

PROCESS DESIGN AND SIMULATION FOR
EXTRACTION OF MILK FAT USING
LIQUID PROPANE

By

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CHAPTER I

INTRODUCTION AND OBJECTIVES

1.1 INTRODUCTION

Milk is a major part of the human diet. It provides 15 essential nutrients for normal growth and all of the 9 essential amino acids through Casein, a protein found only in milk. Also milk lipids contain anti-carcinogenic agents. The role of milk in traditional diet varies widely in different regions of the world. The consumption of milk per person varies from high in North America and Europe to a low in Asia. Per capita consumption of milk and milk products in the USA, in 2006 was 83.9 litres (1). However there has been a steady decline in milk consumption by an average American from 29 gallons a year in 1975 to 24 gallons a year in 1988 and further to 20.86 gallons a year in 2008 (2).

Though the sales of whole milk decreased by 0.3 % in 2008, that of reduced, low and fat-free milk demonstrated an increase of 0.4 %. Whole milk which once held 70 % of the market, was down to less than one third with about 43 % held by reduced milk and 17 % by fat-free milk in 2000 (3). This decline in milk fat consumption posed a considerable problem to the dairy industry. Limited functional properties and dietary values of the fat are considered to be the reasons for reduced consumption. This necessitates the need to modify the properties of milk fat according to specific applications.

Milk fat utilization can be increased by separating the fat into fractions with different physical and chemical properties. Several techniques including crystallization, solvent fractionation and supercritical fluid extraction have been studied. However solvent extraction was observed to give certain advantages like low temperature operation, high purity end products, pollution-free operation and tailored separation based on control of operating conditions.

Supercritical (SC) CO₂ has been extensively used for milk fat fractionation (4). However SC CO₂ failed to remove complex lipids unless an organic co-solvent was used. Also supercritical extraction with CO₂ involves very high extractor pressure. This work examines extraction using liquid propane, at ambient temperature.

1.2 RESEARCH OBJECTIVES

Milk fat fractionation using solvents other than SC CO₂ has not been studied extensively. Propane has been widely used for oil extraction due to its high selectivity for oils. Very little literature is available regarding the use of propane for milk fat fractionation. Yoon et al (1995) (5) studied propane extraction at near supercritical conditions. Hence it is useful to check the feasibility of propane as solvent at sub critical conditions for fractionation of milk fat.

Very few models have been generated for extraction of fat. Martinho *et al.* (2008) (6) and Patrachari (2008) (7) simulated the extraction of soybean oil but no model has ever been generated to simulate the extraction of milk fat as per available literature. Hence it is

necessary to model and simulate the process of milk fat fractionation using propane to determine the feasibility.

The two primary purposes of this study were to design a process to extract milk fat and develop a method to use Aspen PlusTM for liquid-liquid extraction. The objectives of this work include

1. Analyze the literature available on milk fat fractionation using different solvents and propane in particular.
2. Develop a method to use Aspen PlusTM to model liquid-liquid extraction.
3. Develop steady-state process models to represent liquid-liquid extraction of milk fat including dehydration and solvent recovery operations using Aspen PlusTM.
4. Determine the optimum process conditions to maximize the yield of extraction.
5. Perform sensitivity analysis to determine the effects of temperature, pressure and solvent flow-rate on the extraction process.

CHAPTER II

LITRATURE REVIEW

2.1 MILK COMPOSITION

Milk is a complex fluid consisting of several systems. The composition of bovine milk depends on various factors like species, breed, geographical location, stage of lactation and diet of the animal. The market product is fairly constant in composition because of pooling and standardization of fat. In general bovine milk contains 3 to 5 % fat with the rest being water, proteins, carbohydrates, minerals and inorganics. The general composition of bovine milk is given in table 1.

Component	Weight Percent
Water	88.32
Fat	3.25
Carbohydrates	4.52
Protein	3.22
Minerals(ash)	0.69

TABLE 1 COMPOSITION OF BOVINE MILK (8)

2.1.1 Milk Fat

Bovine milk lipid has a very complex fatty acid composition. It has been found to contain around 406 fatty acids, most of which contain less than 1 % of the total lipid. Only 12 fatty acids have been found to be greater than 1 % in composition and around 15 to 20 fatty acids constitute 90 % of the milk fat (9).

Fatty acid	Weight Percent
Butyric	2.31
Caproic	2.31
Caprylic	2.31
Capric	2.31
Lauric	2.37
Myristic	9.13
Pentadecanoic	1.5
Palmitic	25.51
Palmitoleic	2.0
Stearic	11.23
Oleic	25
Linoleic	3.7
Linolenic	2.31

TABLE 2 FATTY ACID COMPOSITION OF BOVINE MILK

The fatty acid composition in milk fat changes throughout the lactation period. In the early stages of lactation, the fat contains mostly long chain fatty acids like palmitic, stearic and linoleic acids where as in the later stages, the fat tends to be short chain fatty acids like butyric, caproic and capric acids. This is because of the fact that in the early

stages of lactation, the animal's energy comes from body stores and so limited fatty acids are available for synthesis of milk fat. Though these changes in the fat composition do not impact the nutritional values of milk significantly, they affect the processing characteristics of milk products. The general composition of bovine milk fat is given in table 2.

The lipids in bovine milk contain several classes. The fatty acid molecules attach themselves to a glycerol molecule and form compounds called mono, di, or triglycerides. Triglycerides form the major part of milk lipids. Other fat compounds include phospholipids and sterols. The sterols are cholesterol, cholesteryl ester and hydrocarbons. Trace amounts of carotenoids, waxes and lipoproteins are also present as minor lipids. Average composition of milk lipids is given in table 3. Though the composition of major lipids is well described, minor lipids are yet to be studied precisely.

Lipid Class	Weight percent
Cholesterol	0.42
1,2-Diacylglycerol	0.28-0.59
Free fatty acids	0.1-0.44
Hydrocarbons	Trace
Monoacylglycerol	0.16-0.38
Phospholipids	0.2-1.00
Triacylglycerol (TG)	97-98

TABLE 3 LIPID CLASSES IN BOVINE MILK (10)

2.1.1.1 Triacylglycerols

Triglyceride (TG) is a three carbon backbone made of three fatty acids attached to a glycerol. The composition of TG depends on the kind of fatty acids present. The accepted structure of TG is 1-random-2-random-3-random distribution. Figure 1 shows the structure of TG, where R refers to a fatty acid. The Rs can all be the same or a combination of different molecules. The sn-1 position is occupied mainly by palmitic acid (34 %) or oleic acid (30 %), the sn-2 position by palmitic acid (32.3 %) and the sn-3 position by butyric acid (35.4 %) (11). As bovine milk lipids contain more than 400 fatty acids, the possible number of triglycerides is 64 million but as only around 10 fatty acids are present in amounts greater than 1 %, theoretically it would be 1000 TG species if all the fatty acids were randomly distributed. The rheological properties, melting points and crystallization behavior of milk fat depend on the structure of TG. (10).

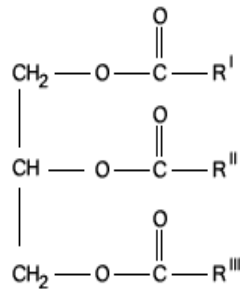


FIGURE 1 STRUCTURE OF TRIGLYCERIDE

2.1.1.2 Phospholipids

Phospholipids account for about 1 % of milk fat and are important components of cell membranes. They have the same type of structure as TG except that they have a phosphate group at the 3rd position on the carbon backbone. They are a source of long

chain poly unsaturated fatty acids. The principle classes of phospholipids are Phosphatidylcholine, Phosphatidylethanolamine, Sphingomyelin and Gangliosides.

2.1.1.3 Sterols

Cholesterol is the major sterol in milk lipids. It is present in amounts ranging from 10 to 20 mg/dl (10). Other sterols like lanosterol, cholesteryl ester are present in trace amounts.

2.1.1.4 Free Fatty acids

The free fatty acid composition of milk fat is very complex. Free fatty acids are the fatty acids unassociated to any other components like glycerol or phosphate groups. It ranges from saturated to highly unsaturated fatty acids. The chain length in fatty acids ranges from 4 to 24 carbons. Milk fat contains about 65 % saturated, 30 % monounsaturated and 5 % poly unsaturated free fatty acids. The saturated fatty acids present in large amounts are palmitic, myristic and stearic acids (10).

2.1.2 Carbohydrates

Bovine milk consists of about 4.7 % carbohydrates that is predominantly lactose with trace amounts of monosaccharides and oligosaccharides.

2.1.3 Proteins

Bovine milk consists of about 3.2 % proteins which include all the 9 essential amino acids required by humans. Approximately 82 % of milk protein is casein, the rest being whey protein. Processing temperatures up to 161°F cause no damage to nutritional and

functional properties of both casein and whey proteins (12). Enzymatic action and exposure to light are major causes for milk protein degradation.

2.1.4 Minerals and Inorganics

Minerals are very important to the human body as they help in oxygen transport, water balance maintenance and bone formation. Milk is a good source of calcium, magnesium, phosphorous, potassium, selenium and zinc. Trace amounts of copper, iron, manganese and sodium are also present.

2.2 PHYSICAL PROPERTIES OF MILK

The melting properties of milk vary over a wide range from -40°F to 104°F as they depend on the melting properties of individual fatty acids and their arrangement on TG molecule. TG in milk is generally in the form of globules surrounded by membranes of protein and phospholipids. These membranes stabilize the globules in the water phase of milk. The milk fat globules range from 1 µm to over 10 µm in size (13).

Degradation of milk fat is caused by enzyme action, exposure to light and oxidation. Enzymatic action that causes degradation is called lypolysis and such enzymes are called lipases. These enzymes remove the fatty acids from triglyceride and the resultant build up of free fatty acids causes undesirable rancid flavors in milk. Lypolysis is avoided by pasteurization which is usually carried out at temperatures around 145°F. Exposure to light causes protein degradation which produces a characteristic off flavor. This can be minimized by using opaque containers (12).

Oxidation of phospholipids also produces off-flavor. This may be stimulated by high heat treatments. Higher heat treatments like Ultra High treatment disrupts and destabilizes the globules resulting in their coagulation. The preferred pasteurization method is High Temperature Short Period (HTSP) which is carried out at 160°F. At this temperature the functional and nutritional values of fat are not destroyed (14). The physical properties of milk fat and lactose are given in table 4.

2.3 NUTRITIONAL VALUES OF MILK FAT

2.3.1 Fatty acids

All fatty acids are not equal from the nutritional perspective. Saturated fatty acids are generally known to increase cholesterol levels which lead to Coronary Heart Disease (CHD) but the effect depends on the varied contribution by individual fatty acids. Short chain fatty acids like butyric, caproic, capric and caprylic acids are metabolized in a way that they either have no effect or lower blood cholesterol levels where as long chain fatty acids like myristic, palmitic and lauric acids raise the levels of Low Density Lipoprotein (LDL), the atherogenic lipoproteins that carry 65 to 70 % of blood cholesterol levels and are generally known as bad cholesterol. Myristic acid is known to have the worst effect on the cholesterol levels (15).

Kratz *et al.* (2002) (16) examined that when the saturated fatty acids are replaced by mono or poly unsaturated fatty acids, smaller LDL particles which exhibit three fold greater risk than larger LDL particles, decreased in number leading to reduced CHD risk.

Nestel *et al.* (1999) (17) studied the possibility of saturated fatty acids inducing other CHD risk factors like endothelial and other arterial dysfunction, insulin resistance, myocardial arrhythmogenicity and hypertension. On the other hand it was found that milk fat contains cardioprotective components like sphingolipids, conjugated linoleic acid, 13-methyltetradecanoic acid and ether lipids (18).

Butyric acid is an important anticancer agent. It has numerous molecular and genetic effects. Butyric acid is a major source of energy for colorectal epithelium. It has anti-colon cancer properties. Butyric acid inhibits the growth of tumor and promotes differentiation (19). Also it may inhibit mammary tumorigenesis (20). It modulates the expression of suppressor genes and oncogenes (21). Butyric acid is also known to have anti-inflammatory and immune suppression properties.

2.3.2 Trans Fatty Acids

Conjugated Linoleic Acid (CLA) is the major trans fatty acid in milk. Its isomer Cis 9, trans 11 named Rumenic Acid is nutritionally the most important (22). Numerous health benefits are associated with CLA. It is found to have antimutagenic, anti-cancer, anti-atherogenic, fat regulating, immune modulating and growth regulating effects (23-25). CLA improved hyperinsulinemia and glucose tolerance in a pre diabetic Zucker Diabetic Fatty rat according to Houseknecht *et al.* (1998) (26). Also combination of two isomers of CLA reduced body weight and this explains the wide spread use of CLA supplements as an aid to weight loss (11).

Component	Molecular formula	Molecular weight	Form	Color	Specific gravity	Melting point, F	Boiling point, F	Solubility in 100 parts water
Lactose	$C_{12}H_{22}O_{11}$	360.31	rhombic	colorless	1.525	395.6	decomposes	17
Linoleic acid	$C_{18}H_{32}O_2$	280.44	Oil	yellow	0.903	49.1	444.2-446	insoluble
Oleic	$C_{18}H_{34}O_2$	282.45	needles	colorless	0.85478	57.2	545-546.8	insoluble
Palmitic acid	$C_{16}H_{32}O_2$	256.42	plates	colorless	0.84970	145.4	520.7	insoluble
Myristic acid	$C_{14}H_{28}O_2$	228.36	leaflets	colorless	0.85370	134.6	482.9	insoluble
Stearic acid	$C_{18}H_{36}O_2$	284.47	monoclinic	-----	0.84769	158	555.8	insoluble
Lauric acid	$C_{12}H_{24}O_2$	200.31	needles	colorless	0.86950	118.4	437	insoluble
Capric acid	$C_{10}H_{20}O_2$	172.26	needles	colorless	0.88987	88.7	514.4 - 518	0.003
Caproic acid	$C_6H_{12}O_2$	116.16	oily liquid	-----	0.92220	29.3	395.6	1.120
Caprylic acid	$C_8H_{16}O_2$	144.21	leaflets	colorless	0.91020	60.8	459.5	0.0715
Linolenic acid	$C_{18}H_{30}O_2$	278.43	----	-----	-----	-----	-----	-----

TABLE 4 PHYSICAL PROPERTIES OF MILK FAT (27)

2.3.3 Phospholipids

Phospholipids are found to possess anti oxidative, anti microbial and anti-viral properties (28). Kingsley (2006) (29) observed that supplementation of Phosphatidylserine (PS) in humans altered neuroendocrine function and positively influenced the muscular soreness. Also oral supplementation of PS with soybean improved exercise capacity during high intensity cycling. McDaniel *et al.* (2003) (30) examined that PS attenuates neuronal effects of aging in animals and also restores memory on a variety of tasks.

