EFFECTS OF TRANSMEMBRANE PRESSURE ON THE PERFORMANCE OF FLAT SHEET MEMBRANE ON MILK PROTEIN SEPARATION

KASMAN BIN MOKTAR

A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering (Biotechnology)

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

ii

I declare that this thesis entitled "Effect of Transmembrane Pressure on the performance of Flat Sheet Membrane on milk protein separation" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :....

Name : Kasman Bin Moktar

Date : May 2009

Special Dedication to my family members,
my friends, my fellow colleague and all faculty members
For all your care, support and believe in me.

ACKNOWLEDGEMENT

I would like to forward my appreciation to my thesis supervisor Ms Shalyda MD Shaarani @MD Nawi and panels Ms.Nasratun Masngut and Ms Asmida Ideris for their guidance and support. I am also thankful for Ms.Sureena Abdullah for her advises and opinions given throughout this study.

I am very thankful to University Malaysia Pahang (UMP) for providing good facilities in the campus. To all the staff in Faculty of Chemical & Natural Resources Engineering, a very big thanks you to all.

My sincere appreciation also extends to all my fellow colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. Thank you for the time sacrificed to accompany me. And last but not least, I am grateful to all my family members.

ABSTRACT

This research is to study the effect of transmembrane pressure on cross flow filtration on protein milk separation. The different transmembrane pressure will be effect the performance of cross flow filtration. By this research, the optimum operation condition of the cross flow filtration will get especially when comes to protein milk separation. In other word, the suitable parameter will be known in order to separate protein milk. Protein milk can be separated from other composition in the skim milk using cross flow filtration. Before using filtration, skim milk should be centrifuged to remove fat and undesired composition to reduce membrane faultiness or membrane blockage. Protein compositions in both permeate and retentate analyzed using Lowry method. From the result, the optimum operation condition for milk protein separation is at TMP 25 psi and flux recorded is 94 ml/ (m².min) and 78 ml/ (m².min). This is experiment using 10K membrane cassette in batch mode.

ABSTRAK

Kajian ini adalah untuk mengetahui kesan tekanan transmembran pada prestasi penyaringan aliran bersilang terutamanya apabila ingin memisahkan protein susu. Pelbagai nilai tekanan transmembrane akan mempengaruhi prestasi penyaringan aliran bersilang. Dengan adanya kajian ini, nilai keadaan optima operasi dapat diketahui terutamanya yang berkaitan dengan pemisahan protein susu daripada susu sejat. Protein susu dapat dipisahkan daripada lain – lain composisi mengunakan kaedah penyaringan membrane. Sebelum menggunakan kaedah penyaringan, susu sejat hendaklah dilakukan pengemparan untuk memisahkan lemak dan komponen lain yang tidak diingini. Selain itu, ia dapat mengurangkan keadaan kegagalan atau membrane tersumbat. Kandungan protein yang melalui membrane dan yang tidak melalui membrane di analisa mengunakan kaedah Lowry. mengikut kajian yang dijalankan, pemisahan protein susu adalah terbaik pada TMP 25 yang mencatatkan nilai flux 94 ml/ (m².min) dan 87 ml/ (m².min). kajian ini mengunakan 10K membrane kaset dalam keadaan ultrafiltration.

TABLE OF CONTENTS

CHAPTER		TITLE	PAGE
	TITI	LE PAGE	i
	DEC	CLARATION	ii
	DED	DICATION	iii
	ACK	KNOWLEDGEMENT	iv
	ABS	TRACT	v
	ABS	TRAK	vi
	TAB	BLE OF CONTENTS	vii
	LIST	Γ OF TABLES	ix
	LIST	Γ OF FIGURES	xi
	LIST	Γ OF ABBREVIATIONS	xii
	LIST	Γ OF SYMBOLS	xiii
	LIST	Γ OF APPENDICES	xiv
1	INT	RODUCTION	1
	1.1	Milk Protein	1
	1.2	Problem statement	2
	1.3	Objective	3
	1.4	Scope of study	3
2	LITI	ERATURE REVIEW	4
	2.1	Milk protein component	4
	2.2	Membrane protein separation	7
		2.2.1 Ultrafiltration	8
		2.2.2 Microfiltration	9
	2.3	Mode of operation	9

