UNIVERSITI PUTRA MALAYSIA

DESIGN, CONSTRUCTION AND TESTING OF FREEZE CONCENTRATOR

ROSNAH BT HAJI SHAMSUDIN

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MASTER OF SCIENCE
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2000

To the most beloved ones:

Mak, Abah dan adik .....

Thanks for your continuos support and encouragement

My husband Azmi bin Peryatin .....

For the aspiration and inspiration

My daughter Nurul Izzah .....

Ease my stress and tension

My parent in law and Brothers in law .....

Thanks for a great understanding

Syaiful, Yus, Mai, Azie, Das, Ros, Kak Zai and Aidy

Thanks for your helpful and comment

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

DESIGN, CONSTRUCTION AND TESTING OF FREEZE CONCENTRATOR

By

ROSNAH BT HAJI SHAMSUDIN

November 2000

Chairman: **Hishamuddin bin Jamaludin, MSc**

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The main aspects of this study, were the design, construction and performance of a freeze concentrator. The design process consists of three main parts: a vertical tube heat exchanger, an auger to scrape the ice formed and a refrigeration unit to bring the temperature to below freezing.

Basically, the freeze concentration process used involves ice formation from a juice solution at the surface of a vertical tube heat exchanger. The ice will be scraped by a rotating auger, which pushes it out through a window and collected in a reservoir. This results in a product of higher solute concentration than its concentration in the feed.

The potential application of the freeze concentrator was experimented using sugar solutions at three different feed concentrations i.e. 7.5%, 10.3% and 12% brix, as well as pineapple juice at 11.4% brix. The freezing point depression for the solutions were respectively –1.07°C, -1.15°C, –1.49°C and –1.49°C. The feed flow rates used for the solutions were respectively 0.65 l/min, 0.5 l/min, 0.43 l/min and 0.45 l/min. Using these process parameters, the total mass of ice after 180 min is 1.75kg for a feed of 7.5% brix and 1.46kg for a feed of 12% brix. From theoretical analysis, the expected total mass of ice produced is 2.6kg for the 7.5% brix and 1.97kg for the 12% brix solution. Therefore, the performance efficiency for the freeze concentrator is around 67% of theory for all three sugar solutions.

Base on the heat balance analysis, the overall thermal performance of this freeze concentration process for the three different feed concentrations shows that about 80% of the refrigeration load is loss. If these losses were not considered, about 87% of the cooling energy transferred is for sensible heat removal and 13% for ice formation.

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

REKABENTUK, PEMBINAAN DAN UJIAN KEATAS ALAT PEMEKATAN JUS SECARA SEJUK BEKU

Oleh

ROSNAH BT HAJI SHAMSUDIN

November 2000

Pengerusi: **Hishamuddin bin Jamaludin, MSc**

Fakulti: **Kejuruteraan**

Perkara utama di dalam kajian ini merangkumi aspek rekabentuk, pembinaan dan prestasi alat pemekat sejuk beku. Proses merekabentuk pemekat sejuk beku ini terbahagi kepada tiga bahagian utama iaitu turus tegak penukar haba, *auger* untuk mengikis ais yang terbentuk dan unit penyejuk untuk menurunkan suhu bawah takat beku.

Pada asasnya, proses pemekat sejuk beku ini bermula dengan pembentukan ais di penukar haba. Ais akan dikikis oleh *auger* yang berpusing ke saluran keluar dan dikumpulkan ditakungan yang disediakan. Ini menghasilkan produk yang mempunyai kepekatan yang lebih tinggi dari kepekatan bahan suap.

Keupayaan pemekat sejuk beku ini diuji dengan menggunakan larutan gula pada tiga kepekatan yang berlainan iaitu 7.5%, 10.3% dan 12.0% brix disamping jus nenas (11.4% brix) juga digunakan. Titik penyejukbekuan pada 7.5%, 10.3% dan

12.0% brix serta jus nenas masing-masing ialah –1.07°C, -1.15°C, –1.49°C dan –1.49°C.

Bagi kepekatan bahan suap 7.5%, 10.3% dan 12% brix dan jus nenas masing-masing kadar alir yang digunakan adalah 0.65 l/min, 0.5 l/min, 0.43 l/min dan 0.45 l/min.

Dengan parameter proses tersebut dan masa operasi 180 min jumlah berat ais yang diperolehi ialah 1.75kg dengan suapan 7.5% brix manakala 1.46kg dengan suapan 12% brix. Manakala secara pengiraan pula berat ais yang patut diperolehi ialah 2.6kg di 7.5% brix dan 1.97kg di 12% brix. Maka pada keseluruhannya keputusan menunjukan keupayaan kecekapan alat pemekat sejuk beku ini adalah lebih kurang 67% berbanding teori bagi ketiga-tiga kepekatan yang berlainan.