Phosphatidylcholine (PC) supports liver recovery from toxic chemical attack or viral damage according to Kidd (2002) (31). It is also believed to be a source of choline which is an essential nutrient for humans. PC reduces life threatening necrotizing enterocolitis in hospitalized preterm infants (32) and protects gastrointestinal mucosa from toxic attack (33).

Spingomyelin inhibits colon carcinogenesis. It is found to reduce the intestinal absorption of cholesterol (34). Spingolipids can act as cellular binding sites and may also have protective capability against bacterial toxins (35). However they may be related to the development of Alzheimer's (18).

2.4 TREATMENT OF MILK FAT

Milk fat varies widely in nutritional aspects and health benefits. Hence it is essential to modify the milk fat before intake. The following processes are employed for the treatment of milk fat.

1. Hydrogenation
2. Interesterification
3. Enzymatic treatment
4. Mixture with other fats and acids
5. Fractionation.

As discussed earlier, milk fat is a mixture of different fatty acids with differing physical properties and so it can be separated into fractions of different chemical compositions and physical properties. This fact makes fractionation advantageous over the other methods (36). Also hydrogenation and interesterification destroy natural flavor and modify the functional and nutritional properties of the milk fat. Distillation, crystallization, solvent extraction and super critical fluid extraction are the methods used to carry out fractionation.

Crystallization at different temperatures has been studied by deMan (1968) (37) and Fjaervol (1970) (38). The separation of uncrystallized fat becomes difficult and the variation in composition remains in the range of natural variation.

2.5 EXTRACTION

Extraction is a mass transfer operation to separate components distributed between two insoluble phases of a mixture. When both the phases are liquid it is known as liquid-liquid extraction (LLE) and when one of them is solid it is called leaching. The mass transfer operations fall into two categories, direct and indirect. Direct operations like distillation, evaporation and zone refining are those which do not utilize added substances

and indirect operations like liquid-liquid extraction, extractive distillation, absorption and adsorption involve a foreign substance like solvent. Liquid-liquid extraction uses an immiscible solvent to remove a key component from a multi component stream. It is preferred over other extraction operations in the following cases

1. Direct methods are expensive
2. Relative volatility is poor
3. Boiling points of liquids are close
4. High vacuum is required
5. Fractional crystallization is to be used
6. Substances are heat sensitive
7. Mixtures form azeotropes

The solution containing the components to be separated is the feed sent to the extraction process. The major component in this solution is called the feed solvent and the other components are called solutes. The immiscible liquid added to the extraction process to separate the components is called the solvent. This solvent strips the solutes from the feed by absorbing them. Of the two streams produced after extraction, the solvent rich stream containing the desired solute is called extract and the residual stream rich in feed solvent is called raffinate. The yield and economics of the extraction process strongly depend on the solvent used, operating conditions, mode of operation and equipment.

2.5.1 Selection of Solvent

The desirable characteristics of a solvent for liquid-liquid extraction (27) are

1. Selectivity: This is defined as the ability of the solvent to preferentially dissolve more of one component than the other.
2. Distribution coefficient: This is also known as Partition ratio and is defined as the ratio of a certain component in extract phase to raffinate phase. The partition ratio of solute should be fairly large.
3. Recoverability: This stands for the ease of separation of the solvent from the extract and raffinate phases.
4. Capacity: This represents the amount of solute loaded per weight of solvent in the extract at the solubility limit.

Other factors include toxicity, flammability, interfacial tension, density, viscosity, boiling point, availability and cost.

2.5.2 Operating Conditions

The yield and selectivity of the extraction process depend on the temperature of the process. The effect of pressure on the extraction process is negligible and hence operating pressure is usually governed by vapor pressure considerations. High temperatures may sometimes be used to minimize mass transfer resistance. Solubility, selectivity and vapor pressure are other conditions to be considered.

2.5.3 Equipment

Various extractors are available for liquid-liquid extraction. They are broadly classified into four categories

1. Mixers and settlers: A battery of mixers and settlers is used when intense mixing and high residence time are required. The mixers can be either

static or agitated. These are generally used for the extraction of metal from the ore.

2. Centrifugal extractor: This type of extractor is used in the pharmaceutical industry. The high speed rotating machine is usually mono-stage but multistage extractor is also available.
3. Counter-current column extractors: Commercially these are the most popular extractors. They are either static or agitated. Many types of these agitators are available.

Selection of equipment is affected by various factors. The following table summarizes the characteristics of different extractors

Property	Mixers & Settlers	Centrifugal extractor	Static column	Agitated column
Number of stages	Low	Low	Moderate	High
Flow rate	High	Low	Moderate	Moderate
Residence time	Very High	Very Low	Moderate	Moderate
Interfacial tension	Moderate to High	Low to Moderate	Low to Moderate	Moderate to High
Viscosity	Low to High	Low to Moderate	Low to Moderate	Low to High
Density Difference	Low to High	Low to Moderate	Low to Moderate	Low to High
Floor space	High	Moderate	Low	Low

TABLE 5 CHARACTERISTICS OF VARIOUS EXTRACTORS (39)

2.6 MILK EXTRACTION PROCESS

The extraction process of milk involves four major steps

1. Dehydration
2. Extraction
3. Solvent recovery
4. Desolventization

In the dehydration process, the volume of milk is reduced by removal of water. As discussed earlier milk contains 88 % water, removing this is important for effective extraction. Concentration of milk protects it against microbial actions. This concentration process is carried out by various methods like reverse osmosis, evaporation, sublimation and freeze drying. Evaporation is the most commonly employed method.

The extraction process is carried out either in cross-current or counter-current mode. In the cross-current mode, both liquid phases are mixed with droplets of one phase suspended in the other but they are separated before leaving each stage. It is used for low capacity multi product batch operations like pharmaceutical and agro chemical processes. It is practical and economical for washing and neutralization operations and also offers good flexibility. In the counter-current extraction scheme, feed (F) and solvent (S) enter the extractor from opposite ends and pass each other counter currently. This mode is used for large volume operations and for an effective use of the solvent. The configurations are explained in figure 2. The solvent is then recovered from the extract stream leaving the liquid-liquid contactor. Evaporation, prevaporation, distillation and flash separation are

the methods generally used to recover solvent. The recovered solvent is recycled for reuse. The raffinate containing non fat milk and solvent is desolventized by steam stripping or evaporation.

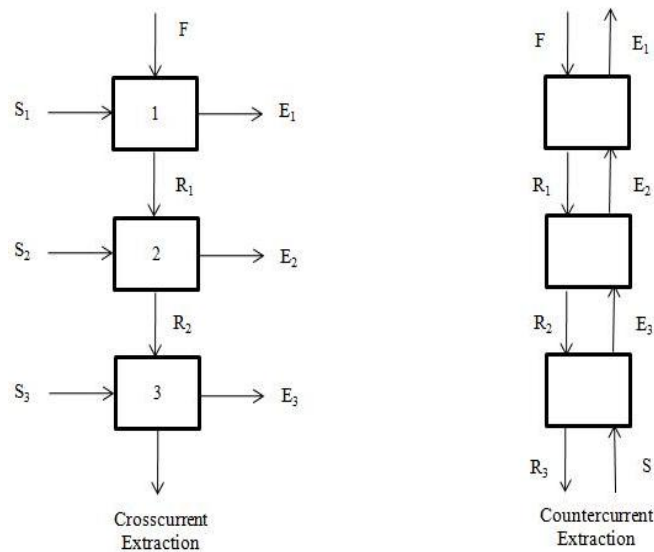


FIGURE 2 MODES OF EXTRACTION

Various methods with different solvents and solvent mixtures have been studied for extraction of lipids from dairy products. Hubbard *et al.* (1977) (40) disclosed the use of ethyl ether, a 2:1 solution of chloroform and methanol for extraction of fatty acids from food products. The samples were digested with HCl before extracting with the solvent. It was determined that though the solvent was effective in extracting lipids, it tends to leave harmful residues making the food unsuitable for consumption.

Melnick (1971) (41) examined extraction of lipids from egg yolk using non-polar solvents like hexane, cyclohexane, heptane and trichloroethylene. These solvents were found to preserve the functional properties of the remaining proteins and leave little

residuals. But polar solvents were said to give better extraction yields. Use of mixture of both polar and non-polar solvents like ethanol-ether was suggested in this patent work.

Carbon dioxide at super critical conditions has been the most popular solvent for lipid extraction from milk fat. Arul *et al.* (1987) (42) carried out extraction of triglycerides from milk fat with super critical CO₂ at temperatures of 122-178°F and pressures 1450-4350 psia. The extraction yielded eight fractions in which the first two were liquids, next three were intermediate in consistency and the last three were solids. As the melting points of the fractions increased, the percentage of long chain fatty acids increased but that of short and medium chain fatty acids decreased. It was reported in this work that milk fat was extracted and separated into fractions rich in short chain fatty acids and fractions rich in long chain fatty acids. The desired fatty acid fraction could then be mixed with the non fat milk.

Shishikura *et al.* (1986) (43) removed 75 % of the triglycerides using a single pass SC CO₂ unit operating at temperatures 104 to 140°F and pressures 1856.5 to 3596.9 psia.

Bhaskar *et al.* (1993) (4) examined fractionation of anhydrous milk fat with SC CO₂ in a system consisting of a packed column and series of separation vessels. Extraction was carried out in a continuous counter-current mode at temperatures in the range 104-167°F, pressures 349 – 493 psia and a solvent to feed ratio of 62. Temperature and pressure were varied from vessel to vessel to enhance precipitation of Triglycerides. The extraction yield attained was 78 % and as observed by Arul *et al* (42), the short and medium chain fatty acids increased in percentage from the first to fifth fraction while long chain fatty acids decreased.

Dimethyl ether (DME) was used as near SC and SC solvent by Fletcher *et al.* (2008) (44). A specialist dairy stream called beta serum consisting of 60 % fat was used as feed. The feed was mixed with DME and passed through a static mixer and then through a series of flash separators at a temperature 121.7°F and pressure 14.5 - 87 psia for 6-12 hours. The raffinate was sometimes reprocessed. High throughput (~ 90 % lipid extraction) was achieved at high feed loadings at the expense of decreased extraction efficiency. DME was found to extract all the complex lipids but not neutral lipids. To remove neutral lipids, feed was first extracted with SC CO₂ and then with DME. However some protein denaturation was observed.

Yoon *et al.* (1995) (5) carried out extraction of milk fat using SC ethylene and liquid propane and compared the results with that of SC CO₂. For SC ethylene the temperature range was 104-140°F and pressures 2175-3625 psia. Solubility of milk fat in SC ethylene was found to be greater than that in SC CO₂ at the same operating conditions. They concluded that SC ethylene gives slightly greater extraction of fat than SC CO₂.

Liquid propane was sent through a column packed with beads coated with milk fat at temperatures from 86 to 194°F and pressures from 500 to 800 psia for at least 2 hours. At these conditions solubility of milk fat in propane was 9 to 10 % (w/w). Below 171.5°F and 400 psia milk fat and propane were found to be miscible. The amount of fat solubilized in propane was higher than that in SC ethylene and significantly higher than SC CO₂. Though propane was found to have greater solvent capability, fractionation of fat in propane was low compared to SC ethylene and SC CO₂.

Non-polar solvents for milk fat extraction may give good extraction yield as most of the milk fat components are non polar

2.7 PROPANE

Propane is a natural organic solvent. It satisfies most of the characteristics of an ideal solvent. The only disadvantages associated with propane are flammability and cost. Table 6 lists the properties of propane. Propane is known to selectively extract lipids from fat due to the fact that the structure of lipids is more similar to propane than any of the other solvents used (5). Also Propane has been extensively used as a major solvent for extraction of fatty acids from vegetable oils (45).

The extraction of sesame seed oil with propane was found to be much faster than with SC CO₂ and also it was determined to be a better solvent than CO₂ for that extraction (46). Propane was more capable than SC CO₂ with lower solvent to feed ratio for extraction of seed oil (47). The maximal yield of extraction of rice bran lipid obtained with propane was higher than that with SC CO₂ (48).

Properties	Propane
Molar mass (g/mol)	44.1
Density (kg/m ³)	583
Melting Point (K)	85.5(305.7°F)
Boiling Point (K)	231.1(-43.7°F)
Flash Point (K)	169.1(-155.3°F)
Auto ignition temperature (K)	813.1(1036.3°F)
Explosive Limits	2.4-9.7 %
Solubility in water @ 273K,g/L	0.04
Critical Temperature (K)	369.52 (205.5°F)
Critical Pressure (bar)	42.49 (717.8 psia)
Vapor Pressure, psia @ 70 ⁰ F	124.9

TABLE 6 PROPERTIES OF PROPANE

CHAPTER III

USING ASPEN PLUS™ FOR LLE

Aspen Plus™ is a powerful process simulation tool. It is extensively used to design a new process, troubleshoot an existing process unit or optimize operations in a process. Using Aspen Plus™, the behavior of the process can be predicted based on basic relations like mass and energy balances and phase equilibrium. This chapter acts as a manual for modeling a liquid-liquid extraction process using Aspen Plus™. Each step involved in developing a steady state model using Aspen Plus™ is explained in detail.

3.1 GLOBAL SPECIFICATIONS

3.1.1 Units of measurement

The input and output units are specified on the *Setup | Global | Specifications* sheet (figure 5). The units of any property in each set can be modified on the *Setup | Units-sets* form according to convenience.

