UTILIZATION OF WHEY BY USING THE MEMBERANE FILTRATION TECHNIQUE

BY

HASSAN MOHAMED HASSAN HASSAAN

B.Sc. Agric. (Dairy Science and Technology) El-Azhar Univ.1981 M.Sc. Agric (Dairy Science and Technology) El-Azhar Univ.1993

A thesis submitted for partial fulfillment of the requirements for the degree of Doctor of Philosophy

in

Agricultural Science (Dairy Science and Technology)

Food Science Department Faculty of Agriculture Ain Shams University

2000

UITLIZATION OF WHEY BY USING THE MEMBERANE FILTRATION TECHNIQUE

\mathbf{BY}

HASSAN MOHAMED HASSAN HASSAAN

B.Sc. Agric. (Dairy Science and Technology) El-Azhar Univ.1981 M.Sc. Agric (Dairy Science and Technology) El-Azhar Univ.1993

Under the Supervision of:

PROF. DR. A. E. HAGRASS

Professor of Dairy Science and Technology, Faculty of Agriculture, Ain Shams University.

PROF. DR. A. E. FAYED

Professor of Dairy Science and Technology, Faculty of Agriculture, Ain Shams University.

PROF. DR. F. M. ABO EL-NAGA

Chief Researcher of Dairy Science and Technology, Dairy Technology Department, Animal Production Research Institute, Agriculture Research Center.

جامعة عين شمس كلية الزراعة قسم علوم الاغذية

رسالة دكتوراه

اسم الطالب : حسن محمد حسن حسان

عنوان الرسالة : الإستفادة من الشرش باستعمال تكنيك الترشيح بالأغشية

اسم الدرجة : دكتوراه

لجنة الإشراف

أ.د . عبد المنعم البدوي هجرس

استاذ علوم و تكنولوجيا الالبان - كلية الزراعة -جامعة عين شمس

أ.د . عاطف السيد فايد

استاذ علوم و تكنولوجيا الالبان - كلية الزراعة- جامعة عين شمس

أ.د. فاروق محمد ابو النجا

رئيس بحوث بقسم بحوث تكنولوجيا الألبان- معهد بحوث الانتاج الحيوانى مركز البحوث الزراعية

جامعة عين شمس كلية الزراعة

رسالة دكتوراه

اسم الطالب : حسن محمد حسن حسان

عنوان الرسالة : الإستفادة من الشرش باستعمال تكنيك الترشيح بالأغشية

الدرجة : درجة الدكتوراه (علوم و تكنولوجيا الألبان)

لجنة الإشراف

أ.د . عبد المنعم البدوي هجرس

استاذ علوم و تكنولوجيا الالبان - كلية الزراعة -جامعة عين شمس

أ.د . عاطف السيد فايد

استاذ علوم و تكنولوجيا الالبان - كلية الزراعة- جامعة عين شمس

أ.د . فاروق محمد ابو النجا

رئيس بحوث بقسم بحوث تكنولوجيا الألبان- معهد بحوث الانتاج الحيواني - مركز البحوث الزراعية

تاريخ البحث 20 / 9 /1993

الدر اسات العليا:

ختم الإجازة أجيزت الرسالة بتاريخ / 2000/ 11 / 7

موافقة مجلس الكلية موافقة مجلس الجامعة 2000 / 2000 / /

الإستفادة من الشرش باستعمال تكنيك الترشيح بالأغشية

ر سالة مقدمة من

حسن محمد حسن حسان

بكالوريوس علوم زراعية (علوم وتكنولوجيا الالبان) جامعة الأزهر 1981 ماجستير في العلوم الزراعية (علوم وتكنولوجيا الالبان) جامعة الأزهر 1993

للحصول على درجة دكتور فلسفة فى العلوم الزراعية (علوم وتكنولوجيا الالبان)

قسم علوم الاغذية كلية الزراعه جامعة عين شمس

Approval Sheet

UTILIZATION OF WHEY BY USING THE MEMBERANE FILTRATION TECHNIQUE

BY

HASSAN MOHAMED HASSAN HASSAAN

B.Sc. Agric. (Dairy Science and Technology) El-Azhar Univ.1981 M.Sc. Agric (Dairy Science and Technology) El-Azhar Univ.1993

This thesis for the Ph. D. degree has been approved by:

PROF. Dr. I. A. ABD EL - GAWAD

Professor of Dairy Science and Technology, Faculty of Agriculture, Cairo University.