Berdasarkan analisis keseimbangan haba, prestasi terma menunjukan alat pemekat sejuk beku tersebut mengalami kehilangan haba sebanyak 80%. Jika kehilangan ini diabaikan, didapati 87% haba efektif digunakan bagi pemindahan haba pelakuran manakala selebihnya digunakan bagi pembentukan ais.

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Last but not least, I would like to express my utmost appreciation and love to my family especially my parent and my husband Azmi bin Peryatin for their cares, encouragement and understanding during my studies.

I certify that an Examination Committee met on 22nd November 2000 to conduct the final examination of Rosnah bt Haji Shamsudin on her Master of Science thesis entitled "Design, Construction and Testing of Freeze Concentrator" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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Date: 0 9 DEC 2000

This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfilment of the requirements for the degree of Master of Science.

KAMIS AWANG, Ph.D.
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Date: 11 JAN 2001

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

(Rosnah bt Haji Shamsudin)

Date: 0 9 DEC 2000

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LISTS OF ABBREVIATIONS

λ = Latent heat of fusion, kJ/kg
ρ_I = Density of ice, kg/m^3
ρ_s = Density of the solution, kg/m^3
ρ = Density, kg/m^3
ρ_a = Density of ash, kg/m^3
ρ_c = Density of carbohydrate, kg/m^3
ρ_F = Density of fat, kg/m^3
ρ_p = Density of protein, kg/m^3
ρ_w = Density of water, kg/m^3
ρ_c = Density of coolant, kg/m^3
ρ_j = Density of juice, kg/m^3
μ_c = Viscosity of coolant, kg/ms
μ_j = Viscosity of juice, kg/ms
ΔT_{ln} = logarithmic mean temperature difference, °C
A = logarithmic mean area heat transferring wall, m^2
A_c = cross – sectional area of tube heat exchanger, m^2
A_j = cross – sectional area of product, m^2
C = Concentrate, % brix
C_p = Specific heat, kJ/kg K
$C_{p,c}$ = Specific heat of coolant, kJ/kg K
$C_{p,j}$ = Specific heat of juice, kJ/kg K
$C_{p,a}$ = Specific heat of ash, kJ/kg K
$C_{p,c}$ = Specific heat of carbohydrate, kJ/kg K
$C_{p,F}$ = Specific heat of fat, kJ/kg K
$C_{p,p}$ = Specific heat of protein, kJ/kg K
$C_{p,w}$ = Specific heat of water, kJ/kg K
C_b = Bulk solute concentration
C_i = Interfacial concentration of solute
$(dC/dT)_{eq}$ = Freezing point depression constant
d_c = diameter of the tube heat exchanger, m
d_s = shaft diameter, m
d_t = inside diameter product cylinder, m
F_c = Flow rate of coolant, l/min
F_j = Flow rate of juice, l/min
h = Heat transfer coefficient, W/m^{2}K
h_c = Heat transfer coefficient of coolant, W/m^{2}K
h_j = Heat transfer coefficient at product (juice) side, W/m^{2}K
I = Ice, % brix
I = Current flow, A
k_c = Mass transfer coefficients
k = Thermal conductivity, W/mK
k_c = Thermal conductivity of coolant, W/mK
k_j = Thermal conductivity of juice, W/mK
k_a = Thermal conductivity of ash, W/mK
k_c = Thermal conductivity of carbohydrate, W/mK
k_F = Thermal conductivity of fat, W/mK
k_p = Thermal conductivity of protein, W/mK

k_w = Thermal conductivity of water, W/mK
ℓ = Length of heat transfer, m
m_A = Fraction of product moisture content
m_s = Fraction of products solids
m_a = Mass fraction of ash
m_c = Mass fraction of carbohydrate
m_F = Mass fraction of fat
m_p = Mass fraction of protein
m_w = Mass fraction of water
$.m_c$ = Mass flow rate of coolant, kg/sec
$.m_j$ = Mass flow rate of juice, kg/sec
$.m_{ice}$ = Mass flow rate of ice, kg/sec
M_A = Molecular weight of water
M_s = Molecular weight of product solids
n = number of rows of scraper blades
N = Shaft speed, rev/sec
Nu = Nusselt – number
P = Power required to rotate blade, W
Pr = Prandtl – number
Q_c = Total heat transfer from coolant to solution, W
Q_{sol} = Rate of sensible heat transfer to solution, W
Q_{ice} = Rate of heat transfer in the formation of ice, W
Q_{loss} = Rate of heat loss, W
Re = Reynolds – number
R_g = Universal gas constant
R_e = External radius heat transferring wall, m
R_i = Internal radius heat transferring wall, m
T_A = Actual freezing point, ° C
T_{Ao} = Freezing point of pure water, ° C
T_b = Bulk temperature, ° C
T_1 = Outlet temperature of ice, ° C
$T_2, T_{c,out}$ = Surface temperature of coolant out, ° C
$T_3, T_{j,in}$ = Inlet temperature of feed/juice, ° C
$T_4, T_{c,in}$ = Surface temperature of coolant in, ° C
T_5 = Inlet temperature of coolant tank, ° C
$T_6, T_{j,out}$ = Outlet temperature of feed/juice, ° C
$\bar{T}$ = Average temperature of coolant, ° C
U = Overall heat transfer coefficient, W/m^2K
υ_c = Velocity of coolant, m/sec
υ_j = Average flow velocity, m/sec
V = Voltage flow, V
x_B = Mole fraction of water
X_A = Mole fraction of water within the product