3.1.2 Stream Class

The default option for stream class is conventional, stated as *CONVEN* in Aspen Plus™. This stream class is used when either no solids are present in the simulation or the present solids are electrolyte salts. This stream class is used with *MIXED* sub-stream and is specified on *Setup | Specifications | Global* form or *Setup | Streamclass | Global* form as

shown in figure 3. The stream class *MIXNC* is used when the simulation contains non-conventional solids without particle size distribution. For solids with particle size distribution, *MIXNCPSD* is used. For conventional solids with and without particle size distribution, *MIXCISLD* and *MIXCIPSD* are used respectively. When both conventional and non conventional solids are present *MIXCINC* and *MCINCPSD* are used while the latter is used for particle size distribution.

For each stream class, a respective sub stream is selected. The stream class specification form is shown in figure 3 and the sub stream selection form is shown in figure 4.

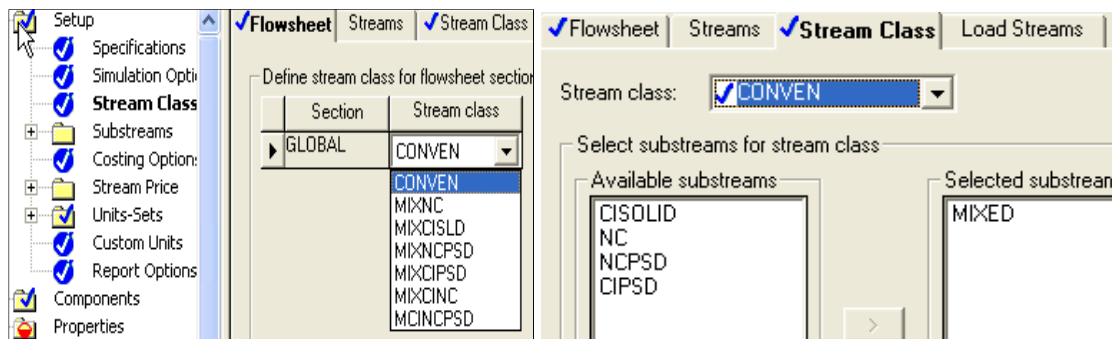


FIGURE 3 STREAM CLASS SPECIFICATION FIGURE 4 SUB-STREAM SPECIFICATION

The flow basis can be mass or mole. The valid phases are vapor-liquid-liquid or liquid-only for solvent extraction. It is always a good practice to set the valid phase option to vapor-liquid-liquid when not sure about the presence of vapor. Either yes, no or dirty water is selected for the free water option on the *Setup | Specifications | Global* form (figure 5). Detailed description of free water method is given in section 3.3.2.

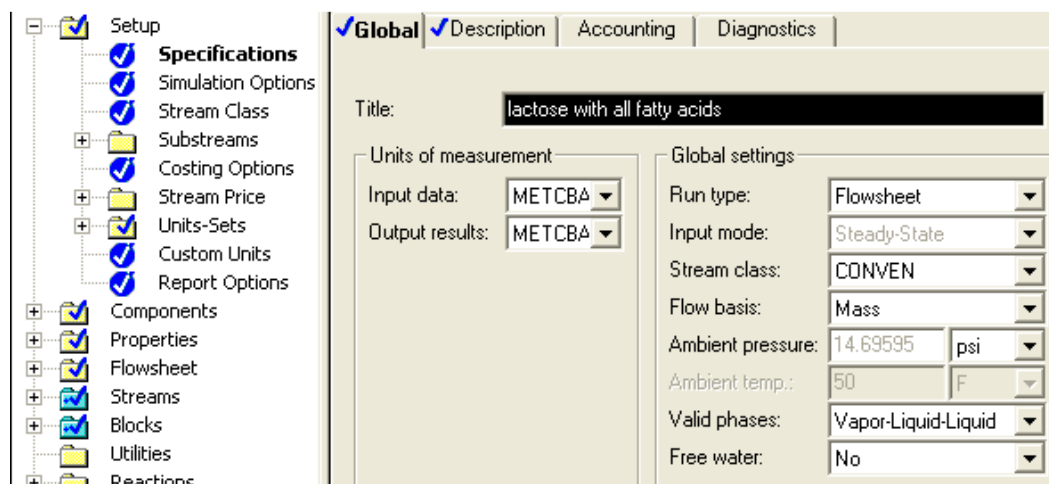


FIGURE 5 GLOBAL SPECIFICATION SHEET

3.1.3 Flash options

In the *Setup* | *Simulation options* | *Flash convergence* form (figure 6), the upper and lower limits for temperature and pressure are usually left as default values. They can be changed if needed.

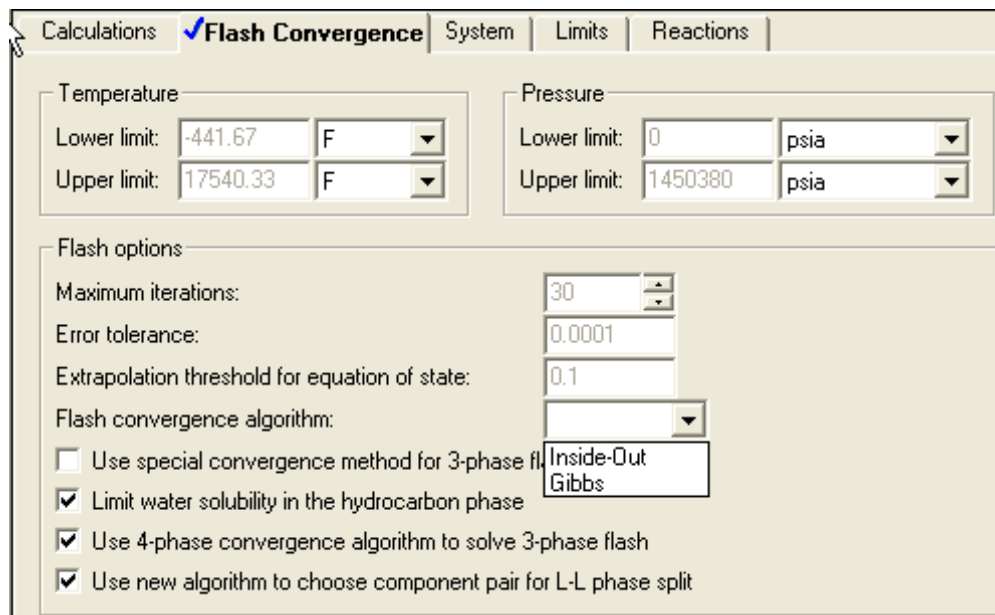


FIGURE 6 FLASH CONVERGENCE SPECIFICATION SHEET

3.1.3.1 Extrapolation Threshold for equation of state

In Aspen Plus™, at specified composition, temperature and pressure, all equations of state use a root finder to calculate molar volume. During the iterative calculation of the molar volume, at certain specifications, a real root may not exist. In such a situation, Aspen Plus™ extrapolates the root and gives an estimate such that the unit converges. The extrapolation threshold controls this estimation of the root. As the threshold value decreases the chances of occurrence of extrapolation decrease.

3.1.3.2 Flash convergence algorithm

Aspen Plus™ has two algorithms for flash convergence. Either Inside-out or Gibbs can be used for sequential modular calculations. The default algorithm used in Aspen Plus™ is Inside-out for all flash calculations except three-phase true-species electrolyte calculations. Gibbs algorithm is preferred for three-phase calculations and when convergence problems arise with the inside-out algorithm.

3.1.3.3 Water solubility

The option *Limit water solubility for hydrocarbon phase* allows Aspen Plus™ to override the water solubility calculated by the specified physical property method and limit the water solubility in the organic phase. This option is used when water is highly soluble in the organic phase. Checking or unchecking the box for this option makes no difference in the results if the water solubility is not significant.

3.1.3.4 4-phase convergence algorithm

The 4-phase convergence algorithm is more rigorous than the 3-phase and so is preferred for three phase calculations. Vapor, liquid and liquid are the three phases while water being the fourth phase in this algorithm.

All the Flash options are given on the *Simulation options* | *Flash Convergence* form as shown in the figure 6.

3.2 COMPONENTS

3.2.1 Component types

In general, all the components are conventional. Non-conventional components are not pure chemical species but are complex mixtures. They cannot be characterized by molecular weight (49). The properties of conventional components are already present in the built-in databanks of Aspen Plus™ but those of non-conventional components are calculated. Methods for calculating enthalpy, density and component attributers are specified in the *Properties* | *Advanced* | *NC Props* form. The types of components are shown in figure 7.

Component ID	Type	Comp
LACTOSE	Conventional	SUCR
WATER	Conventional	
PROPANE	Solid	
OLEIC-01	Nonconventional	
CAPROIC	Pseudocomponent	
PALMITIC	Assay	
STEARIC	Blend	
	Hypothetical liquid	
	Polymer	
	Oligomer	
	Segment	

FIGURE 7 COMPONENT TYPES

User Defined Component Wizard

Welcome to the User Defined Component Wizard, the quickest way to enter properties for User Defined Component. This wizard will lead you through the steps to enter the required physical properties for the User Defined Component based on its type.

Component ID: LACTOSE Type: Conventional

Formula: C12H22O11

FIGURE 8 USER DEFINED WIZARD

3.2.2 Adding new component

A new component not present in the built-in databanks is specified using the user-defined component wizard shown in the figure 8. Molecular weight, chemical formula and structure of the component are required while other properties like normal boiling point and specific gravity are specified if available, in the conventional component basic data form as shown in figure 10. Structure of the component is specified using any of the following three methods:

1. Meaning Oriented Interface (MOI) file obtained in databases like NIST (50) is imported.
2. Molecule connectivity is specified.
3. The structure is drawn using respective buttons (figure 9).

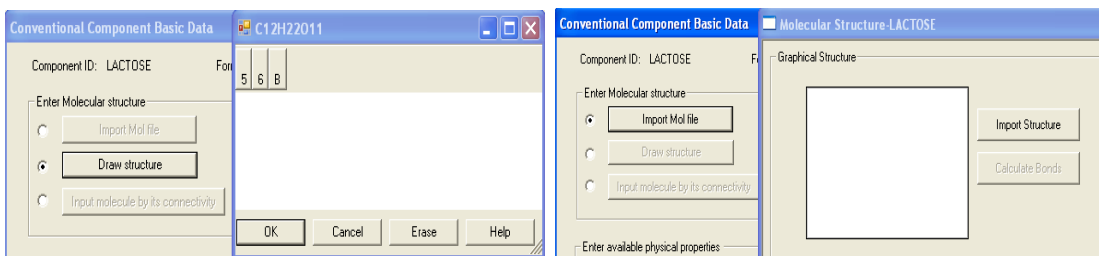


FIGURE 9 STRUCTURE SPECIFICATION SHEETS

To draw a structure on the draw structure form, single click of the left mouse button produces a carbon atom, right click on the atom erases it and double click allows the user to change the carbon atom to another atom. Connecting the atoms produces a single bond and clicking on the bond allows the user to change it to double or triple bond. The wizard is shown in figure 9. On the conventional component additional data form, if available, further information is specified and the properties are evaluated using NIST Thermodynamic Data Engine (TDE). TDE evaluation form is shown in figure 11.

Conventional Component Basic Data [?] [X]

Component ID: LACTOSE Formula: C₁₂H₂₂O₁₁

Enter Molecular structure

Import Mol file
 Draw structure Structure was drawn.
 Input molecule by its connectivity

Enter available physical properties

Molecular weight:
 Normal boiling point:
 Specific gravity at 60 deg F:
 Standard enthalpy of formation:
 Standard Gibbs energy of formation:

Click Next> to continue or Finish to save the above properties and exit.

FIGURE 10 REQUIRED PROPERTY DATA SHEET

Conventional Component Additional Data [?] [X]

Click buttons 1 to 5 to enter additional properties or data.

Liquid density
 Vapor pressure data
 Extended Antoine vapor pressure coefficients
 Ideal gas heat capacity data
 Ideal gas heat capacity polynomial coefficients

Evaluate using TDE
 Estimate using Aspen Plus property estimate system

FIGURE 11 ADDITIONAL PROPERTY DATA SHEET

3.3 PROPERTIES

3.3.1 Property method

The property method for the process is specified *on properties | Specifications | Global* form as shown in the figure 12. The general methods used to model the Liquid-Liquid equilibrium in Aspen Plus™ are given in the table 7. A detailed description about the property models used for LLE is given in Chapter 4.

Activity Coefficient option sets	Equation of state option sets
UNIF-LL	SR-POLAR
UNIF-DMD	SRK
UNIF-LBY	PRMHV2
NRTL	PRWS
UNIQUAC	RKSMHV2
-----	RKSWS
-----	PSRK

TABLE 7 THERMODYNAMIC MODELS FOR LLE (49)

The property method selection assistant is available on the *Specifications | Global* form as a button as shown in figure 12 by an arrow. A list of suitable property methods is generated based on either component type or process type. Once component type is selected, further options, like pressure conditions, are selected to obtain a suggested property method.

Once the model is selected, the parameters can be viewed by selecting the option *Retrieve parameter results* from the Tools tab. They can be seen on the *Properties | Parameters | Results* form. The binary interaction parameters are generated from databanks like LLE-ASPEN, LLE-LIT, and VLE-IG.