PROF. Dr. A. A. ASKER

Professor of Dairy Science and Technology, Faculty of Agriculture, Ain Shams University.

PROF. Dr. A. E. HAGRASS

Professor of Dairy Science and Technology, Faculty of Agriculture, Ain Shams University.

Date of examination: / / 2000

ACKNOWLEDGEMENT

I would like to express my thanks and gratitude to **Prof. Dr. A. E. Hagrass**, Prof. of Dairy Science and Technology and **Prof. Dr. A. E. Fayed**, Prof. of Dairy Science and Technology, Food Science Department, for their fruitful efforts during their supervising all the stages of this investigation.

Deep gratitude is also extended to **Prof. Dr. F. M. Abo El- Naga,** Chief Researcher of Dairy Science and Technology, Dairy technology Department, Animal Production Research Institute, Ministry of Agriculture for his supervision and kind through out this work.

Thanks also should be sent to all colleagues at Ain Shams University for every help and facilities offered to make this work possible. I thank all my friends in the Dairy Technology Department, Dairy Processing Unit and Dairy Chemistry and Microbiology Departments at Animal Production Research Institute for their help in those occasions, I needed to do two things at the same time and above all for their moral support. I specially thank my friend Dr. Hazem El- Mahdy, for his guidance in the statistical work.

To my parents, my lovely wife and my lovable son for doing every thing they could for me, with their love, admiration, sacrifice and appreciation.

ABSTRACT

Hassan Mohamed Hassan Hassaan, Utilization of whey by using the membrane filtration technique. Unpublished Doctor of Philosophy dissertation, Department of Food Science, Faculty of Agriculture, Ain Shams University, 2000.

The possibility of complete utilization of derived whey in some varieties of frozen desserts was aimed to study depending on some effective techniques.

Whey was centrifuged, ultrafiltrated (UF) to concentration factor (CF) of 20 and diafiltrated (DF) after the dilution with tap water amounted 20 times of the yielded UF whey protein concentrate (UFWPC). The obtained UF whey permeate was concentrated by reverse osmosis (RO) to CF of 3.

The DFWPC was used as a milk solids not fat (MSNF) source proportionally in combination with skimmilk powder (SMP) in frozen yoghurt at the level of 25, 50, 75 and 100%. The control of frozen yoghurt based on 3% fat, 12% MSNF, 12% sucrose, 5% corn syrup and 0.25% CMC stabilizer was done using SMP as MNSF supply.

The results revealed that, the levels of total nitrogen, specific gravity (sp. gr.), freezing point (fp), dynamic viscosity (dv), consistency coefficient (cc) and yield stress (ys) increased as the protein of DFWPC in frozen yoghurt mix rised. While, the overrun, melting resistance and count of lactic acid bacteria of the resultant frozen yoghurt decreased by increasing the DFWPC level. The overall organoleptic quality of samples of 25% DFWPC frozen yoghurt was superior to them.

The RO concentrate of UF whey permeate was exposed for acidic lactose conversion compared with the enzymatic one. The enzymatic hydrolysis was carried out at 5 or 40 °C and pH 6.5 using certain concentrations of β - galactosidase of nil, 125, 250, 500, 750 and 1000 U / ml and incubated for 6 h. The acidic hydrolysis was done at 40, 50 and 60 °C for 24h at pH 1.2 or 1.7 using concentrated HCl. The obtained acidic hydrolyzed RO concentrate of UF whey

permeate (ACP) at pH 1.2 and originalized to pH 5.5, using any group of the following three alkaline agents namely sodium & potassium

carbonate (1:1); sodium & potassium carbonate in combination with sodium bicarbonate (1:1:2) or sodium carbonate & bicarbonate in combination with potassium hydroxide (1:2:3), was used for Strawberry water ice based on 30, 25 and 20 % sucrose, 0.5% CMC stabilizer and 0.001% natural red colour & flavour essence. The control was made using tap water instead of ACP.

The acidic conversion of lactose was slowly at the beginning and rapid near the end of the conversion period conversely to enzymatic one. The highest level of enzymatic lactose conversion (88.74%) was obtained at 5 °C using 1000 U/ml, while the corresponding figure of acidic conversion (74.67%) was achieved at pH 1.2 and 50°C. The ACP with conversion level of about 74% gained at pH 1.2 and 40 °C, that was converted into water ice mix, led to increase the levels of dry matter, total sugar, ash, sp. gr., dv, cc as well as ys of the mix and decrease in its fp. The levels of sp. gr. and overrun of ACP water ice, especially at 25% sucrose, were higher than those of the control. The ACP frozen product of the third alkaline agent, especially at 25% sucrose, resisted to melt more than the others involving the control. The palatability of ACP water ices with 25% sucrose and originalized with the first alkaline agent group as the control was superior to them.