		2.3.1 Cross flow filtration	10
	2.4	Driving force	12
	2.5	Membrane fouling and cleaning	12
	2.6	Centrifugation	14
3	ME	ГНОДОГОСУ	16
	3.1	Water flux	16
	3.2	Operating parameters	18
	3.3	Membrane cassette selection	19
	3.4	Protein concentration	20
	3.5	Protein analysis	21
		3.5.1 Reagent preparation	21
		3.5.2 Protein determination	21
	3.6	Membrane cleaning	22
	3.7	Standard curve	22
	3.8	Process summary of filtration	23
4	RES	SULTS AND DISCUSSION	25
	4.1	Introduction	25
	4.2	Transmembrane pressure and Flux	26
	4.3	Transmembrane pressure and concentration	28
	4.4	Separation behavior of cross flow filtration	32
	4.5	Difference separation between permeate and	
		retentate with varies TMP	33
5	CON	NCLUSION AND RECOMMENDATION	34
	5.1	Conclusion	34
	5.2	Recommendations	35
	REF	FRENCES	36
	APP	ENDICES	38

LIST OF TABLES

TABLE NO.	TITLE	PAGE
Table 1	Biochemical characteristics of milk proteins	5
Table 2	Molecular size of milk component	7

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
Figure 1	Milk component diagram	2
Figure 2	Size distribution of major milk components	6
Figure 2.1	Spectrum diagram of membrane	8
Figure 2.2	Dead end filtration Vs Cross flow filtration	10
Figure 2.3	Cross flow filtration	11
Figure 2.4	Skim milk centrifugation	14
Figure 3.0	Kvick Lab Cross Flow filtration	17
Figure 3.1	Kvick Lab Cross Flow system flow	17
Figure 3.2	Membrane cassettes	20
Figure 3.3	Standard curve	23
Figure 3.4	Process summaries for protein milk separation	24
Figure 4.0	Graph of flux and varies of TMP	26
Figure 4.1	Permeate protein milk flux at different TMP	27
Figure 4.2	Protein concentration in permeate with	
	various TMP	28
Figure 4.3	Protein concentrations in retentate with	
	various TMP	29
Figure 4.4	Total protein concentration in permeate	
	with various TMP	30
Figure 4.5	Total protein concentration at retentate	
	with different TMP	31
Figure 4.6	Separation behavior at permeate at the best	
	TMP protein separation	32

Figure 4.7 Fractions of protein concentration between permeate and retentate with different TMP 33

LIST OF ABBREVIATIONS

CFF Cross flow filtration

Da Dalton

FDA's Food & Drug Administration

MF Microfiltration
MW Molecular weight

MWCO Molecular Weight Cut Off

NF Nano filtration

NFF Normal Flow Filtration

OD Optical density
PV Permeate volume
RO Reverse Osmosis
RV Retentate volume

TMP Transmembrane Pressure

UF Ultrafiltration

UTMP Uniform Transmembran pressure

Vcf Cross flow velocity

LIST OF SYMBOLS

 ΔP - Differnce of pressure

 ΔPTM - Difference of transmembrane pressure

JC - Cleaning flux

JT - Total flux JW - Water flux

L - Liter

 α - Alpha

 β - Beta

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Standard curve and water flux	38
В	Operating parameter and total protein	
	concentration for each TMP	39
C	Protein concentration profile at	
	various TMP	40
D	Spectrum membrane diagram and	
	properties of milk component	45
Е	Cross flow filtration equipments	48

CHAPTER 1

INTRODUCTION

1.1 Milk protein

The word protein can be defined as any group of complex organic compounds, consisting essentially of combinations of amino acids in peptide linkages, which contain carbon, hydrogen, oxygen, nitrogen and usually sulfur. Widely distributed in plants and animals, proteins are principal constituent of the protoplasm of all cells and are essential to life. Protein is derived from Greek word meaning 'first' or 'primary', because of the fundamental role of proteins in sustaining life (Morris, 1992). Milk is an emulsion of fat globules, and a suspension of casein micelles (casein, calcium, phosphorous), all of which are suspended in an aqueous phase that contains solublized lactose, whey proteins, and some minerals. Leukocytes in milk are part of the suspended phase. This can be shown clearly as in Figure 1.