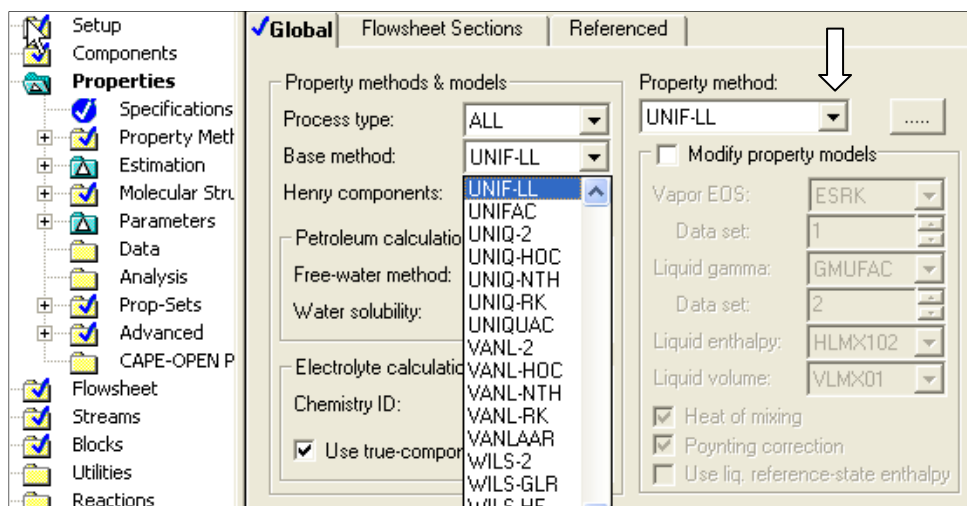


FIGURE 12 PROPERTY METHOD SPECIFICATION SHEET

If regressed data is available for any component, then they can be specified on the *Properties* | *Parameters* | *Binary interaction* form as shown in figure 14. If no data is available and the parameters are not obtained from the databank, then parameters for a similar component are used or they may be estimated using *Properties* | *Estimation* | *Input* form shown in figure 13. UNIF-LL method is preferred if the binary parameters are estimated.

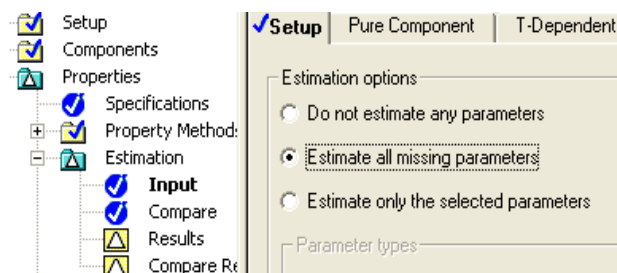


FIGURE 13 PARAMETER ESTIMATION FORM

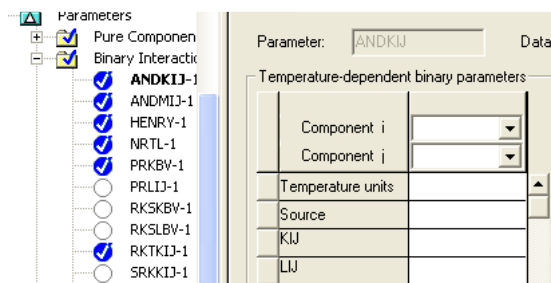


FIGURE 14 BINARY PARAMETERS SPECIFICATION

3.3.2 Free-Water Method

In three-phase calculations, Aspen PlusTM provides an option *Free water* which can be set to *YES*, *NO* or *DIRTY WATER* as shown in figure 15. Setting this option to *YES* allows Aspen PlusTM to assume and treat the second liquid phase in the vapor-liquid-liquid phase system as pure water. Free water is the pure water layer in the two liquid phases. Solubility of organics in water is treated as zero. This option is generally used when solubility of organic phase in water is insignificant like refining applications. Any of the four water solubility methods (0 1 2 3) is used to calculate the solubility of water in the organic phase. If a free-water method is specified, a free-water property method is used for stream properties; else a primary property method is used. When a free-water method is used, either water is specified as a component or water basis is selected as dry in the *Properties | Prop-Set | qualifiers* sheet (49). Free water calculations are rigorous, except for the assumption of pure water, but faster than the three phase calculations and also require less property data interpretation. Free water is generally used for a water-hydrocarbon system with insignificant solubility of water in the hydrocarbon phase.

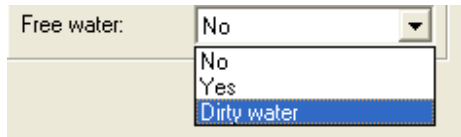


FIGURE 15 FREE-WATER SPECIFICATION

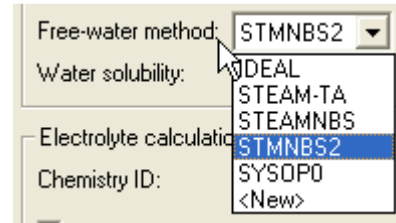


FIGURE 16 FREE-WATER PROPERTY METHOD

The K-value of Free-water phase is calculated as

$$K_W^* = \varphi_W^{*,l} / \varphi_W^v \dots\dots\dots (3.1)$$

K_W^* is the free-water phase K-value

$\varphi_W^{*,l}$ is fugacity coefficient of pure liquid phase calculated using Free-water property method.

φ_W^v is fugacity coefficient of water in vapor phase mixture calculated using a primary property method.

The K-value of water in the organic phase is calculated as

$$Y_W \varphi_W^V = X_W \gamma_W \varphi_W^{*L} \dots\dots\dots (3.2)$$

But
$$K_W = Y_W / X_W \dots\dots\dots (3.3)$$

Hence
$$K_W^* = \gamma_W \varphi_W^{*,l} / \varphi_W^v \dots\dots\dots (3.4)$$

γ_W is the activity coefficient of water in the organic phase and is calculated using one of the four solu-water options (0 1 2 3).

Y_W is the vapor fraction of water in the organic phase

X_W is the liquid fraction of water in the organic phase.

The Free-water property method is specified in *the Properties|Specifications|Global* sheet shown in figure 16. *STEAM-TA* and *STEAMNBS / STEAMNBS2* are the steam table property methods available in Aspen. *STEAM-TA* is used as the default free-water property method in Aspen Plus TM. Although all the models are accurate enough for process calculations, convergence problems arise with *STEAM-TA* as the correlations used in it fail to provide continuity at the boundaries. This problem does not arise in *STEAMNBS* and hence *STEAMNBS* is preferred in certain applications. It is used with SRK, BWRS, MXBONNEL and GRAYSON 2 property methods as it extrapolates better. This feature is necessary as the properties of water are frequently requested out of the range of the steam tables.

Both *STEAMNBS* and *STEAMNBS2* use the same equation but a different root search method. The convergence problem may arise with *STEAMNBS2* also. The thermodynamic model, transport model and range of temperature and pressure for each property method are given in table 8.

Property Method	Thermodynamic property model	Transport property model	Range of Temperature(K) for use	Maximum Pressure(bar) for use
STEAM-TA	ASME1967	IAPS	273.15-1073	1000
STEAMNBS/STEAMNBS2	NBS/NRC1984	IAPS	273.15-2000	10000

TABLE 8 PROPERTY MODEL FOR FREE WATER METHODS (49)

With a free water property method, flash 2 with a water decant stream is used. This block has phase qualifiers and performs a vapor-liquid-free water flash where as Flash 3

performs only rigorous 3-phase (vapor-liquid-liquid) calculations. A Decanter can also be used with a Liquid-Free water phase qualifier, but does not work well for free water calculations and gives inconsistent temperatures. Hence flash 2 is usually preferred. If the decant water stream is specified then the pure water phase is placed in the decant stream else the water is mixed with organic phase. When the phase qualifier is changed from vapor-liquid –free water to vapor-liquid-liquid, free water specification is ignored and dirty water calculations are carried out. For Distillation models the free water calculations are carried out only in the condenser except for RadFrac, MultiFrac and PetroFrac.

3.3.3 Dirty water

The dirty water option is used in applications involving concern over the solubility of organic phase in water though the solubility is not significant. This is mostly used in environmental studies. Dirty water method uses the special water solubility methods to calculate solubility of water in the organic phase like the free-water method. Besides this, it uses special method to calculate the amount of organics allowable in the water phase.

The k value is calculated as

$$K_i = \frac{\gamma_i \phi_i^l}{\phi_i^v} \dots\dots\dots (3.5)$$

Where

$$\gamma_i = \frac{1}{X_i^{sol}} \dots\dots\dots (3.6)$$

The solubility of component i in water, X_i^{sol} is calculated from the Hydrocarbon solubility model (HCSOL). Table 9 summarizes the general usage of the three options.

Free water options	General usage
YES	Refining applications
DIRTY WATER	Environmental study
NO	Water-higher alcohol systems

TABLE 9 APPLICATION OF FREE WATER OPTIONS

3.3.4 Water Solubility Methods

The various water solubility methods as shown in figure 17 are used to calculate the organic phase properties. These methods help determine the liquid fugacity in the organic phase. The k-value of water in the organic phase is calculated as

$$K_W = \gamma_W \phi_W^{*L} / \phi_W^V \dots \dots \dots (3.7)$$

γ_W , the activity coefficient of water in organic phase and ϕ_W^V , the fugacity coefficient of water in vapor phase mixture are now calculated accordingly as given in the table 10.

“The limiting solubility of water in the organic phase is the mole fraction weighted average of the solubilities of water in the individual organic species.”

$$X_W^{SOL} = \frac{\sum_i X_i \frac{X_W^{SOL}}{1 - X_W^{SOL}}}{1 + \sum_i X_i \frac{X_W^{SOL}}{1 - X_W^{SOL}}} \dots \dots \dots (3.8)$$

X_i is the water free mole fraction of the i^{th} organic species.

X_W^{SOL} is the mole fraction of soluble water in the i^{th} organic species calculated from water solubility method.

Solu-water option	γ_W calculation	ϕ_W^V calculation	Assumptions
0	$\gamma_W = \frac{1}{X_W^{sol}}$	Free water property method	Organic phase saturated Vapor phase mostly water
1	$\gamma_W = \frac{1}{X_W^{sol}}$	Primary property method	Organic phase saturated Vapor phase mostly organic
2	$\gamma_W = f(T, X_W)$ $\gamma_W = \frac{1}{X_W^{sol}}$ when $X_W = X_W^{sol}$	Primary property method	Not enough to form a second liquid phase
3	Primary property method	Primary property method	None
4	$\gamma_W = 1$	Primary property method	Water solubility is 1
5	$\gamma_W = f(T, X_W)$ $\gamma_W = \frac{1}{X_W^{sol}}$ when $X_W = X_W^{sol}$	Free water property method	Ideal vapor

TABLE 10 WATER SOLUBILITY METHODS ((49))

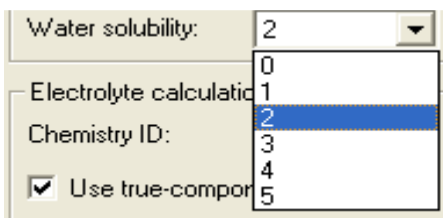


FIGURE 17 WATER SOLUBILITY METHOD SPECIFICATION

Methods 0, 1, 2 and 3 are generally used when free water option is specified. Method 2 is good for unsaturated systems. Method 4 is used in VLE systems when liquid phase is mostly water. For rigorous three-phase calculations, method 3 is used as default but only when regressed binary interaction parameters are available from liquid-liquid equilibrium data.

3.4 UNIT OPERATIONS

The Aspen Plus™ model library provides a wide range of unit operations. For liquid-liquid extraction processes, Aspen Plus™ provides flashes, decanter and extraction column. Flash 3, Decanter and Extract are exclusively used for the rigorous three-phase calculations.

3.4.1 FLASH 3

The unit operation, Flash 3 has two liquid and one vapor outlet streams. It performs vapor-liquid-liquid equilibrium calculations and is used to model a single stage separator. The vapor outlet stream may sometimes be zero in which case, a decanter is used. Flash conditions are specified on the *input | specifications* form. Outlet temperature, outlet pressure, heat duty and vapor fraction are the available options. Any two of the four can be given but heat duty and vapor fractions cannot be specified simultaneously. The component with the highest mole fraction in the second liquid phase is specified as the key component in 2nd liquid phase on the *Input | Key components* form shown in figure 18. If extract is the second liquid phase then solvent is specified as the key component. If nothing is specified, Aspen Plus™ considers the highest density phase as second liquid phase.

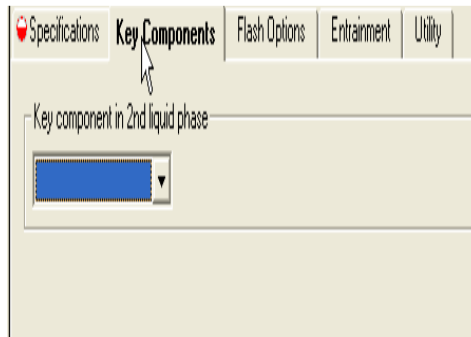


FIGURE 18 KEY COMPONENT SPECIFICATION FORM

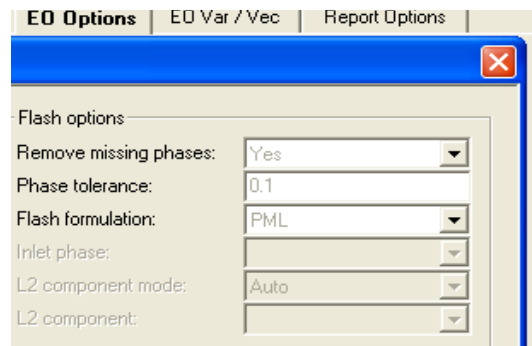


FIGURE 19 EO OPTIONS FORM

In the *Input* | *flash options* form estimates for temperature and pressure can be given but not required. Maximum iterations and error tolerance are changed if convergence problems come up in the simulation. The Block options form is used to override the global default values. Property methods can be changed for each block if necessary. If a property method different from the entire simulation is to be specified then it is done in the *Block options* | *Properties* form. Similarly the free-water property method and water solubility can be changed in the same form and flash convergence methods in the simulation options form. All block properties can be changed on the *Flash block* | *options* form shown in figure 20.

In the additional Equation oriented options form shown in figure 19, *Remove missing phase* option, when set to yes checks each flash of the block for missing phases. A phase

is considered missing if the extended phase fraction is beyond the range 0 to +1 by the amount of phase tolerance. The normal range of extended phase fraction is -1 to +2. This option is effectual in evading the problems related to sub-cooling or super-heating and also in reducing the size of the problem. For a 3-phase flash if both the liquid phase compositions are identical, then one phase is removed as missing phase. Also if an algorithm of any phase fails to converge then it is removed as missing phase. Automatic phase removal does not take place with a decanter and a flash 3. The value for the phase tolerance should not be too close to zero as it prevents systems which start as slightly sub-cooled or super-heated from entering the 2-phase region (49). L_2 component mode set to *auto* lets Aspen Plus™ selects the key component in the second liquid phase and set to *CompID* allows the user to specify the L_2 component. Specification of L_2 component augments the problem robustness. Component specified as L_2 component should have greater composition in L_2 than in L_1 .