CHAPTER I

INTRODUCTION

Water is removed from foods to provide microbiological stability, to reduce deteriorative chemical reactions and to reduce storage and transportation costs. This water can be removed by heating and cooling. Distillation or evaporation by addition of heat is not the only method of removing water from fruit juices. Water may also be removed from juices by freezing out as crystals of solid ice. This process is called **Freeze Concentration**. In freeze concentration water is partly separated from the aqueous solution by crystallization and the ice is then separated from the concentrated liquid phase (Maguer et al, 1986).

Freeze concentration has been used to concentrate fruit juices, coffee extract, vinegar, beer, wine, pickling brines and liquid smoke, to recover potable water from brackish water and sea water and to concentrate toxic wastes and paper-mill black liquor.

1.1 The Advantages of Freeze Concentration

Conventional method of concentration processes such as evaporation or distillation has some disadvantages. During evaporation of water from aqueous solutions the volatile responsible for aroma and fragrance are also driven off. While equipment to condense and return this desirable volatile has been developed, the recombined end product is inferior to the starting material.

Distillation requires the addition of heat This heat brings about a breakdown in the chemical structure of the food liquid causing a change in flavour, a diminution of vitamin contents and other nutritive property Even when essence recovery systems are employed and all volatile condensed and added back to the concentrate, the original flavor is not restored (Norman et al, 1977) Other methods for the removal of water are reverse osmosis (hyper filtration) and ultra filtration In these methods water and some solutes in a solution are selectively removed through a semi permeable membrane (Fellows, 1988)

Freeze concentration involves the concentration of an aqueous solution by partial freezing and subsequent separation of the resulting ice crystals It is considered to be one of the most advantageous concentration processes because of the many positive characteristics related with its application It is capable of concentrating various comestible liquids without appreciable change in flavor, aroma, color or nutritive value The concentrate contains almost all the original amounts of solutes present in the liquid food (Tasoula et al, 1990) These low temperatures are needed to preserve texture of the food by retarding chemical changes, by retarding the action of food enzymes and by eliminating the growth of microorganisms capable of growth near or below 0 °C

1.2 The Disadvantages of Freeze Concentration

Freeze concentration process involves the fractional crystallization of ice from liquid foods Although this process is traditional (it has long been used to enhance the alcoholic content of cider) it has found only limited and spasmodic use in the food industry (Brennan et al, 1990).

Freeze concentration disadvantages are threefold. Firstly, the degree of concentration achievable is limited. Secondly, suspended matter in the feed can serve as heterogeneous nuclei. This can lead to concentrates, which are pale in colour and lacking in flavour since pigmented particles, and suspended essential oils are removed from the system at the center of ice crystals. Thirdly, the process has proven to be more expensive than evaporation. The capital cost of the plant is not the only feature contributing to the economics of the process, since the fraction of the soluble solids of the feed material discharged with the ice crystals or the melted water rather than with the concentrate is highly important (Brennan et al, 1990).

Process improvements over the years have been directed to cost-effective reduction of this loss. The problem to be solved is the generation of ice crystals substantially free of inclusions of mother liquor and the separation of these from the system clean of any adhering concentrate (Brennan et al, 1990).

1.3 Objective

The objectives of this project are:

1. To design and construct a Freeze Concentrator for the concentration of sugar solution.
2. To test and evaluate the performance of the Freeze Concentrator in freezing sugar solution at various feed concentrations.
3. To determine the rate of ice formation with a shell in tube heat exchanger that is provided with an auger scraper at various feed concentrations.

CHAPTER II

LITERATURE REVIEW

2.1 Definitions

In freeze concentration water is first partly segregated from the aqueous solution by crystallization and thereupon the ice is separated from the concentrated liquid phase. The crystallization and separation operations can be performed in the same apparatus. In actual practice, however, these operations are performed in two separate apparatus (Thijssen, 1974b). These are the crystallizer and separator.

