diatomaceous earth filtration system has a diatomaceous earth cost of US$ 130,000 per year and produces extra diatomaceous earth waste that needs disposal treatment. As a result, the membrane filtration process is better than conventional process both technically and economically.

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