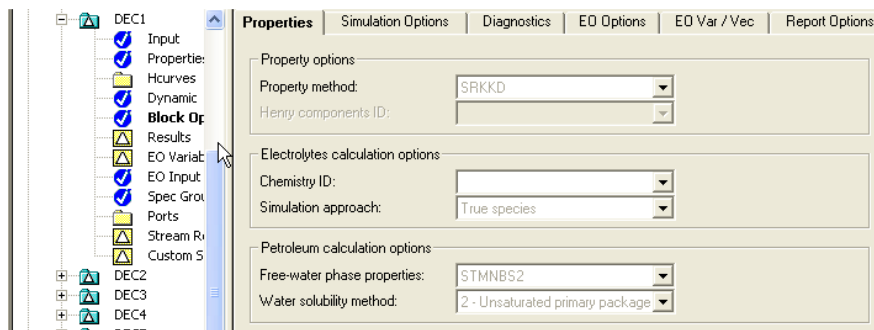


FIGURE 20 BLOCK OPTIONS SPECIFICATION FORM

3.4.2 Decanter

The Decanter just like the Flash 3 determines the thermal and phase conditions of the two liquid phases. It is a single stage separator with no vapor phase. Decanter operating conditions, pressure and temperature/heat duty are specified in *the Input | specifications*

form shown in figure 21. Key component is specified in a similar manner to that of flash 3. The phase split is determined by either of the two methods, equating component fugacities of the two liquid phases and minimizing Gibbs free energy. It is specified on *Input | Calculation options* form shown in figure 22. The option equating component fugacities allows users to select the source for liquid- liquid coefficients. By default they are calculated from the primary property method. This can be changed to a built-in correlation but requires the user to provide the coefficients or a user subroutine that requires a FORTRAN program. Minimizing Gibbs free energy method can be selected only when global physical property method and block property method are the same. The Decanter calculates solutions though the minimum Gibbs free energy is not achieved.

The screenshot shows a software interface with a tabbed menu at the top: 'Specifications' (selected), 'Calculation Options', 'Efficiency', 'Entrainment', and 'Utility'. The main area is titled 'Decanter specifications' and contains several input fields and lists:

- Pressure:** 140 psia
- Temperature:** 70 F
- Heat duty:** identify 2nd liquid phase
- Available components:** A list box containing LACTOSE, WATER, OLEIC-01, CAPROIC, PALMITIC, STEARIC, MYRISTIC, LINDLEIC, N-DOD-01, BUTYRIC, and LINDLENI.
- Key components:** A list box containing PROPANE.
- Key component threshold for 2nd liquid phase:** Component mole fraction: 0.5

FIGURE 21 DECANTER INPUT FORM

FIGURE 22 DECANter CALCULATION OPTIONS FORM

3.4.3 Extract

This unit operation is used especially for liquid-liquid equilibrium calculations. The number of equilibrium stages is specified in the setup specs sheet shown in figure 23. Temperature profile or heat duty profile is specified if an adiabatic column is not used. Estimates for pressure and temperature are provided. The pressure profile form is shown in figure 25. Block options are set similar to a flash 3 or a decanter.

Flash 2 with a decant water stream is used when a free water method is specified.

Stage	Pressure
1	140
*	

FIGURE 23 PRESSURE PROFILE

Stage	Temperature
1	20
2	22
*	

Stage	Heat duty
*	

FIGURE 24 EXTRACT INPUT FORM

CHAPTER IV

PROCESS DESIGN

This chapter deals with the detailed description of the process design. The input specifications for each form in Aspen PlusTM and the thermo dynamic model are explained in detail.

4.1 GLOBAL SPECIFICATION

4.1.1 Units of measurement

The input and output units were specified as METCBAR in the *Setup | Global | Specifications* sheet. METCBAR is the metric units with temperature in degree centigrade and pressure in bar. But the units of temperature and pressure were changed from “C” and “BAR” to “F” and “PSIA” respectively in the *Setup | Units-sets | METCBAR* form for convenience.

4.1.2 Stream Class

The stream class was specified as *CONVEN* as the simulation contains no solids. The sub-stream was specified as *MIXED*.

4.1.3 Simulation options

The run type was flow sheet and the flow basis was specified as mass. The valid phases are vapor-liquid-liquid as the presence of vapor in the simulation is uncertain. Free water was selected as *No* since the solubility of organic phase in water is significant in the simulation. If Free-water method is selected, Aspen PlusTM gives 100 % separation of water and organic phase which is not true in reality.

On the *Setup | Simulation Options | Flash Convergence* form the upper and lower limits for temperature and pressure were left as default values. Also *Maximum iterations*, *Error tolerance* and *Extrapolation threshold for equation of state* were specified as default values since no convergence problems were confronted. The Gibbs method was selected for the *Flash convergence algorithm* as the simulation involves three phase rigorous calculations. The option *Limit water solubility in the hydrocarbon phase* was not selected, as the solubility data calculated from the primary property method was preferred and the *4-phase convergence algorithm* was used to obtain better output.

4.2 COMPONENTS

All the components were specified as *Conventional*. Except for Lactose, Triglycerides and Phospholipids all the components of milk are found in Aspen PlusTM database. The composition of milk used in this simulation is shown in figure 26.

Component ID	Lactose
Type	Conventional
Formula	$C_{12}H_{22}O_{11}$
Molecular weight	342.3
Specific Gravity at 60 ⁰ F	1.525

TABLE 11 LACTOSE INPUT SPECIFICATION (27)

To define triglycerides and phospholipids as user defined components, pure component properties must be calculated using group contribution methods. As discussed earlier, the number of triglycerides can be over a thousand and predicting pure component parameters for all the triglycerides is a tedious job. Also, no experimental data is available to estimate binary interaction parameters. Hence free fatty acids are used to represent milk lipids.

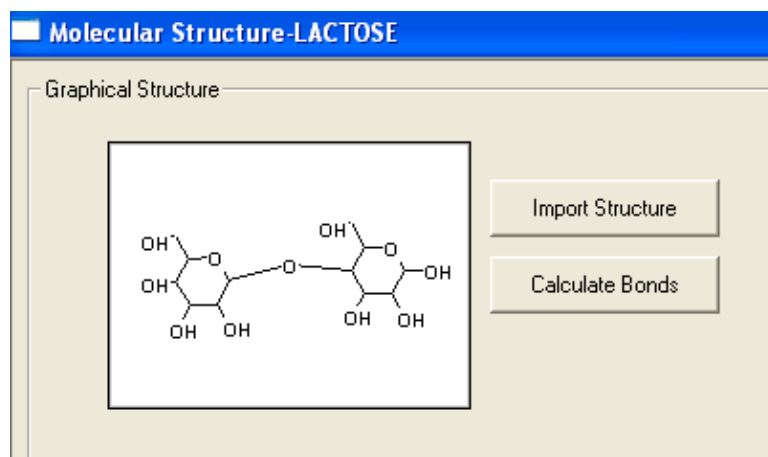


FIGURE 25 STRUCTURE OF LACTOSE

State variables		Composition	
Temperature	70 F	Mass-Frac	
Pressure	140 psia	Component	Value
Total flow:	Mass	LACTOSE	0.046
	1000 kg/min	WATER	0.93
Solvent:		PROPANE	0
		OLEIC-01	0.00075
		CAPROIC	0.00075
		PALMITIC	0.00897
		STEARIC	0.00365
		MYRISTIC	0.00297
		LINOLEIC	0.0012
		N-DOD-01	0.00077
		BUTYRIC	0.00075
		LINOLENI	0.00075
		CAPRIC	0.00075
		CAPRYLIC	0.00075

FIGURE 26 INPUT SPECIFICATION OF MILK STREAM

Lactose is specified through a user define wizard as described in chapter 3. The structure of Lactose (figure 25) is imported as a Meaning Oriented Interface (MOI) file downloaded from the NIST database (50). The properties of Lactose are evaluated from TDE after the input specifications (table 11) are entered in the wizard.

4.3 PROPERTIES

4.3.1 Thermodynamic property model

Uncertainty in physical properties can be the weakest link in simulating a process. Therefore a reliable technique for property estimation is a must. Accuracy of a process design greatly depends on the thermodynamic model used. Hence selection of thermodynamic model is the crucial step in process simulation. The selection of a property model is based on the following factors.

1. Type of mixture
2. Type of molecules

3. Operating conditions
4. Required properties
5. Level of accuracy
6. Availability of data.

Thermodynamic models are classified into two types, Equation-Of-State (EOS) and activity Coefficient models. In EOS models, all the properties are derived from EOS for both phases where as in activity coefficient models, vapor phase properties are derived from EOS and the liquid phase properties are derived from pure component properties.

The EOS method is generally used for systems containing non-polar or weakly polar components. For ideal or slightly non-ideal systems, thermodynamic properties can be predicted using minimum component data. It is most suitable to model hydrocarbon systems. The EOS method can be used for wide range of temperature and pressure from subcritical to supercritical regions. It is specifically used for systems operating at supercritical conditions. Activity coefficient model is used for complex mixtures at low pressures. This method does not predict well at or near the critical region. It is generally used for non-ideal liquids.

Aspen Plus TM has huge database containing numerous thermodynamic models that include solid and electrolyte models along with the classical thermodynamic models. Each thermodynamic model in Aspen Plus TM is based on either EOS or an activity coefficient model. There are around 80 EOS based models and 30 activity coefficient based thermodynamic models in the Aspen Plus TM database.

For vapor-liquid-liquid phase calculations, either the EOS or the activity coefficient method can be used. The models generally used for LLE are listed in table 7 in chapter 3. The activity coefficient method can model VLLE only at low pressures. For pressures higher than 10 atm, an EOS method is needed (49). Also for activity coefficient models, binary interaction parameters must be obtained either from a database or experimental data. For highly non-ideal multi-component systems at high pressures, flexible and predictive EOS should be used. A flexible EOS has advanced mixing rules which help model highly non-ideal and polar systems over a wide range of temperature and pressure.

Milk fractionation involves a highly non-ideal water-hydrocarbon system containing both polar and non-polar components at a pressure greater than 10 atm. Hence a flexible EOS method should be used. The system was modeled using the Soave-Redlich-Kwong-Kabadi-Danner (SRKKD) equation. This method is recommended for rigorous three phase calculation for water-hydrocarbon systems by the API Technical Data book (51).

SRKKD is an EOS method developed specially for water-hydrocarbon system (52). Kabadi and Danner proposed a two parameter mixing rule for the SRK equation of state. These mixing rules allow this method to model water-hydrocarbon immiscibility. The form of equation-of-state is

$$p = \frac{RT}{V_m + c - b} - \frac{a}{(V_m + c)(V_m + c + b)} \dots \dots \dots (4.1)$$

$$a = a_0 + a_{KD} \dots \dots \dots (4.2)$$

a_0 is the standard quadratic mixing term used in SRK

a_{KD} is the Kabadi-Danner term for water

$$a_{KD} = \sum_{i=1}^n a''_{wi} x_w^2 x_i \dots \dots \dots (4.3)$$

where $a''_{wi} = G_i \left[1 - \left(\frac{T}{T_{cw}} \right)^{0.8} \right] \dots \dots \dots (4.4)$

And $G_i = \sum g_i$

g_i is the group contribution parameter for groups constituting hydrocarbons.

4.3.2 Water Solubility method

Water solubility is calculated using method 2 (table 10, chapter 3). Though method 3 is the default option for three-phase systems, it is not opted due to lack of binary interaction parameters regressed from liquid-liquid equilibrium data. Method 2 is preferred to method 5 as the latter does not use a primary property method for vapor phase calculations. Method 1 is not preferred as it does not have correction for unsaturated systems like method 2.

4.3.3 Free Water property method

Though no free water is selected, *Free-water method* is changed from *STEAM-TA*, the default option, to *STEAMNBS*. As stated in earlier chapter, *STEAMNBS* is the preferred method for certain equations of state like SRK due to its extrapolation capability. Since SRK is one such equation of state (EOS) *STEAMNBS* is used instead of *STEAM-TA*.

4.4 PROCESS DESCRIPTION

The entire process was divided into 4 sub-sections as discussed in the literature review to facilitate better analysis of the whole process (figure 27). This section deals with the detailed explanation of the processes and unit operations in each sub-section.

The following assumptions were made for modeling the process

1. The solids in the milk were neglected.
2. Water content in milk was assumed to be 93 %.
3. Fatty acids represent the total milk fat.
4. The process is in steady-state.
5. Flow rate of milk is 1000 kg/min.

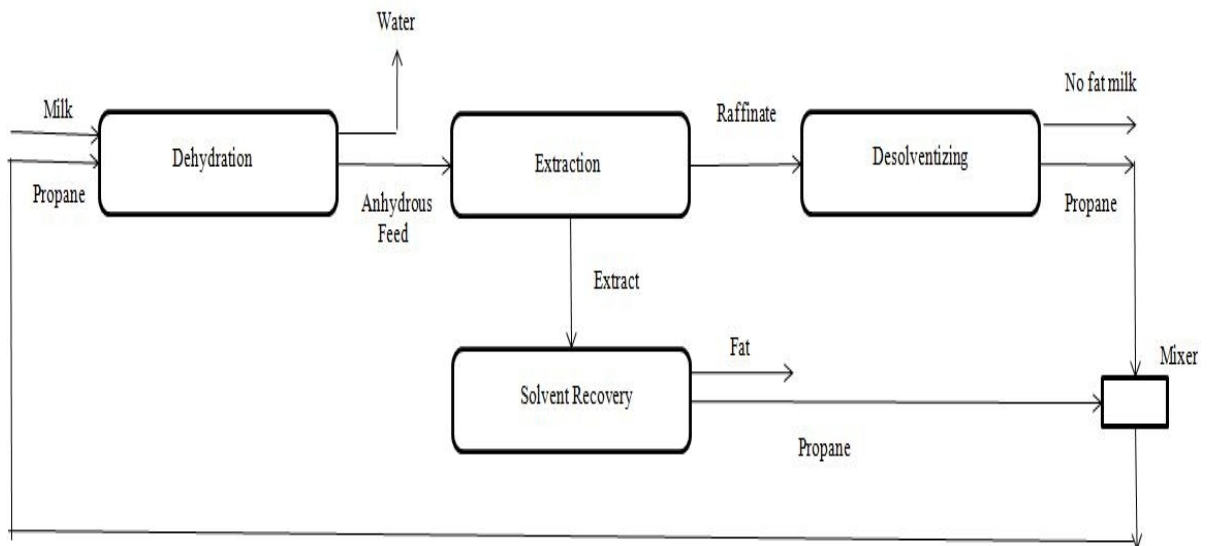


FIGURE 27 PROCESS FLOW-SHEET

4.4.1 Dehydration Unit

As a first step in the extraction process, milk is dehydrated to increase the concentration of milk fat. This dehydration (water removal) process is carried out through flash separation in a cross-current mode (figure 28). The separator is modeled as a Decanter. Milk and propane are fed to a mixer at 70°F and 140 psia with a mass flow rate of 1000 Kg/min and 1 Kg/min respectively. The feed flow-rate is assumed and the solvent flow-rate is based on a sensitivity analysis.

