

UNIVERSITI PUTRA MALAYSIA

RECOVERY OF LOW LACTOSE GOAT'S MILK USING CROSS-FLOW ULTRAFILTRATION MEMBRANE

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

June 2016

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Dedicate to my husband, Faiz, and my parents who have always encouraged me to follow my passion. Thank you for your loving support.

2016, Serdang, Malaysia



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

RECOVERY OF LOW LACTOSE GOAT'S MILK USING CROSS-FLOW ULTRAFILTRATION MEMBRANE

By

NUR SOFUWANI BINTI ZAINUL ABIDIN

June 2016

Chair: Assoc. Prof. Datin Siti Aslina Binti Hussain, PhD Faculty: Engineering

An extensive amount of research has reported on the use of ultrafiltration (UF) membrane, particularly in the improvement of membrane performance efficiency on cow's milk. However, a very limited number of researches reported on using UF for producing low-lactose goat's milk due to inherently low lactose. Nonetheless, goat's milk is still not suitable to be consumed in a large amount by people who are lactose intolerant, especially among Asians, where over 90% of the populations are suffering from lactose intolerance.

Until today, fouling and concentration polarization (CP) on membrane surface in cross-flow hollow fiber UF unit are the major problems in the dairy industry. Discovery on how to overcome the problem is still in a hot debate due to the nature's complex composition in milk. One way to overcome this problem is by evaluating the effects of processing parameters such as trans-membrane pressure (TMP) and feed-flow rate on flux (J), lactose rejection (Ri), concentration factor (CF), and accumulation rate (AR) during the fractionation of lactose.

In terms of lactose fractionation for 5 KDa and 10 KDa UF membranes, the TMPs examined were 0.41, 0.55, and 0.69 bars, while feed flow-rates examined were 0.18, 0.34, 0.54, and 0.74 L/min. 5 KDa membrane shows that feed flow-rate and flux behave in a direct relationship, while an inverse relationship in 10 KDa membrane. Both membranes showed that TMP 0.55 bar exhibit the best flux value without reaching the limiting flux region, but with feed flow rate of 0.74 L/min in 5 KDa, while 0.18 L/min in 10 KDa membrane.

In statistical analysis software (SAS), feed flow-rate of 0.74 L/min was significantly greater (P < 0.05) in 5 KDa, while in 10 KDa membrane, flow-rate of 0.18 L/min gave the best condition (P < 0.05) that required in the lactose fractionation. Lactose rejection percentage (%Ri) is the lowest with 77.71% in 5 KDa membrane while 66.28% in 10 KDa membrane. This can be summarized

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that the best parameters for 5 KDa membrane is at TMP 0.55 bar with feed flowrate of 0.74 L/min, while for 10 KDa membrane is at TMP 0.55 bar with feed flowrate of 0.18 L/min. Due to higher flux value and lowest lactose rejection obtained from low feed flow-rate, 10 KDa UF membrane size was chosen over 5 KDa.

The competitiveness between the concentrated milk obtained from 10 KDa UF membrane in this study and the commercial milk powder were compared in terms of nutrition facts and lactose concentration. The concentrated milk contained 5.63 g per 100 ml lactose concentration, which ranked at the second lowest concentration in the range of 2.81 to 7.91 g per 100 ml, proved that it is similar and comparable in standard as to commercial milk. As a conclusion, a high degree of lactose removal from goat's milk could be achieved by 10 KDa UF membrane in a cross-flow hollow fiber system, which proved that different outcomes between 5 KDa and 10 KDa membranes and feed flow-rate required is closely associated to UF pore size and molecular weight of feed solute particles.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PEMULIHAN SUSU KAMBING RENDAH LAKTOSA MENGGUNAKAN MEMBRAN ULTRATURASAN ALIRAN SILANG

Oleh

NUR SOFUWANI BINTI ZAINUL ABIDIN

Jun 2016

Pengerusi: Prof. Madya Datin Siti Aslina Binti Hussain, PhD Fakulti: Kejuruteraan

Banyak kajian telah dilaporkan mengenai penggunaan membran ultraturasan (UF), terutamanya dalam peningkatan kecekapan prestasi membran pada susu lembu. Walaubagaimanapun penyelidikan yang dilaporkan terhadap pemulihan susu kambing rendah laktosa amat terhad kerana kandungan laktosa yang sememangnya lebih rendah. Walaupun susu kambing mempunyai laktosa yang lebih rendah, ia masih tidak sesuai untuk diambil dalam jumlah yang besar untuk orang-orang yang tidak toleran terhadap laktosa, terutamanya bagi orang-orang Asia di mana lebih 90% daripada populasi ini mengalami intoleransi laktosa.

