THE USE OF MILK PERMEATE AS A BASE FOR SOME NEW FOOD PRODUCTS BY

AHMED HASSAN AHMED ZAGHLOUL



A thesis submitted in partial fulfilment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

in

Agricultural Science

(Dairy Science and Technology)

49205

Department of Food Science

Faculty of Agriculture Ain Shams University

1993

Approval sheet

THE USE OF MILK PERMEATE AS A BASE FOR SOME NEW FOOD PRODUCTS

BY

AHMED HASSAN AHMED ZAGHLOUL

B.Sc. (Dairy Science & Technology),
Al-Azhar Univ., 1979
M.Sc. (Dairy Science & Technology),
Ain Shams Univ., 1986

This thesis for ph.D. degree has been approved by :

Prof. Dr. M. K. Ibrahim

Prof. of Dairy Science and Technology, Fac. of Agric. Cairo University .

Prof. Dr. M. A. El-GHANDOUR

Prof. of Dairy Science and Technology, Fac. of Agric. Ain Shams University .

Prof. Dr. G. A. MAHRAN

Prof. of Dairy Science and Technology, Fac. of Agric. Ain Shams University, and Head of Department . (Supervision)

Date of approval : | } / 9 / 1993 .



The Use of Milk Permeate as A Base For Some New Food Products

BY

AHMED HASSAN AHMED ZAGHLOUL

B.Sc. (Dairy Science & Technology), Al-Azhar Univ., 1979

M.Sc. (Dairy Science & Technology), Ain Shams Univ., 1986

Under the Supervision of :

Prof. Dr. G. A. MAHRAN
Prof. of Dairy Science & Technology
and Head of Bept. of Food Sci. Fac.
of Agric. Ain Shams Univ.

Prof. Dr. M. H. ABD EL-SALAM Prof. of Dairy Science and Head of Food Technology & Nutrition Division, National Research Center.

Dr. H. F. HAGGAG Assoc. Prof. of Dairy Science & Technology, Fac. of Agric., Ain Shams Univ.

Abstract

The lactulose is a derivative of lactose known for its nutritional and medical importance. Firstly, the suitability of a colourimetric method for its determination on the basis of its fructose moiety was investigated. Secondly, the optimum conditions for the preparation of lactulose from UF milk (or whey) permeate was studied. Finally, the laboratory prepared lactulose, pure lactulose and pure lactose were tried in yoghurt production from buffaloe's milk in order to improve its nutritive value and therapeutic effect. Attained results revealed that fortified yoghurt with 0.4% lactulose showed good properties.

Key words: Lactose, Lactulose, Permeate, Whey and Yoghurt.

Acknowledgement

The author would like to express his deep gratitude and appreciation to Prof. Dr. G. A. Mahran head of Food Science Department and Dr. H. F. Haggag Associate Professor of Dairying at the same Department, Faculty of Agriculture, Ain Shams University for suggesting the proplem, kind, encouragement and constructive advice during conducting this investigation and preparation of the text.

Deep gratitude is also extended to Prof. Dr. M. H. Abd El - Salam Professor of Dairying and Head of the Division of Food Technology and Nutrition, National Research Center (NRC) for the help, guid once and suggestions he kindly offered during the investigation and writing this manuscript.

The author wishes also to express his gratefulness to Prof. Dr. M. B. Mahfouz Professor of Dairying at the Dept. of Dairying and Food Technology at N.R.C. for his intrest throughout this study.

Thanks also are due to all members of the Department of Food Science, Ain Shams University and the Department of Dairying and Food Technology, N.R.C. for every possible help and facilities offered to make this work possible.

CONTENTS

	Page
1-INTRODUCTION	. 1
2-REVIEW OF LITERATURE	. 3
2.1 - Lactulose; preparation and properties	. 3
2.2 - Preparation	. з
2.3 - Nutritional and medical uses of lactulose	. 9
3-MATERIALS AND METHODS	
3.1 - Materials	14
3.1.1 - Chemicals	14 14
3.2 - Preparation of lactulose from lactose solution or permeate	14
3.2.1 - Alkali isomerization	14
solution	15
concentrate	15
3.2.4 - Ion exchanger chromatography	15 15
3.2.4.1-Preparation of cation exchange resin 3.2.4.2-Preparation of anion exchange resin	13
(Dowex 1)	16
3.2.4.3-Cation/Anion exchange chromatography 3.2.4.4-Regeneration of boron - selective	16
resin (18A - 743)	16 17
2.2.4.3-Kemoval of donon	17
3.3 - Analysis of permeate and isomerized permeate	
and lactose solution	17
3.3.1 - Lactulose determination	17
3.3.2 - Lactose determination	18
3.3.3 - Boron determination	20
3.4 - Manufacture of yoghurt	21
3.5 — Analysis of yoghurt	22
3.5.1 - Chemical analysis	
3.5.1.1-Determination of total solids	22
3.5.1.2-Determination of acidity	22
t m l t-lintagmination of oil nalis	-3-3