The results led to concluded that, the full utilization of whey could be achieved via the use of DFWPC as partial (25%) MSNF supply in frozen yoghurt making, Likewise, ACP could be used for water ice making provided that more discharging of minerals is needed to expand the utilization possibilities. Nanofiltration or ion exchanges process was recommended in this respect.

Key words: - Mozzarella whey, ultrafiltration, diafiltration, reverse osmosis, lactose conversion, frozen yoghurt, water ice.

CONTENTS

			Page	
I.		Introduction	1	
II	Review of Literature			
	1.	Adaptation of ultrafiltration whey	3	
		derivatives for some varieties of		
		frozen desserts.		
	1.1.	Utilization of whey protein	3	
		concentrates (WPC) in frozen		
		dessert manufacture.		
	1.1.1.	Ice cream	4	
	1.1.2.	Ice milk	12	
	1.1.3.	Frozen yoghurt	12	
	1.2.	Application of lactose conversion	13	
		in frozen dessert manufacture.		
	1.2.1.	Significance of lactose conversion	13	
	1.2.2.	Ways of lactose conversion	16	
	1.2.2.1.	Enzymatic hydrolysis of lactose	16	
	1.2.2.2.	1.2.2.2. Acidic hydrolysis of lactose		
	1.2.3.	Varieties of frozen desserts made	22	
		with hydrolyzed lactose		
	1.2.3.1.	Ice cream	22	
	1.2.3.2.	Ice milk	25	
	1.2.3.3.	Frozen yoghurt	26	
	1.2.3.4.		27	
III.	Materials and Methods			
	1.	Materials	29	
	I.1	Buffalo's milk	29	
	I.2.	Ordinary whey	29	
	I.3	Skimmilk powder	29	
	1.4.	Sucrose	29	
	1.5.	Corn syrup	29	
	1.6.	Sodium carboxy methyl	31	
		cellulose		

1.7.	Vanillia	31
1.8.	Artificial flavour	31
1.9.	Red colour	31
1.10.	β-Galactosidase enzyme	31
1.11.	Lactic acid starter strains	31
2.	Experimental procedures	32
2.1.	Ultrafiltration (UF) fractionation	32
	of whey	
2.2.	Utilization of diafiltrated whey	32
	protein concentration (DFWPC)	
	in frozen yoghurt manufacture	
2.2.1.	Preparation of yoghurt	32
2.2.2.	Manufacture of frozen yoghurt	33
2.3.	Lactose conversion	35
2.3.1.	Enzymatic hydrolysis	35
2.3.2.	Acidic hydrolysis	35
2.4.	Utilization of acidic hydrolyzed	35
	RO concentrate of UF whey	
	permeate in water-ice making	
2.4.1.	Originalization of the acidic	35
	hydrolyzed RO concentrate of	
	UF whey permeate	
2.4.2.	Water ice manufacture	36
3.	Methods of Analysis	38
3.1.	Chemical Analysis	38
3.1.1	Determination of dry matter	38
	content	
3.1.2.	Determination of fat content	38
3.1.3.	Determination of total protein	38
	content	
3.1.4	Determination of ash content	38
3.1.5	Measurement of pH value	38
3.1.6.	Determination of titratable	38
	acidity content	
3.1.7.	Determination of lactose content	39
	and its convert sugar	