The input conditions of milk and propane are based on the vapor pressure considerations of propane (table 6, chapter2). At ambient temperature, pressure is selected such that both milk and propane are in the liquid phase with negligible vapor fraction. This operating condition avoids the cost of compressing propane. Ambient temperature is selected for the ease of handling propane.

A temperature change due to mixing occurs and changes the mixture temperature to 101°F. The mixture is then cooled back to the operating conditions of 70°F and 140 psia and then sent to a decanter (DEC 1). The key component is water to identify the first liquid phase. The key component to identify the second liquid phase in the decanter is propane and the phase equilibrium is calculated by equating the component fugacities of the two liquid phases. The specifications of the block property method and water solubility method are same as the global specifications. The option *Remove missing phases* is set to *yes* with a phase tolerance of 0.1.

In the cross-current operation, the raffinate (RAFF 1) from the decanter is again mixed with a fresh stream of propane (PROP 2) and sent to a second decanter (DEC 2) operating

at 30°F and 140 psia while the extract stream (EXT 1) from the first decanter (DEC 1) is cooled to 30°F and decanted (DEC 3) for further separation. The raffinate streams from the second (RAFF 2) and the third (RAFF 3) decanters are then heated and sent to a flash drum (FLSH 3) operating at 348°F and 140 psia to vaporize the solvent. At these conditions, water is sub-cooled liquid while propane is super-heated vapor. At 140 psia propane starts vaporizing from 77°F and this vaporization increases with increase in temperature. At 140 psia, the saturation temperature of water is 353°F and so any further increase in temperature results in vaporization of water along with propane. The operating conditions of FLSH 3 are chosen to maximize the purity of the vapor and the liquid streams. The vaporized solvent is mixed with the solvent recovered from the extraction process.

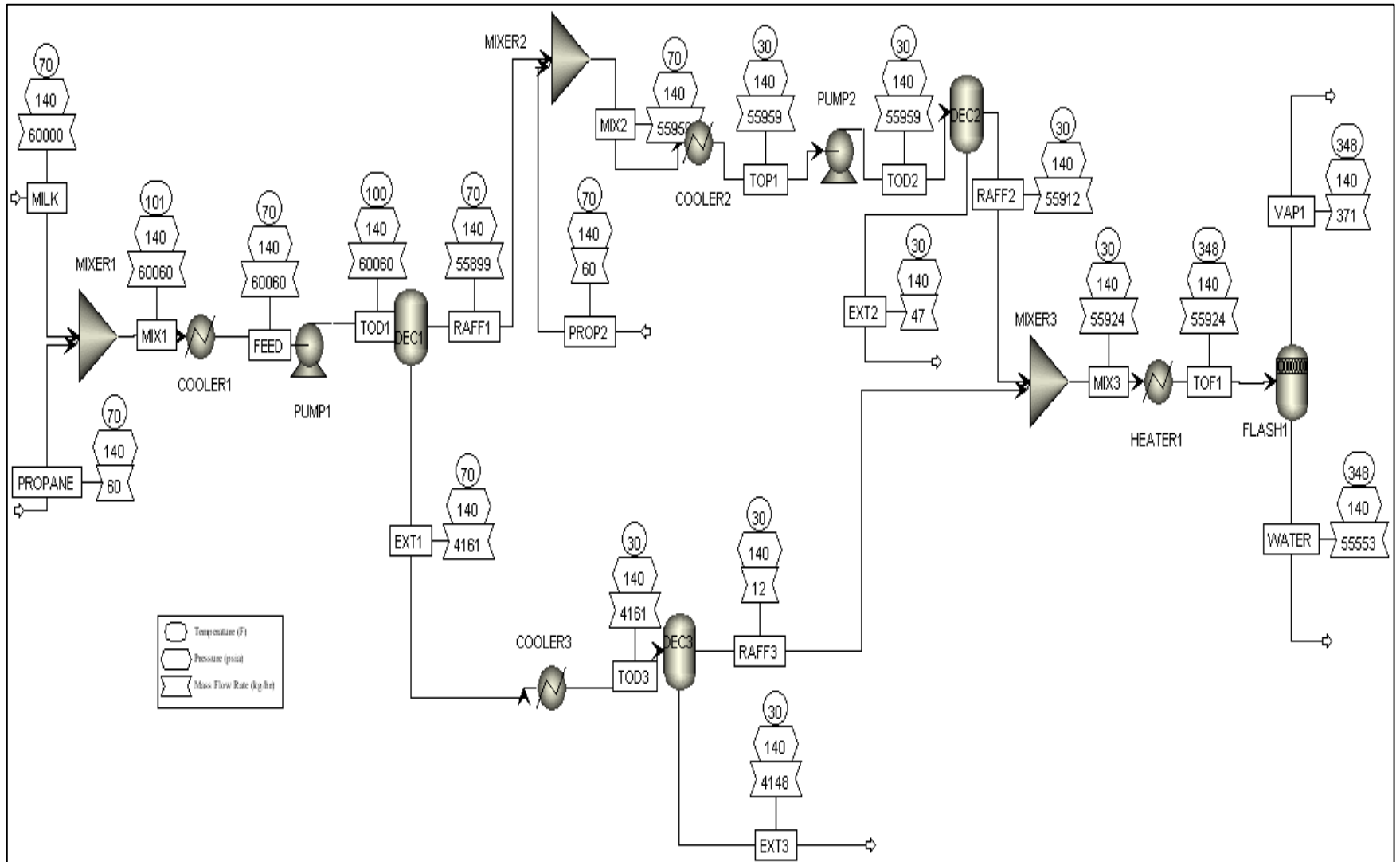


FIGURE 28 DEHYDRATION UNIT PROCESS FLOW SHEET

4.4.2 Extraction

The extract from DEC 3 is the input stream for the extraction process. This dehydrated milk stream is sent through a series of decanters and an extract column at varied temperatures but at a constant pressure of 140 psia. The pressure changers are modeled as pumps which take care of any line losses. The input stream (EXT 3) is sent to a decanter (DEC 4) operating at 50°F and 140 psia. In DEC4, the phase equilibrium composition is determined by minimizing the Gibbs free energy. The raffinate from this decanter, named as RAFF 4 in the flow sheet (figure 28) is mixed with a stream of propane (PROP 3) at 70°F, 140 psia and a mass flow rate of 0.1 kg/min.

This mixture (MIX 3) is then cooled and decanted at 30°F in DEC 5. The raffinate is fed to an extraction column (EXCT 1) at the top while propane at 60°F and 140 psia with mass flow rate of 25 kg/min is fed at the bottom of the tower. The extraction column is modeled as a two staged counter-current liquid-liquid contactor operating adiabatically at 60°F and 140 psia. The number of stages and operating conditions of the extract are determined from the optimization analysis explained in chapter 5.

The raffinate from the extract, BTM 1 is sent through series of two more decanters operating at 70°F and 100°F respectively for further extraction (figure 28). The raffinates from these two decanters (DEC 6 and DEC 7) and the EXCT 1 are combined to give a stream of milk with the least possible amount of fat (MIX 5). This stream is sent to desolventizing section to obtain solvent free non-fat milk. All the extract streams from this section are then mixed and sent to Solvent recovery unit.

4.4.3 Desolventizing

The raffinate from the extraction process is desolventized by a flash evaporation process which is based on the difference in the boiling points of propane and non-fat milk. The flash drum is modeled as a FLASH 2 which is available in the model library of Aspen PlusTM. The input stream (MIX 5) is flashed at 200°F and 60 psia.

At constant pressure, operating temperature is selected to be a value higher than the boiling point of propane and lower than that of Lactose. The upper limit of the temperature is determined by the influence of heat treatment on lactose. The pure solvent stream (RECPROP 1) which is then mixed with the solvent stream (RECPROP 2) recovered from dehydration unit and the vapor stream from solvent recovery unit (figure 30). The recovered solvent is then compressed and recycled.

4.4.4 Solvent Recovery

In this unit, propane is recovered from the extract stream. The recovery process is carried out in a vapor-liquid flash drum at a temperature of 270°F and pressure of 60 psia (figure 31). At these conditions of temperature and pressure, propane vaporizes while milk fat remains liquid enabling separation of solvent from the extract. The vaporized solvent stream contains small amounts of lactose and milk fat. This stream is cooled down to 150°F and flashed again to liquefy the fatty acids and lactose. This stream is mixed with the liquid stream of FLASH 4 to give the pure milk fat stream. The recovered solvent (RECPROP 1) is added to the pure solvent stream from the desolventizing section and recycled.