Sehingga hari ini, kekotoran dan penumpuan polarisasi (CP) pada permukaan membran dalam unit aliran silang serat berongga UF adalah masalah utama dalam industri tenusu dan penemuan mengenai bagaimana untuk mengatasi masalah ini masih dalam perdebatan yang panas kerana sifat komposisi susu yang kompleks. Salah satu cara untuk mengatasi masalah ini adalah dengan menilai kesan parameter pemprosesan seperti tekanan trans-membran (TMP), dan kadar aliran suapan ke atas peratus penolakan laktosa (Ri), faktor kepekatan (CF), dan kadar akumulasi (AR) semasa fraksinasi latosa.

Dalam fraksinasi laktosa, TMP yang diperiksa adalah 0.41, 0.55, and 0.69 bar, manakala kadar aliran suapan yang diperiksa adalah 0.18, 0.34, 0.54, dan 0.74 L/min. Dalam membran 5 KDa, kadar aliran suapan dan fluks bertindak balas dalam hubungan secara langsung, manakala bertindak balas dalam hubungan bertentangan dalam membran 10 KDa Kedua-dua membran menunjukkan bahawa TMP 0.55 bar menghasilkan nilai fluks terbaik tanpa melepasi garisan had fluks, tetapi pada kadar aliran suapan 0.74 L/min untuk 5 KDa, manakala 0.18 L/min untuk 10 KDa.

Menggunakan perisian analisis statistik (SAS), kadar aliran suapan 0.74 L/min adalah jauh lebih besar (P < 0.05) dalam membran 5 KDa, manakala dalam membran 10 KDa, kadar aliran suapan 0.18 L/min memberikan keadaan terbaik



(P < 0.05) yang diperlukan dalam fraksinasi laktosa. Peratus penolakan laktosa (%Ri) adalah yang paling rendah dengan 77.71% dalam membran 5 KDa manakala 66.28% dalam membran 10 KDa. Ini boleh dirumuskan bahawa parameter terbaik untuk membran 5 KDa adalah pada TMP 0.55 bar dengan kadar aliran suapan 0.74 L/min, manakala bagi membran 10 KDa adalah pada TMP 0.55 bar dengan kadar aliran suapan 0.18 L/min. Disebabkan oleh nilai fluks yang lebih tinggi dan peratus penolakan laktosa yang paling rendah diperolehi dari kadar aliran suapan yang rendah, maka membran UF bersaiz 10 KDa dipilih berbanding 5 KDa.

Daya saing di antara susu pekat yang diperoleh dari membran 10 KDa hasil dari kajian dan susu tepung komersial telah dibandingkan dari segi fakta nutrisi dan kepekatan laktosa. Susu pekat mengandungi 5.63 g per 100 ml kepekatan laktosa, yang berkedudukan kedua terendah dalam julat 2.81 ke 7.91 g per 100 ml, membuktikan bahawa susu pekat adalah setaraf dengan susu komersial. Kesimpulannya, penyingkiran laktosa yang tinggi daripada susu kambing boleh dicapai menggunakan membran UF 10 KDa dalam sistem aliran silang serat berongga, membuktikan bahawa keputusan yang berbeza di antara 5 KDa dan 10 KDa dan kadar aliran suapan yang diperlukan berkait rapat dengan saiz liang UF dan berat molekul zarah suapan bahan larut.

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LIST OF ABBREVIATIONS

Analysis of Variance
Concentration Factor
Cross-flow filtration
Concentration polarization
High Performance Liquid Chromatography
Microfiltration
Sodium Hydroxide
Nanofiltration
Normal Flow Filtration
Level of significance
Reverse Osmosis
Statistical analysis system
Transmembrane Pressure
Ultrafiltration