Schwartzberg (1972), described freeze concentration as a process in which: a) cooling is used to selectively freeze solvent from solutions; b) solute concentrations in residual solution consequently increase; and c) concentrated solution and solidified solvent are separated when desired concentrations are reached.

Freeze concentration involves partial freezing of the product and removal of the pure ice crystals, thus leaving behind all of the non aqueous constituents in diminished quantity of water The major limitations of the process include relatively high cost, difficulties in effective separation of ice crystals without loss of food solids and a relatively low upper limit on total solid concentration in the product (Fennema, 1975).

2.2 Freeze Concentration of Foods

The basic components of a freeze concentration system is as shown in figure 2.1. A refrigeration unit is required to provide the driving force for ice crystallization.

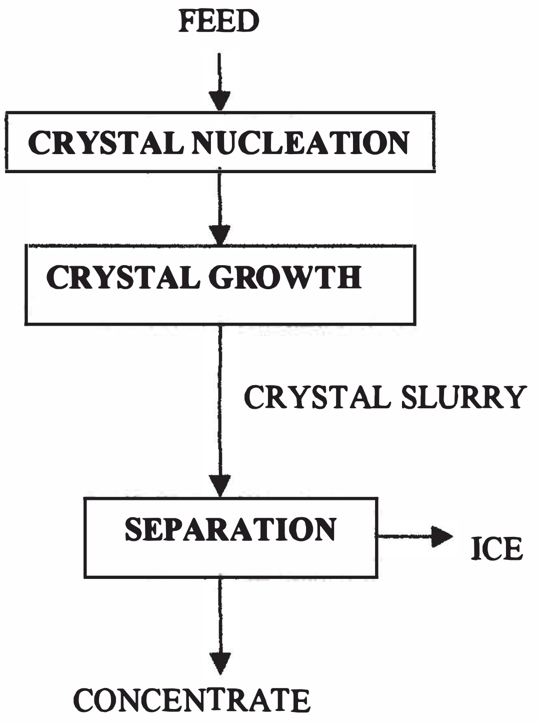

Figure 2.1: Diagram of a basic freeze concentration process

2.2.1 The Freezing Point

The *freezing point* of a liquid is that temperature at which the liquid is in equilibrium with the solid. The freezing point of a solution is lower than that of a pure solvent. The freezing point of food is lower than that of pure water. The addition of a nonvolatile solute (sugar) to water lowers the vapor pressure of the water solution of sugar and the freezing point of the water solution will be lower than that of pure water. Because of the high content of water in most foods, most of them freezes solidly at temperatures between 0° and –3° C (Norman et al, 1978).

The actual freezing process in food products is somewhat more complex than freezing of pure water. Figure 2.2 compares the freezing curves of water with aqueous solution containing a solute. In water, the temperature decreases as heat is removed from the system until the freezing point is reached. After a small amount of supercooling, the temperature remains constant as the latent heat is removed from the water system. Following this latent heat removal, the temperature decreases again as energy is removed (Heldman, 1979).

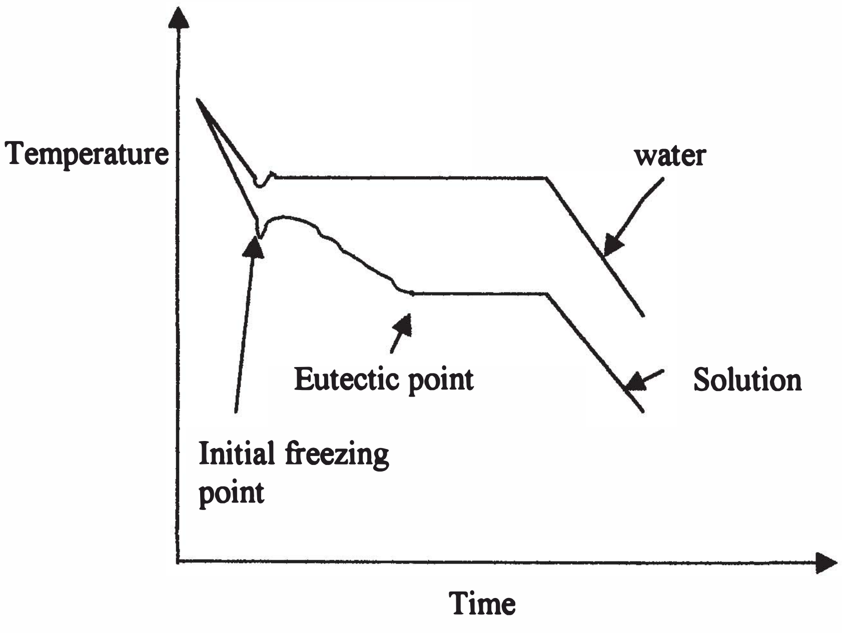

Figure 2.2: Comparison of freezing curves for pure water and an aqueous solution containing one solute