	3.1.8	Calculation	of	retention	39	
		coefficient.			40	
	3.1.9.	, , , , , , , , , , , , , , , , , , ,				
	3.2.	Physical Analys			41	
	3.2.1 Measurement of specific gravity				41 41	
	3.2.2	Measurement of freezing point				
	3.2.3	Calculation of t	he ove	rrun	41	
	3.2.4.	Determination resistance	of th	e melting	41	
	3.3.	Rheological ana	alysis		41	
	3.4	Microbiologica	l analys	sis	42	
	3.4.1.	Standard plate of	count (SPC)	42	
	3.4.2	Lactic Acid Bac	cteria		42	
	3.4.3.	Coliform count			42	
	3.5	Organoleptic ev	aluatic	on	43	
	3.6.	Statistical analy	'sis		43	
IV.		Results and Discu	ussion		44	
		-				
	1.	Fractionation of	-	•	44	
	1.	Fractionation of membrane filtrat	-	•	44	
	1.	membrane filtrat Utilization of dia	tions a filtrate	nd ed whey	44	
	1.	membrane filtrat Utilization of dia protein concentra	tions a filtrate ate (DI	nd ed whey EWPC) in	44	
		membrane filtrate Utilization of diate protein concentrate frozen yoghurt m	ions ar filtrate ate (DI nanufae	nd ed whey EWPC) in ecture.		
	1. 1.1.	membrane filtrat Utilization of dia protein concentra frozen yoghurt m Fractionation of w	tions and filtrate ate (DI nanufactor)	nd ed whey EWPC) in ecture.	44	
	1.1.	membrane filtrate Utilization of diagonate protein concentrate frozen yoghurt m Fractionation of w membrane filtration	tions and filtrate to the contract of the cont	nd ed whey EWPC) in ecture.	44	
		membrane filtrate Utilization of diate protein concentrate frozen yoghurt membrane filtration Ultrafiltration and	filtrate (DI nanufactor) they by diafilts	nd ed whey EWPC) in ecture.		
	1.1. 1.1.1.	membrane filtrate Utilization of diag protein concentration frozen yoghurt m Fractionation of w membrane filtration Ultrafiltration and fractionation of w	tions and filtrate ate (DI anufact) whey by ons diafilts they	nd ed whey EWPC) in ecture.	44	
	1.1.	membrane filtrate Utilization of diagonate of the concentration of with the concentration of with the concentration of with the concentration of the concent	tions and filtrate tate (DI nanufactor) and they by the diafilts they UF – w	ed whey EWPC) in ecture. ration	44	
	1.1. 1.1.1.	membrane filtrate Utilization of diagonal protein concentration frozen yoghurt membrane filtration Ultrafiltration and fractionation of well Concentration of Upermeate by rever	tions and filtrate ate (DI nanufact) they by they be diafilted by the work of	nd ed whey EWPC) in cture. ration hey osis	44	
	1.1. 1.1.1.	membrane filtrate Utilization of diagonal protein concentrate frozen yoghurt membrane filtration Ultrafiltration and fractionation of well Concentration of Upermeate by rever Application of diagonal	filtrate ate (DI anufacte) by they by	ed whey EWPC) in ecture. ration hey osis d whey	44	
	1.1. 1.1.1. 1.1.2.	membrane filtrate Utilization of diagonal protein concentration of we membrane filtration under fractionation of we concentration of we concentration of the permeate by revert Application of diagonal protein concentration of the protein concentration c	filtrate ate (DI anuface whey by ons diafilte hey UF – w se osm filtrate e (DFV	ed whey EWPC) in ecture. ration hey osis d whey	44 44 47	
	1.1. 1.1.1. 1.1.2. 1.2.	membrane filtrate Utilization of diagonate of the protein concentration of we membrane filtration and fractionation of we concentration of the permeate by rever Application of diagonate of the protein concentrate frozen yoghurt material of the protein yoghurt material of the protein yoghurt material yoghurt mat	filtrate ate (DI anufac whey by ons diafilte they UF – w se osm filtrate e (DFV aking	ed whey EWPC) in cture. ration hey osis d whey VPC) in	44 44 47 48	
	1.1. 1.1.1. 1.1.2. 1.2.	membrane filtrate Utilization of diagonal protein concentrate frozen yoghurt management of war and fractionation of war actionation of diagonal protein concentrate frozen yoghurt man actionation of yoghurt man actionation y	filtrate ate (DI anuface whey by ons diafilte they UF – w se osm filtrate e (DFV aking en yogh	ed whey EWPC) in cture. ration hey osis d whey VPC) in	44 44 47 48	
	1.1. 1.1.1. 1.1.2. 1.2. 1.2.1 1.2.1.1.	membrane filtrate Utilization of diagonal protein concentrate frozen yoghurt membrane filtration Ultrafiltration and fractionation of well concentration of Well permeate by rever application of diagonal protein concentrate frozen yoghurt material composition of the properties of frozen yoghurt material compositions.	filtrate ate (DI anuface whey by ons diafilte they UF – w se osm filtrate e (DFV uking en yogh	ed whey EWPC) in cture. ration hey osis d whey VPC) in	44 47 48 48	
	1.1. 1.1.1. 1.1.2. 1.2.	membrane filtrate Utilization of diagonal protein concentrate frozen yoghurt management of war and fractionation of war actionation of diagonal protein concentrate frozen yoghurt man actionation of yoghurt man actionation y	filtrate ate (DI anuface whey by ons diafilte they UF – w se osm filtrate e (DFV uking en yogh	ed whey EWPC) in cture. ration hey osis d whey VPC) in	44 44 47 48	