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LIST OF GLOSSARY

Allergy Concentration Factor	A hypersensitivity reaction initiated by specific immunologic mechanisms. The volume reduction achieved by concentration, i.e. the ratio of initial volume of feed to the final volume of
Concentration Polarization	concentrate/retentate The accumulation of retained molecules (gel layer) on the upstream surface of the membrane
Cow's milk protein allergen	A hypersensitivity reaction to cow's milk protein initiated by specific immunologic mechanisms allergy
Cow's milk protein	A non-allergic hypersensitivity to cow's milk protein
Cross-flow filtration	A process where the feed stream flows parallel to the membrane face. Applied pressure causes one portion of the flow stream to pass through the membrane (filtrate) while the remainder (retentate) is recirculated back to the feed reservoir
Cross-flow Rate	The recirculating volumetric flow rate of the feed solution through the cassette assembly. Flow rate is measured at the retentate, and is typically recorded as liters/minute
Feed	The solution to be concentrated or fractionated
Flux	The rate of extraction of permeate measured in litres per square meter of membrane surface area per hour
Food allergy	An adverse health effect arising from a specific immune response that occurs reproducibly on exposure to a given food. Also known as food hypersensitivity
Food intolerance	A non-allergic food hypersensitivity
Gastrointestinal allergy	allergy in which upper gastrointestinal symptoms may occur within minutes and lower gastrointestinal symptoms may occur either immediately or with a delay of up to several hours
Gel Layer	The microscopically thin layer of molecules that forms on the upstream side of the membrane. It causes a xvi

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reduction in the filtrate flow rate and may increase the retention of molecules that would normally cross into the filtrate

Deposition and accumulation of feed components on the membrane surface and/or within the pores of the membrane. Causes an irreversible flux decline during processing

The filtrate, the liquid passing through the membrane

The amount of product (mass or activity) recovered after processing compared to the amount in the starting sample. Usually expressed as a percentage of starting material.

The concentrate, the retained liquid Milk - Fat

Protein, lactose, minerals, vitamins, and enzymes.

Fat + Solids Non -fat

It is the driving force for liquid transport through the ultrafiltration membrane. Calculated as the average pressure applied to the membrane minus any filtrate pressure. In most cases, pressure at filtrate port equals zero The water filtrate flux rate over the TMP for a given membrane Skim milk - Casein micelles

Membrane fouling

Permeate

Product Recovery

Retentate Skim milk (plasma) Solid Non-fat

Total solids Trans-membrane Pressure

Water Permeability

Whey (serum)

LIST OF NOTATIONS

A	Membrane effective area (m ²)		
Cf	Concentration of solute in the feed stream (mg/mL)		
Ср	Concentration of solute in the permeate (mg/mL)		
Cr	Concentration of solute in the retentate stream (mg/mL)		
J	Permeating Flux (L/m ² .hr)		
L	Liter		
Μ	Meter		
R	Lactose recovery or transmission of lactose in		
	permeate		
Ri	Rejection of Lactose or lactose left in the retentate		
t	Time (minute)		
Vp	Permeate volume (L)		

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CHAPTER 1

INTRODUCTION

1.1 Background

In Malaysia, the government has begun its focus on increasing local milk production within the dairy sector due to economic changes among consumers (Wouters, 2011). In fact, researchers reported that more goats are being reared at present in Malaysia compared to cow in year 2003, as to meet the requirements of consumers for high quality, healthy, and a variety of food at reasonable price (Rezai et al., 2011; Liana et al., 2010; Omar, 2010; Arshad et al., 2006). Additionally, many factors, such as education and modernization, have increased consciousness among consumers towards their routine diet (Quah & Tan, 2010; Ishida et al., 2003). These statements, moreover, are supported by the data gathered by FAO 2015, which claimed a significant increase in the total milk production in Malaysia that rose from 0.03 million tons (MT) in 1990 to 0.08 MT in 2013. The increment in the production value of milk in Malaysia was approximately threefold and had been expected to rise throughout the next decade.

Goat's milk is a complex biological fluid that contains proteins, lipids, lactose (carbohydrate), minerals, bacteria, and various micronutrients. The two types of proteins in the milk are the predominant 80% caseins (α S1, α S2, β , κ) and 20% whey (α -lactalbumin and β –lactoglobulin). Besides, Mahmood and Usman (2010) reported that goat's milk contained slightly lower lactose, which was 4.39 ± 0.34%, compared to 4.51 ± 0.38% of lactose in cow's milk. This is supported by most previous studies, provided that goat' s milk was found to have 4.08% of lactose, which is lower in comparison to 4.78% of lactose content in cow' s milk (Saini & Gill, 1991; International Dairy Federation, 1986; Posati & Orr, 1976). Basically, lactose (β -D-galactopyranosyl-(1 \rightarrow 4)-D-glucose) is a natural sugar and the main carbohydrate in all mammalian milks.