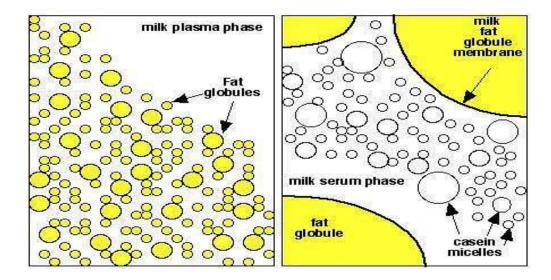


Figure 1: Milk component diagram

Application of the membrane technology in processing milk and dairy product have been shown successfully since 1970 (Y.Poulit, 2008) and this widely used nowadays. Membrane filtrations commonly use to concentrate milk protein from skim milk. The membrane separation is base on molecular cut off (MWCO) of the targeted product. In protein separation technology, membrane technology is more popular than other protein separation method.

1.2 Problem statement

The identification of the problem is to determine operating condition to investigate the effectiveness of cross flow filtration in protein milk separation through identification of the optimum parameter condition. The optimum parameters are varies with other operation condition. Transmembrane pressure one of parameters that influence the performance of cross flow filtration and need to studied and identified. The way to identify which transmembrane pressure is required in protein milk separation is by conducting an experiment. By using different transmembrane pressure, the correct value of transmembrane pressure may be can be

determined. Beside that other parameter such as velocity and pressure in both permeate and retentate can be identified.

1.3 Objectives

The objective of this study is to optimize the usage of cross flow filtration system in milk protein separation and to determine the effects of transmembrane pressure (TMP) on cross flow filtration performance.

1.4 Scope of study

The objective can be achieved by varying the transmembrane pressure until get the optimum separation on milk protein separation. This study will be prepared by using cross flow flat sheet membrane. The other parameter concerned on this experiment is cross flow velocity and pressure both permeate and retentate stream by using 10 K membrane cassette.

CHAPTER 2

LITERATURE REVIEW

2.1 Milk protein component

The total protein component of milk is composed of numerous specific proteins. The primary group of milk proteins is the caseins. There are 3 or 4 caseins in the milk of most species, the different caseins are distinct molecules but are similar in structure. All other proteins found in milk are grouped together under the name of whey proteins. The major whey proteins in cow milk are β-lactoglobulin and α -lactalbumin. The major milk proteins, including the caseins, β -lactoglobulin and α-lactalbumin, are synthesized in the mammary epithelial cells and are only produced by the mammary gland. The immunoglobulin and serum albumin in milk are not synthesized by the epithelial cells. Instead, they are absorbed from the blood (both serum albumin and the imunoglobulins). An exception to this is that a limited amount of immunoglobulin is synthesized by lymphocytes which reside in the mammary tissue (called plasma cells). These latter cells provide the mammary gland with local immunity. Caseins have an appropriate amino acid composition that is important for growth and development of the nursing young. This high quality protein in cow milk is one of the key reasons why milk is such an important human food.

Caseins are highly digestible in the intestine and are a high quality source of amino acids. Most whey proteins are relatively less digestible in the intestine, although all of them are digested to some degree. When substantial whey protein is not digested fully in the intestine, some of the intact protein may stimulate a localized intestinal or a systemic immune response. This is sometimes referred to as milk protein allergy and is most often thought to be caused by \(\beta-lactoglobulin. Properties of protein milk can be seeing in table 1. Casein is the highest percentage protein in skim milk (3% -55%) compare to other protein whey such as alpha-Lactalbumin (2% - 5%).