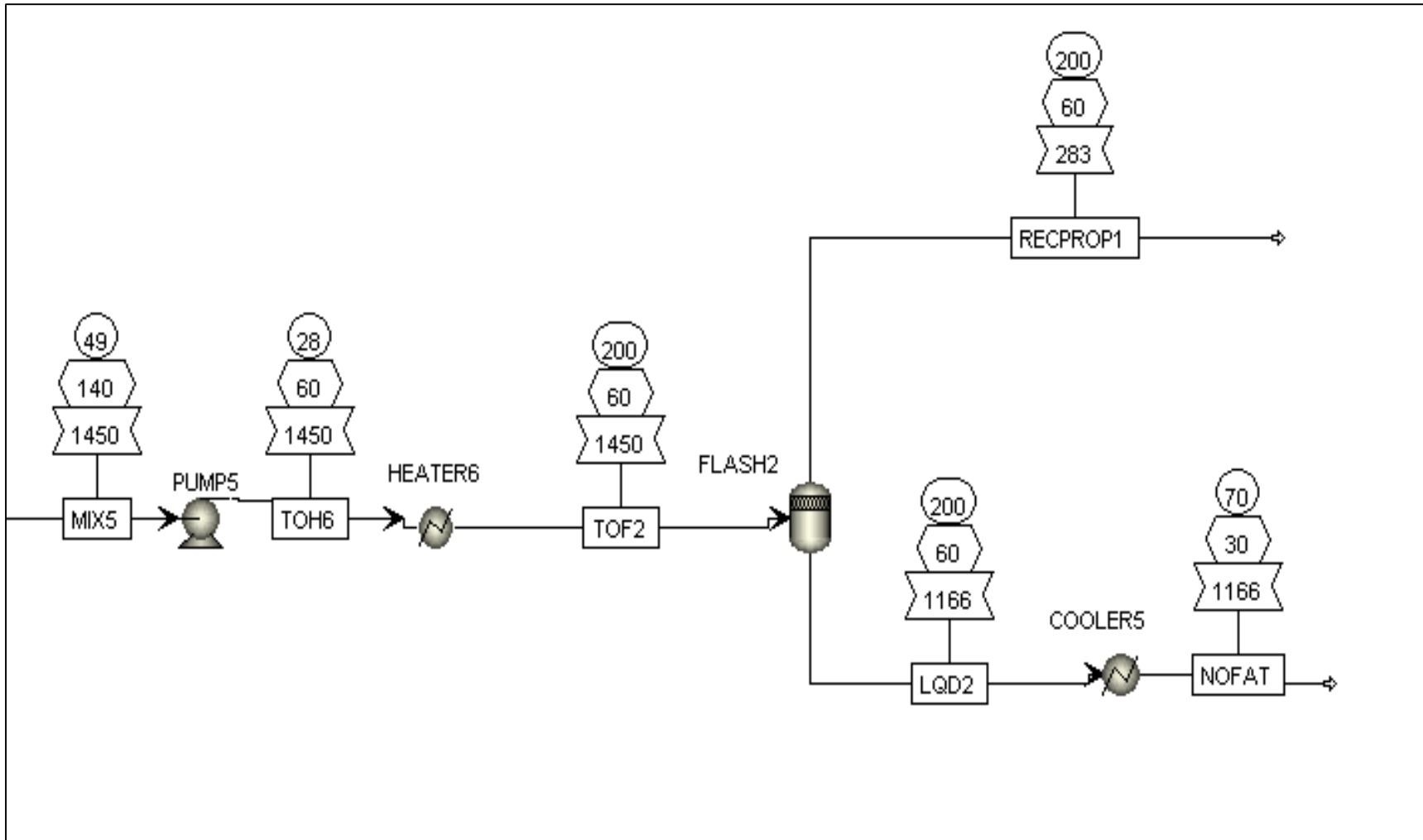


FIGURE 30 DESOLVENTIZING UNIT FLOW-SHEET

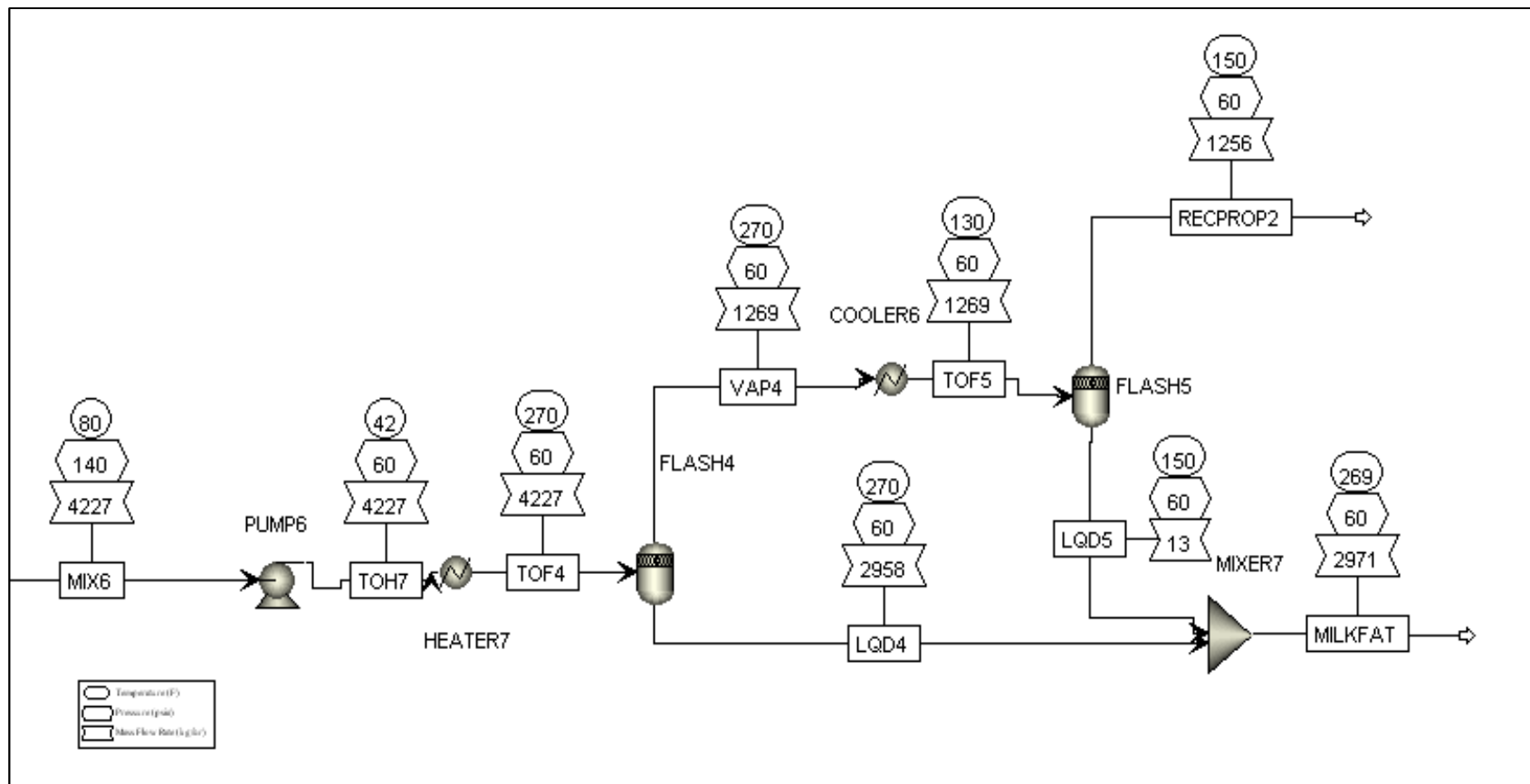


FIGURE 31 SOLVENT RECOVERY UNIT FLOW-SHEET

CHAPTER V

RESULTS AND DISCUSSION

5.1 DEHYDRATION UNIT

The purpose of the dehydration unit is to achieve maximum possible removal of water from milk. A number of test cases were run prior to the final simulation to determine the optimum flow conditions. The cases assumed different compositions of the milk stream. In each case, milk and propane are mixed and the mixture is flashed in a decanter at 70°F and 140 psia (figure 28).

5.1.1 Test Case 1

In this case, the input stream MILK was assumed to contain only Lactose (4.6 %) and water (95.4 %). At constant operating conditions, the separation was carried out at constant feed flow-rate of 1000 Kg/min and different solvent flow-rates. The criteria for selecting optimum solvent to feed ratio (S/F) are minimum use of solvent, maximum recovery of lactose from the water-rich phase and maximum removal of water from feed. Maximum recovery of lactose is determined by minimum lactose in the raffinate and maximum removal of water is determined by minimum water in the extract. Lactose in the raffinate and water in the extract are plotted against S/F and varied from 0.001 to 2 (figure 32). Lactose in the raffinate (RAFF 1) is observed to decrease with an increase in

S/F and the minimum is obtained at S/F of 2. Water in extract (EXCT 1) initially decreases with an increase in S/F, reaches a minimum at S/F of 0.1 and then starts increasing (figure 33).

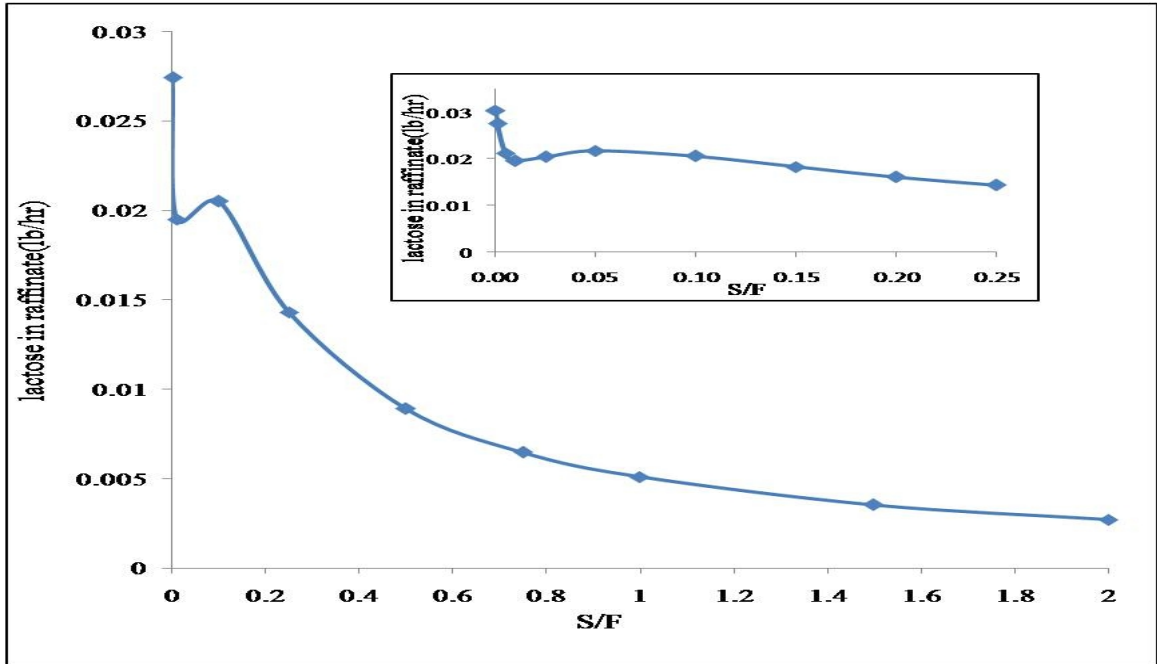


FIGURE 32 LACTOSE RECOVERY FOR CASE 1 (LWP)

Lactose is more soluble in propane than in water and so most of the lactose is recovered. This recovery increases when the amount of propane is increased. Along with lactose some water is also extracted into the propane rich phase and hence results in the decreasing and increasing trends of lactose and water respectively. At S/F of 0.1, two of our criteria i.e., minimum use of solvent and maximum removal of water are achieved. Though total recovery of lactose is not satisfied at this solvent to feed ratio, the amount of lactose in RAFF 1 is negligible when compared to that in the feed. For accurate values of optimum S/F, the plots were studied on an enlarged scale of S/F varying from 0.0001 to 0.25 (insert in figures 32 & 33). The optimum S/F is selected as 0.05 as it gives the

minimum water in extract. Also lactose in the raffinate did not vary much with S/F (insert in figure 32).

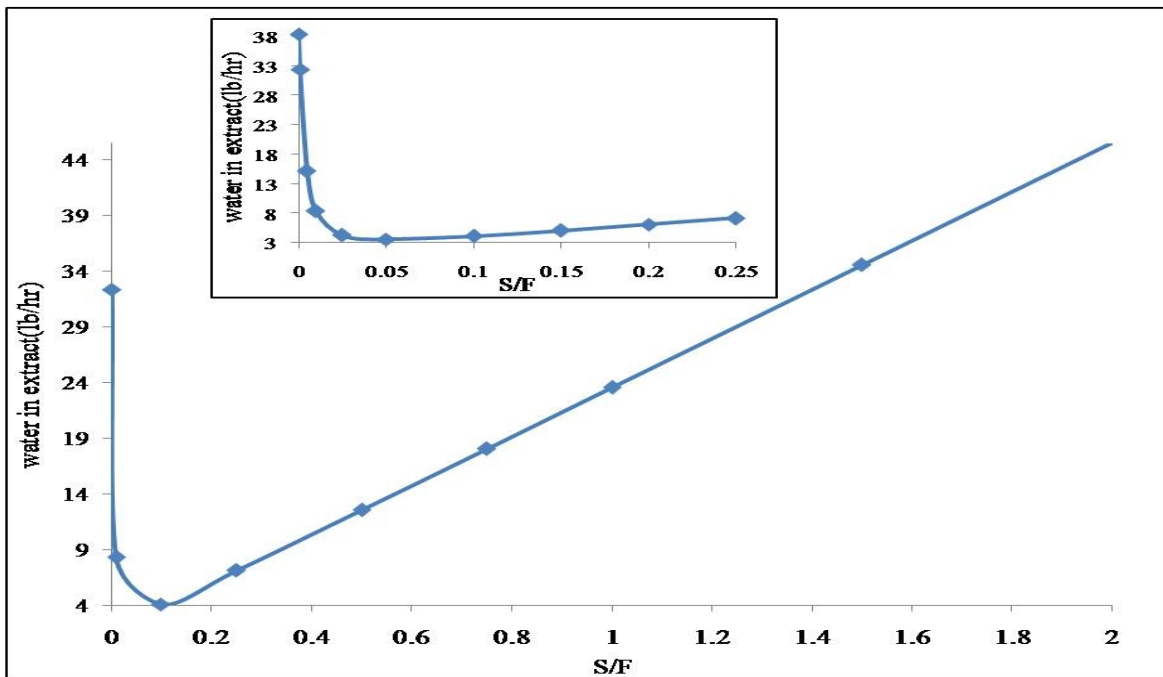


FIGURE 33 WATER REMOVAL FOR CASE 1(LWP)

5.1.2 Test Case 2

In this case the components of Milk were assumed to be oleic acid (3.2 %) and water (96.8 %). The solvent to feed ratio sensitivity analysis shows that with increasing S/F, the recovery of oleic acid increases (figure 34) and removal of water decreases (figure 35). The optimum S/F is selected to be the one which gives maximum removal of water for the same reasons stated in case 1. The optimum S/F in this case is 0.15. This run requires more propane compared to the previous run as solubility of lactose in propane is higher than that of oleic acid in propane.

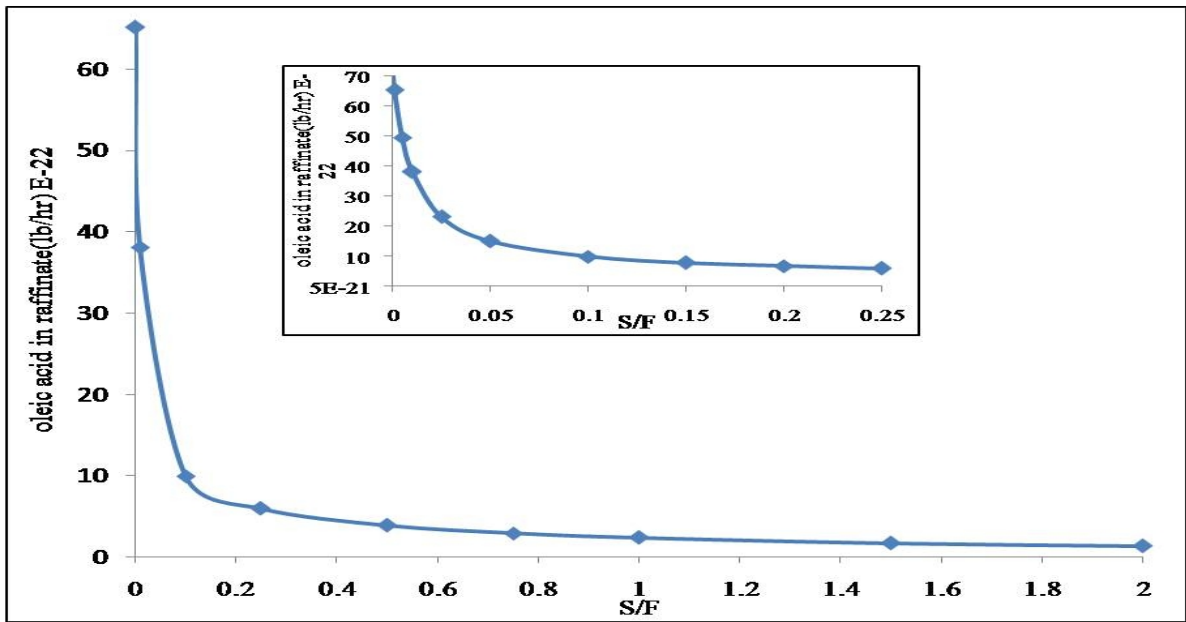


FIGURE 34 OLEIC ACID RECOVERY FOR CASE 2 (OWP)