•	age
3.5.1.4-Determination of soluble nitrogen	22
3.5.1.5-Determination of total volatile fatty acids	22
3.5.1.6-Determination of acetaldehyde	23
3.5.1.7-Determination of carbohydrates	24
3.5.2 - Microbiological analysis	25
3.5.3 - Assessment of organoleptic properties	26
3.5.4 - Statistical analysis	27
-EXPERMINTAL	
4.1 - Part 1	
Development of a method for the determination	
of lactulose in the presence of other sugars	28
4.1.1 - Selection of wavelength for lactulose	
determination by TBA reaction	29
4.1.2 - Selection of time and temperature for	
TBA reaction with lactulose	29
4.1.3 - Effect of lactose present in lactulose	
solution on the colour formation with	7.0
TBA	30
4.2 - Part II	
Ample condition for maximum production of	
lactulose from ultrafiltration milk and whey	
permeate	31
4.2.1 - Selection of isomerization agent	31
4.2.2 - Effect of heating time and duration	
on the formation of lactulose from	
lactose solution	32
4.2.3 - The formation of the brown colour	
on alkalı isomerization of lactose solution and milk ultrafiltrate	32
4.2.4 - Alkali isomerization and formation	32
of lactulose from permeate of	
different NaCl content	32
4.2.5 - Separation and concentration of	
lactulose solution by ultraosmosis	33
4.2.6 - Attempts of purify the lactulose	
solution by ion exchange resin	33
4.3 - Part III	
Production of yaghurt fortified with	
lactulose	35
5-RESULTS AND DISCUSSION	
5.1 - Part I	
5.1.1 - Selection of wavelength for lactulose	
determination by TBA reaction	37
5.1.2 - Selection of time and temperature for	٠,
TPA praction with lactulose	37

	Page
5.1.3 - Linearity and accuracy of lactulose	
determination by TBA reaction	41
5.1.4 - Effect of lactose present in lactulose	
solution on the colour formation with	
TBA	44
126	• •
5.2 - Part II	
	47
5.2.1 - Selection of the isomerization agent	47
5.2.2 - Effect of heating time and duration	
on the formation of lactulose from	
lactose solution	47
5.2.3 - The formation of the brown colour on	
alkali isomerization of lactose	
solution and milk ultrafiltrate	53
5.2.4 - Alkali isomerization and formation of	
lactulose from permeate and different	
NaCl content	59
5.2.5 - Separation and concentration of	
lactulose solution by Ultraosmosis	71
5.2.6 - Attempts to purify the lactulose	-
solution by ion exchange resins	76
Sold to the same of the same o	, 0
5.3 - Part III	
5.3.1 - Gross composition	40
5.3.2 - Galactose, Lactulose and lactose	-00
	100
content yoghurt	
5.3.2.1-Identification	
5.3.2.2-Quantitative determination	
5.3.3 - Lactic acid bacterial count (LABC)	
5.3.4 - Organoleptic properties	110
6-SUMMARY AND CONCLUSIONS	
6.1 - Part I	
Development of a method for the dtermination	
of lactulose in the presence of other sugars	. 113
6.2 - Part II	
Ample conditions for maximum production of	
lactulose from ultrafiltration milk and whey	
permeate	114
<i>p</i> = 1 = 1	
6.3 - Part III	
Production of yoghurt fortified with	
lactulose	117
idituiose	/
7-REFERENCES	1 10
/ TRANSPORTED	7
C ADADIO CIRCIADO	
8-ARABIC SUMMARY	127

LIST OF TABLES

Table	Title	Page
i	Optical density at 432.5 nm of the reaction of lactulose and lactose (0.05 mg/ml) with TBA at 90 C and in a boiling water bath for variable periods .	40
2	Absorbance at 432.5 nm of reaction mixture of solutions of different lactulose contents (0.02-0.2 mg/ml) with TBA in a boiling water bath for 6 and 8 minutes .	42
3	Repeatability and accuracy of lactulose $\{L*\}$ determination by the TBA method .	45
4	Calculated lactulose content of solutions containing increasing quantities of lactose.	46
5	Lactulose content formed in 5% lactose solution after alkali isomerization at 70 C/30 minutes using different agents .	48
6	Effect of heating time and duration on the lactulose formation during the alkali isomerization of 5% lactose solution .	49
7	The effect of heating at 120 C/ 15 min. on the lactulose formation during the alkali isomerization of 5% lactose solution .	52
8	Effect of the sodium chloride (NaCl) on the % lactulose formation during alkali isomerization of permeate .	60
9	Lactulose content and percentage lactose transformation into lactulose in lactose solution after alkali isomerization for different periods at 70 C in open air .	65
10	<pre>tactulose content and percentage lactose transformation into lactulose in ultrafil- tration milk permeate solution after alkali isomerization for different periods at 70 C in open air .</pre>	66
11	Lactulose content and percentage lactose transformation into lactulose in lactose solution after alkali isomerization for	
	different periods at 70 C under nitropen .	67