Nevertheless, Lehr and Chang (2010) reported that the average daily intake of milk in Malaysia was far less than in Scandinavia as a majority of Malaysians were found sensitive to lactose and this sensitivity is called 'lactose intolerance'. Moreover, Asmawi (2006) also proved that over 80% of Malaysians suffered from lactose intolerance. In short, lactose intolerance is the lack of lactase enzyme to digest sugar lactose, which normally disappears after weaning for most humans (Leonard, 2013). In fact, it has been proved that due to the lower lactose and other biological fluid properties, goat's milk is healthier than cow's milk for humans. Thus, as reported by Pouliot (2008) and Robinson and Tamime (1991), since in the early 1970s, a huge idea to separate milk components at the molecular level via separation technique to better utilize each component have been implemented in the dairy industry.

Additionally, previous studies reported that several technologies could actually remove lactose from milk (Baker, 2005; Khider et al., 2004; Barba et al., 2001; Barba & Beolchini, 2000; Pouliot et al., 1999; Hall, 1995). First, lactose is hydrolyzed into glucose and galactose, but this method is not preferred due to the disadvantage in terms of sweetness. Besides, Novalin et al., (2005) and Mahoney (1992) reported that the sweetness increased up to 70% for sucrose and this high level of sweetness can be an advantage or disadvantage; depending on the purpose of the products. Second is through enzymatic hydrolysis, but it involves high enzyme cost (Bylund, 1995). The third alternative is to separate lactose via crystallization, however, it is limited to only by-products from whey or whey permeated (Vyas, 2003). Lastly, the fourth approach is chromatographic separation, proposed by Barillas and Solomons (1987), where milk is fractionated where lactose fraction is separated and the salts remain in the protein fraction or the protein/fat fraction. The advantage of the process is that instead of permeate, a pure lactose solution is obtained and all substances significant to the taste, including salts, remain in the milk. However, it is a time-consuming and complex process, and besides, expensive equipment is required.

At present, the membrane technology has become widely used because it does not require phase change in dewatering, unlike condensation and evaporation, hence an energy saving process, a non-thermal technique, higher separation efficiency, and organoleptic characteristics of the milk product may be retained (Humphrey & Siebert, 1992). It also replaced some conventional concentration techniques (Jelen & Tossavainen, 2003; Humphrey & Siebert, 1992; International Dairy Federation, 1991). In dairy industry today, there are four types of pressure-driven membrane filtration processes used, which are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) (Pouliot, 2008).

Ultrafiltration (UF) is a medium pressure-driven membrane filtration process that emerged in the 1970s, which can separate components on a molecular basis. UF has molecular weight cut-off (MWCO) in the range of 1-500 KDa and corresponds to a nominal pore diameter of 1 to 100 nm (Kulkarni et al., 2001; Zall, 1987). The cut-off means molecular weight of the smallest molecule that cannot pass through the membrane. The pore size of the UF membrane is selected based on the size of the molecules being separated.

In UF, water, minerals, sugars (lactose), urea, amino acids, organic acids, and vitamins pass through the membrane (Shakeel-Ur-Rehman, 2009; Nielsen, 2000), while retaining 20-40% of non-protein nitrogen compounds, up to 10% lactose and minerals, and other ions attached to proteins (calcium, magnesium, phosphate, and citrate). In other words, larger molecules, such as proteins, fat, and carbohydrates, are fully or partially retained; depending on the pore size of the membrane used (Mulder, 1991). UF was mainly used for producing low lactose dairy products from cow's milk (Patel et al., 1991; Edelsten et al., 1983; Kosikowski, 1979). Hence, applying UF membrane with MWCO greater than 10 KDa would lead to increased transmission or loss of essential milk proteins, while using UF membrane with pore size smaller than 5 KDa may cause inefficiency in the UF process due to a significant reduction in lactose transmission. That is why the most common cut-off in dairy standard is 10 KDa.

Generally, the membrane technique method possesses two possible phenomena that can affect the accuracy or the operation effectiveness, which are concentration polarization (CP) and fouling at the membrane surface (Ochando-Pulido et al., 2015; Norafifah et al., 2015; Castro & Gerla, 2005). Membrane fouling is the accumulation of soil, or foulant, on the surface or within the pores of a membrane. Fouling prolongs processing times, increases energy and cleaning costs, decreases separation efficiency, and may lead to irreversible clogging of the membrane (Brans et al., 2004; Choi, 2003). The severity of fouling may be controlled and reduced, but it is impossible to be completely vanished (Howell & Finnigan, 1991). Meanwhile, CP is the accumulation of excess particles in a thin layer adjacent to the membrane surface and is inherent of all membrane filtration processes. It may increase resistance to solvent flow, and thus, reduce the permeate flux (Song & Elimelech, 1995).