Table1: Biochemical characteristics of milk proteins (RD Bremel, University of Wisconsin)

Protein	Approx.% of	Isoelectric	Molecular
riotem	skim milk protein	point	weight
alpha-Casein	45-55	4.1	23,000
kappa-Casein	8-15	4.1	19,000
beta-Casein	25-35	4.5	24,000
gamma-Casein	3-7	5.8-6.0	
alpha-Lactalbumin	2-5	5.1	14,437
beta-Lactoglobulin	7-12	5.3	18,000
Blood serum albumin	0.7-1.3	4.7	68,000
Lactoferrin	0.2-0.8		87,000
Immunoglobulins:			
IgG1	1-2		160,000
IgG2	0.2-0.5		160,000
IgM	0.1-0.2		~1,000,000
IgA	0.05-0.10		~400,000
Proteose peptone fraction	2-6	3.3-3.7	4,100 to
Trocose peptone fraction	2-0	5.5-5.1	200,000

Major size distribution of milk component (Cheryan and Alvarez, 1995) can be interpreted as in figure 2. Most components such as whey proteins, casein micelles, fat and bacteria can be see clearly the size of each component. Milk protein is in range 1 to 500 while fat and bacteria are having size greater than 500.

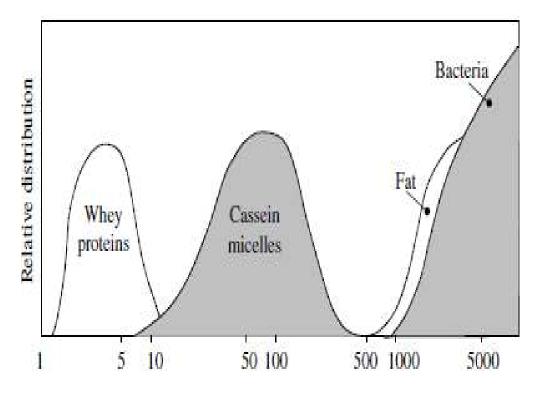


Figure 2: Size distribution of major milk components

Another component and size distribution can be see in Table 2. Both chloride ion and calcium ion have a same diameter (0.4nm) and water 0.3nm in diameter. The protein (casein) has diameter size 25-130 nm while fat is 2000-10000nm. In protein milk separation (filtration) it is necessary to remove fat first before undergoes filtration process. This is due to minimizing the layer forming at the membrane surface during filtration.

Table 2: Molecular size of milk component

Component	Molecular weight	Diameter (nm)
Water	18	0.3
Chloride ion	35	0.4
Calcium ion	40	0.4
Lactose	342	0.8
a-lactalbumin	14500	3
b-lactalbumin	36000	4
Blood serum albumin	69000	5
Casein micelles (milk protein in solution)	107–109	25–130
Fat	-	2000–10000

2.2 Membrane protein separation

Membrane filtration becomes more popular bioseparation application especially among dairy product. The principle of membrane separation is utilized in the dairy industry serve as a different purpose. Membrane filtration separation technique can be classified as Reverse osmosis, Nanofiltration, Ultrafiltration and Microfiltration base on pore size. Reverse osmosis use for dehydration of whey. Nanofiltration usually used when partial desalination of whey. While Ultrafiltration is typically used to concentrate of milk proteins in milk and whey and for protein standardization of milk intended for cheese, youghurt and some other products. For Microfiltration, basically used for reduction of bacteria in skim milk, whey and brine, besides defatting of whey (Goude-dranche, Fauquant, &maubois, 2000) intended for whey protein concentrate and for protein fractionation. RO, NF, UF, and MF cover entire separation domain of milk constituents, from casein micelles to monovalent

ions (Y.Pouliot, 2008). Beside protein separation, UF and MF also used to concentrate juice (B. Sarkar et al, 2008). To separate protein milk, the type of filtration has to determine. Figure 2.1 is spectrum diagram of membrane. By using spectrum diagram, the separation process can be decided.