Table	Title	age
12	Lactulose content and percentage lactose transformation into lactulose in ultrafil-tration milk permeate solution after alkali isomerization for different periods at 70 C under nitrogen.	68
13	Factorial analysis and simple effect of lactulose formation using 5% lactose solution with different heat duration and various atmospheric of reaction (open air & under nitrogen).	69
14	Factorial analysis and simple effect of lactulose formation using permeate (from milk ultrafiltration) with different heat duration and various atmospheric of reaction (open air & under nitrogen).	70
15	Lactose content of retentate (concentration factor 5) and permeate durong ultraosmosis and diafiltration of alkali isomerized 5% lactose solution .	73
16	Lactulose content of retentate (concentration factor 5) and permeate during ultraosmosis and diafiltration of alkali isomerized 5% lactose solution .	74
17	Boron content of retentate (concentration factor 5) and permeate during ultraosmosis and diafiltration of alkali isomerized 5% lactose solution.	75
18	Changes in the copmosition of alkali isome- rized lactose solution during chromatography on strong cationic and anionic exchange resins.	78
19	The changes in lactose, lactulose and boron content of concentrated isomerized permeate, by ultraosmosis during the course of purification.	79
20	The changes in lactose, lactulose and boron content of concentrated isomerized permeate during purification by ion exchange resins.	80
21	The pH of fresh and stored yoghurt (one week at refrigerator) manufactured by adding lactulose or lactose with different ratios to fresh buffaloe's milk .	87

Table	Title	Page
32	Factorial analysis and simple effect of adding lactose and lactulose as well as storage period on the acetaldehyde content of resultant yoghurt.	99
3 3	Galactose, lactulose and lactose content of the fresh and stored treated yoghurt manufactured with added different concentrations of lactose and lactulose. (g/100 g sample).	105
34	The effect of adding lactulose and lactose to buffaloe's milk on the lactic acid bacerial count of the resultant yoghurt (average of three replicates).	108
35	Factotial analysis and simple effect of adding lactose and lactulose as well as storage period on the lactic acid bacterial count (LABC) content of resultant yoghurt.	109
36	Organoleptic properties of the resultant yoghurt manufactured by adding different ratios of laboratory prepared and pure lactulose as well as lactose. (average of	117
	three replicates) .	112

LIST OF FIGURES

Figure	Title	Page
i	The role of lactulose for health .	1 1
2	Absorption curve of reaction mixture of lactulose solution (0.05 mg/ml) with TBA solution .	38
3	Absorption curves of the reaction mixtures of fructose, lactose, glucose and galactose solutions (0.05 mg/ml) with TBA.	3 9
4	Standard curve of lactulose determination by TBA reaction ,	43
5	Effect of heating time and duration on the % lactulose formation during alkali isomerization of 5% lactose solution .	50
6	Absorption spectra of lactose and permeate solutions befor isomerization.	54
7	Absorption spectra of lactose and permeate solutions after alkali isomerization at 70 °C for 1 hour.	55
8	Absorption spectra of lactose and permeate solutions after alkali isomerization for 3 hours at 70 °C.	56
9	Absorption spectra of lactose and permeate solutions after alkali isomerization at 70 °C for 2 hours under nitrogen.	57
10	Absorption spectra of lactose and permeate solutions after alkali isomerization at 70 °C for 2 hours in open air.	58
11	Effect of salt on the % lactulose formation during alkali isomerization of permeate.	61
12	Permeation rate of the lactose solution	72

Figure	Title	Page
13	The changes with lactose and lactulose contents of concentrated isomerized permeate solution during the course of	
	purification.	81
14	Preparation of lactulose from permeate .	84
15	Resolution of galactose and lactose from processed yoghurt (manufactured from standardized buffaloe's milk, 3% fat as a control) on a carbohydrate analysis column	
	at ambient temperature.	103
16	Resolution of galactose, lactulose and lactose from processed yoghurt (manufactured from standardized buffaloe's milk fortified with lactulose) on a carbohydrate analysis column at ambient	
	temperature.	104