1.2 Problem Statement

An extensive amount of researches has been conducted and reported on the use of UF in dairy industry, particularly in the lactose separation process, and most researchers investigated the improvement of membrane performance efficiency only on cow's milk, while very limited research reported using UF on producing low-lactose goat's milk. This is due to the inherently lower lactose in goat's milk, ~4.39% as compared to cow's milk, 4.51% (Mahmood & Usman, 2010).

Even though goat's milk naturally has lower lactose than cow's milk (~4.39% compared to 4.51%), however, when consumed in a large amount, those intolerant to lactose may suffer several inconvenient symptoms, such as bloating, nausea, and diarrhea (Hogenauer & Hammer, 2010; Swagerty et al., 2002). This is due to the different collection of protein composition in goat's milk that form a weaker and softer bolus curd which coagulates faster during digestion (Cooke, 2010; Haenlein, 2004; López-Aliaga, 2003; Park, 1994), and smaller size of fat globules that is one fifth the size of fat globules in cow's milk which offer naturally homogenized mixture of fat in milk (Park et al., 2007; Alférez, 2001). Hence, goat's milk is originally not suitable as an alternative to cow's milk when a particular amount is consumed.

Therefore, goat's milk had been used throughout this research as it may contribute to the Malaysian dairy industry and a variety of products for Asians where over 90% of the population suffers from lactose intolerance (Sloan, 1999). Hence, the novelty of this research is the removal of lactose using goat's milk as a raw material. Moreover, goat's milk contains many benefits compared to other mammal milk because it is very close to human's breast milk, naturally homogenized, less likely to trigger lactose intolerance and irritability, easily digested, has better buffering quality which is good for ulcer treatment, and matches up to the human body.

In fact, recently people had realized the benefits of goat's milk as compared to cow's milk, where reported that more goats are being reared at present in Malaysia compared to cow in year 2003, as to meet the requirements of consumers for high quality, healthy, and a variety of food at reasonable price (Rezai et al., 2011; Liana et al., 2010; Omar, 2010; Arshad et al., 2006). Goat farming in Malaysia specifically has increased substantially during the last two decades, which is over 45% from 331 thousands (K) in 1990 to 482 K number of heads in 2013 (FAO, 2015). This leads to a good opportunity for Malaysia's dairy industry to utilize goat's milk for a low-lactose product.

C

Fouling and concentration polarization on membrane surface during goat's milk processing which deteriorating the flux and gave negative impact on product yield are the major problems in the dairy industry. Until today, the issue concerning how to overcome fouling in cross-flow hollow fiber ultrafiltration unit is still debated due to the complex composition characteristic of milk that consists of proteins, minerals, lactose, and fat which contributes to the major foulants during the dairy UF process (Cheryan, 1998; Zeman & Zydney, 1996). Hence, this study focuses on determining the best condition of processing parameters and evaluating the effects of the selected parameters on flux (J),

lactose rejection (Ri), concentration factor (CF), and accumulation rate (AR) as Yeh et al., (2003) and Cheryan (19980) reported that the most significant operating conditions in UF are trans-membrane pressure, cross flow velocity, feed concentration, and temperature.

Particularly, in aspects of lactose removal with sizes of 342 Da from goat's milk, fractionation via reverse osmosis (RO) and nanofiltration (NF) membrane are more efficient, but they need higher operating pressure and higher cost consumption compared to the UF process (Namvar-Mahboub & Pakizeh, 2012). Thus, UF was used in this study since it has been mainly used for producing low lactose dairy products from cow's milk (Patel et al, 1991; Edelsten et al, 1983; Kosikowski, 1979).

1.3 Objectives

- 1. To evaluate the effects of processing parameters on flux (J), lactose rejection (Ri), concentration factor (CF), and accumulation rate (AR) in the fractionation of lactose using cross-flow ultrafiltration membrane.
- 2. To evaluate and compare the quality of concentrated goat's milk powder with commercialized milk products in terms of milk composition and lactose concentration.

1.4 Scopes of Work

The scopes of work in this research had been limited to determine the performances of two sizes of MWCO 5 KDa and 10 KDa UF membranes in a cross-flow filtration unit by means of lactose concentration, permeate flux (J), and lactose rejection percentage (%Ri), and accordingly, to select the best processing parameters examined, which were TMP and feed flow-rate. Hence, in order to ensure the comparability of low lactose goat's milk produced with other commercialized milk, all the milk samples were compared after atomization by spray-drying in terms of lactose percentage range and nutritional composition. Nonetheless, this research was not extended to determine the amount of protein content in concentrated milk.

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PUBLICATIONS

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