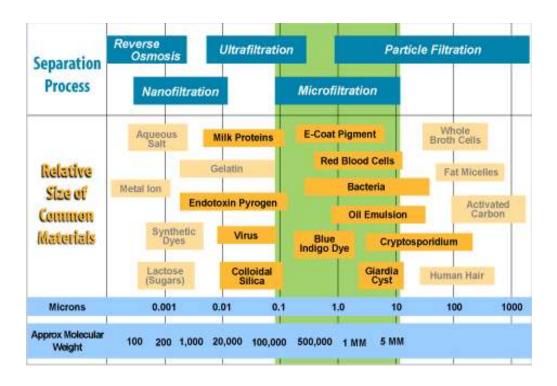


Figure 2.1: Spectrum diagram of membrane

2.2.1 Ultrafiltration (UF)

Ultrafiltration is a separation process using membranes with pore sizes in the range of 0.1 to 0.001 micron as be seeing in Figure 2.1. Typically, ultrafiltration will remove high molecular-weight substances, colloidal materials, and organic and inorganic polymeric molecules. Low molecular-weight organics and ions such as sodium, calcium, magnesium chloride, and sulfate are not removed. Because only high-molecular weight species are removed, the osmotic pressure differential across the membrane surface is negligible. Low applied pressures are therefore sufficient to achieve high flux rates from an ultrafiltration membrane. UF also use in whey treatment to recover cheese manufacturing byproduct (M.S. Yorgun *et al.* 2007).

2.2.2 Microfiltration

Microfiltration is a filtration process which removes contaminants or undesired substances from a fluid (liquid & gas) by passage through a microporous membrane. A typical microfiltration membrane pore size range is 0.1 to 10 micrometres (µm). Microfiltration is not fundamentally different from reverse osmosis, ultrafiltration or nanofiltration, except in terms of the size of the molecules it retains. The use of microfiltration not only standardize of protein content but also the casein to total protein ratio (Johnson & Lucy, 2006). Developed by Professor Sigmondy University of Goettingen, Germany, in 1935, membrane filters were first commercially produced by Sartorius GmbH a few years later. Membrane filters found immediate application in the field of microbiology and in particular in assessment of safe drinking water. Further development of microfilters in the mid 1970's lead by the FDA's requirement for non-fibre releasing filters to be used in the production of injectable solutions. Microporous membranes are used by the microelectronics industry as an integral part of water production. Membrane filters are widely used in biotechnology and food and beverage applications where sterile product is required (Yves Pouliot, 2008).

2.3 Mode of operation

Membrane flow system can be divided by two systems, dead end filtration and cross flow filtration or tangential flow filtration. For many reason, the cross flow filtration is most widely used in dairy product industry due to the advantages of the system. Figure 2.2 shows that the differences of dead end and cross flow filtration. In dead end filtration, clogging layer rapidly increase and need backwash frequently compare to cross flow filtration.

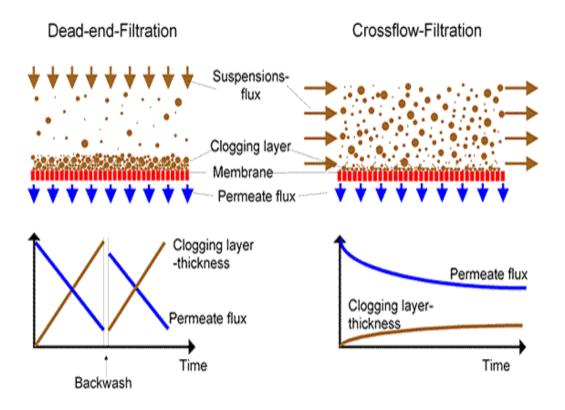


Figure 2.2 Dead end filtration Vs Cross flow filtration

Both normal flow and cross flow technologies purify bioprocess solutions by removing contaminates with a fixed porous medium, yet each format has unique advantages. Generally, normal flow filters (NFF) or dead end filtration are used where clarification and/or bio-burden reduction is desired in relatively low solid streams, for protecting or enhancing downstream operations, or when final polishing is required to achieve sterility. Cross flow filters (CFF) are best suited for higher solids, more viscous feed solutions, and/or where concentration or purification of cells or target species is desired.

2.3.1 Cross flow filtration

In cross-flow filtration, a feed liquor to be filtered flows through an overflow channel, whereby the feed is directed against the surface of the filter element tangentially. The filter splits the feed into a concentrate (retentate) and a filtrate