1.2.1.1.3.	Ash and total sugars contents	50
1.2.1.1.4.	Titratable acidity and pH value	50
1.2.1.2.	Physical properties	50
1.2.1.2.1	Specific gravity and freezing point	51
1.2.1.3.	Rheological properties	51
1.2.1.3.1.	Dynamic viscosity	51
1.2.1.3.2.	Consistency coefficient and Yield	55
	stress	
1.2.2	Properties of resultant frozen	55
	yoghurt	
1.2.2.1.	Physical properties	55
1.2.2.1.1.	Specific gravity and gravity and	55
	overrun percent	
1.2.2.1.2.	Melting resistance	61
1.2.2.2.	Bacterial aspects	63
1.2.2.2.1.	Lactic acid bacteria count	63
1.2.2.2.2.	Coliform count	63
1.2.3.	Organoleptic quality	63
1.2.3.1.	Appearance	63
1.2.3.3	Consistency	66
1.2.3.3.	Flavour	66
1.2.3.4	Total score	67
1.2.3.5	Ranking test	67
2.	Utilization of lactose converted	70
	RO concentrate of UF- whey	
	permeate in water ice making	
2.1.	Enzymatic and acidic lactose	70
	conversion of RO concentrate of	
	UF – whey permeate	
2.1.1	Enzymatic lactose conversion	71
2.1.1.1.	Effect of the enzyme concentration	71
	in combination with the	
	temperature	
2.1.1.2.	Stability of pH value during	77
	enzymatic lactose conversion	
2.1.2.	Acidic lactose conversion	81

		2.2.	Properties of strawberry wat	er ice	88
			mix		
		2.2.1	Chemical composition		88
		2.2.1.1.	Dry matter content		88
		2.2.1.2.	Total nitrogen content		88
		2.2.1.3.	Total sugar content		90
		2.2.1.4.	Ash content		90
		2.2.1.5.	Titratable acidity		91
		2.2.2.	Physical properties of the		91
			strawberry water ice mixes		
		2.2.2.1.	Specific gravity		91
		2.2.2.2	Freezing point		91
		2.2.2.3.	Rheological properties		94
		2.2.2.3.1.	Dynamic viscosity		96
		2.2.2.3.2.	Consistency coefficient and stress	yield	96
		2.3.	Properties of the resultant		99
			strawberry water ice		
		2.3.1.	Physical properties		99
		2.3.1.1.	Specific gravity and overrun		99
		2.3.1.2.	Melting resistance		103
		2.3.2.	Bacterial aspects		105
		2.3.3.	Organoleptic quality		105
		2.3.3.1	Appearance		105
		2.3.3.2	Consistency		108
		2.3.3.3.	Flavour		108
		2.3.3.4	Total score		108
		2.3.3.5.	Ranking test		109
V.		Summai	_	Conclusi	
	112		•		
VI.		Referen	ces		
	118				
VII.	132	Append	ices		

VIII. Arabic summary

LIST OF TABLES

No		Page
1	Some properties of lactose and its convert	15
2	sugar compared with sucrose.	20
2	Gross composition of buffalo's milk, whey and skimmilk powder.	30
3	The formulas (kg/100kg) of frozen yoghurt mix made by substitution of skimmilk powder (SMP) with diafiltrated whey protein concentrate (DFWPC).	34
4	The formulas (kg/100kg) of strawberry water-ice mix made by substitution of water with acidic hydrolyzed RO concentrate of UF-whey permeate (ACP)	37
5	Gross composition percentage (on dry basis) of whey derivative of Mozzarella cheese as affected by the process of centrifugation, ultrafiltration (UF), diafiltration (DF) and reverse osmosis (RO).	45
6	Retention coefficient of whey constituents as a function of ultrafiltration (UF) of whey, diafiltration (DF) of UF whey protein concentrate and reverse osmosis (RO) of UF- whey permeate.	46
7	Chemical composition of frozen yoghurt mix as affected by the replacement level of skimmilk powder (SMP) with diafiltrated whey protein concentrate (DFWPC).	49
8	Physical and rheological properties of frozen yoghurt mix as affected the replacement level of skimmilk powder	52