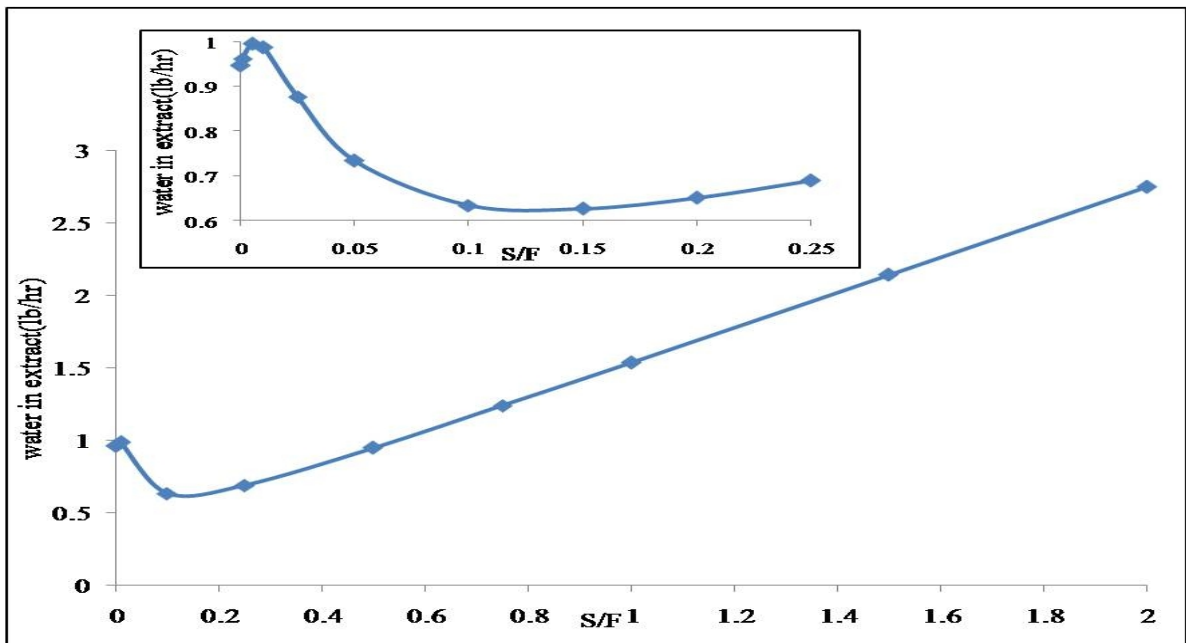


FIGURE 35 WATER REMOVAL FOR CASE 2(OWP)

5.1.3 Test Case 3

The constituents of milk in this case were assumed to be lactose (4.6 %), oleic acid (3.2 %) and water (92.2 %). Oleic acid, one of the most abundant fatty acids in milk was used as a model compound to represent the entire suite of fatty acids. The goal of this run was

to minimize lactose and oleic acid in the raffinate (RAFF 1) and water in the extract (EXT 1).

S/F sensitivity analysis

Lactose in the raffinate (figure 36) decreases with increasing S/F where as water in the extract increases with increasing S/F (figure 37). Recovery of Oleic acid (figure 37) follows a trend similar to lactose. As the amount of oleic acid and lactose present in raffinate are small, minimizing the use of solvent and maximizing the removal of water are considered to be the criteria. Maximum removal of water is achieved at S/F of 0.1 and hence it is selected as the optimum solvent to feed ratio.

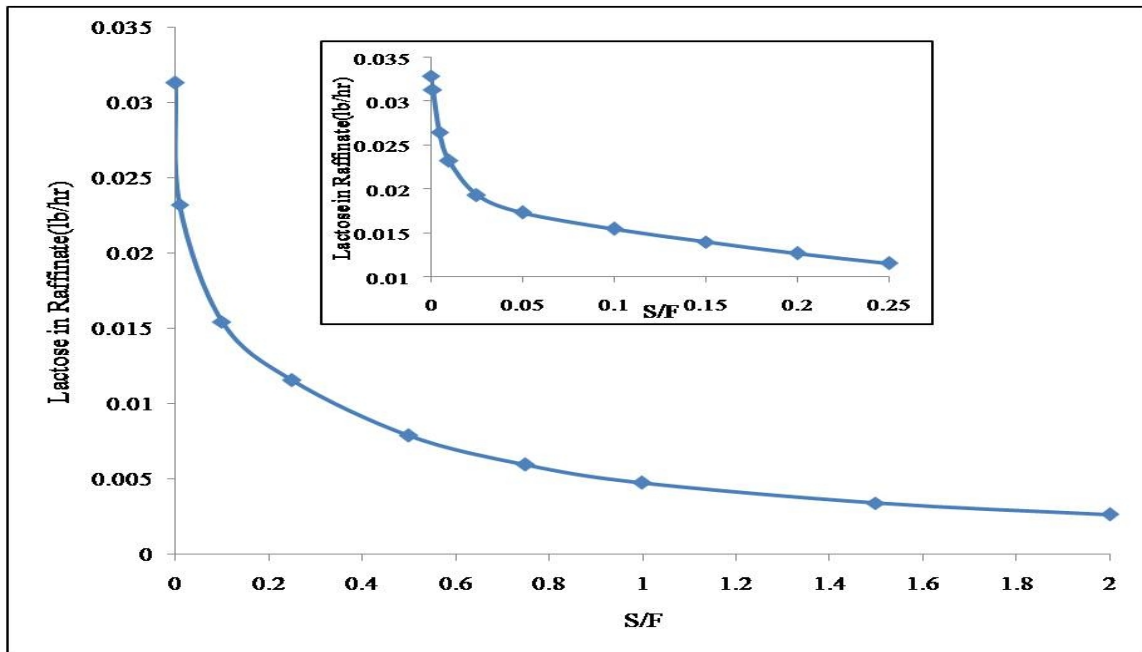


FIGURE 36 LACTOSE RECOVERY FOR CASE 3 (LOWP)

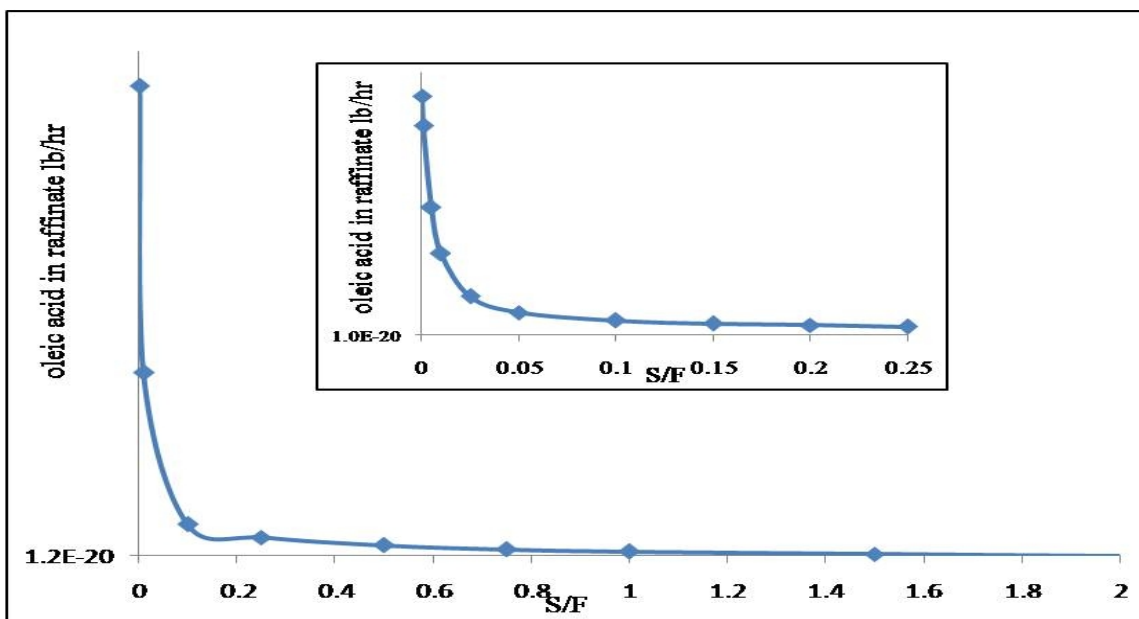


FIGURE 37 OLEIC ACID RECOVERY FOR CASE 3 (LOWP)

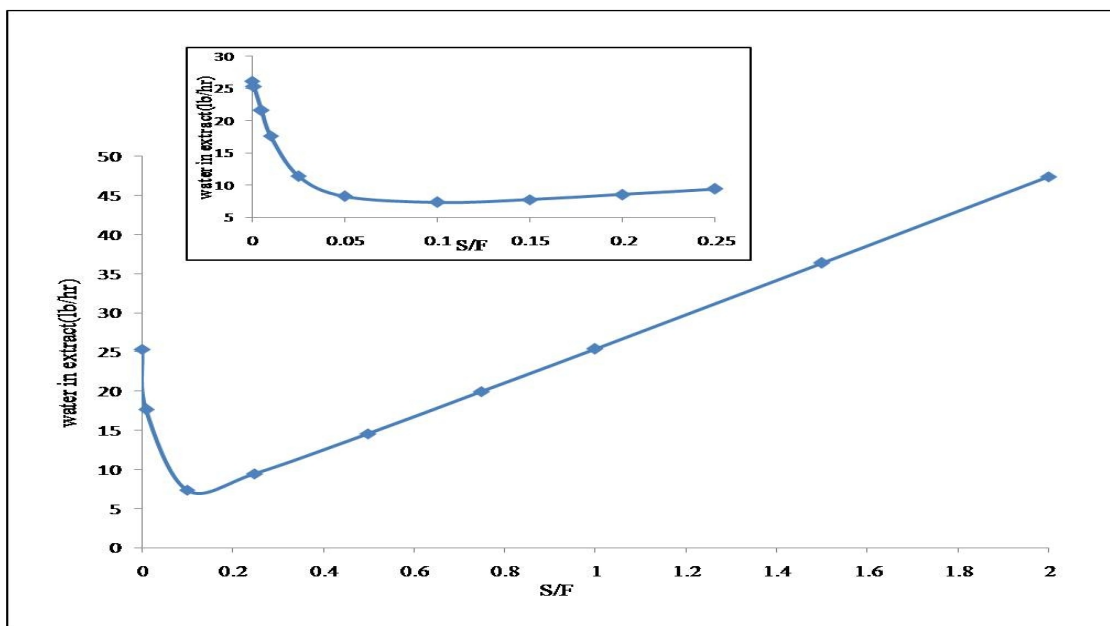


FIGURE 38 WATER REMOVAL FOR CASE (LOWP)

Saturation test

For further extraction, the raffinate obtained in the above run was sent through a series of three decanters. A fresh stream of propane was mixed with each raffinate stream before it enters the next decanter (figure 39).

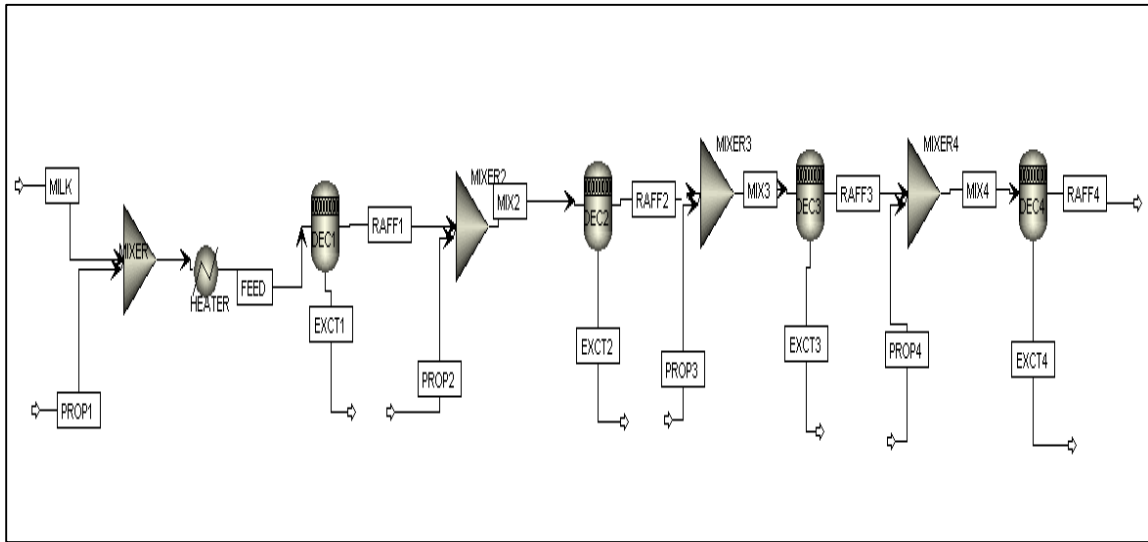


FIGURE 39 CROSS-CURRENT DEHYDRATION RUN

This cross-current operation was carried out at constant temperature and pressure of 70°F and 140 psia. The propane solvent is fed to each decanter at the same flow-rate. There is no significant change in the amount of propane present in RAFF 3 and RAFF 4 as compared to RAFF 2 (Table 12). All of the propane added as fresh stream comes out in the extract streams. Addition of propane has no effect on extraction after DEC 2. This shows that the water in RAFF 2 is saturated with propane and hence no further change in the amount of propane in the raffinate streams. The same trend was observed for different solvent to feed ratios.

Also no further extraction takes place after DEC 3 as recovery of lactose and removal of water reached optimum. As the separation that takes place in DEC 3 is insignificant, considering it is not economically feasible. Hence two decanters are optimum for the dehydration unit.

COMPONENT	WEIGHT %			
	RAFF1	RAFF2	RAFF3	RAFF4
WATER	99.99	99.96	99.96	99.96
PROPANE	5.63E-03	4.29E-02	4.29E-02	4.29E-02
Lactose	2.25E-07	1.31E-11	0.0	0.0
Oleic acid	1.18E-07	2.5E-10	0.0	0.0

TABLE 12 STREAM RESULTS FOR SATURATION TEST

Mode of operation

The cross-current mode of operation was compared with a counter-current mode to determine optimal operation. The two decanters were replaced by an extractor operating at the same conditions. The feed and solvent were passed counter currently in the extractor. The S/F sensitivity analysis was carried out for all the three cases; 2 decanters operating in cross-current mode, 2- staged and 3-staged extractor operating in counter-current fashion.

Lactose recovery increases with increasing S/F in all the three cases and no significant variation is observed among them (figure 40). Conversely, the variation between cross and counter-current modes is different for water removal. The cross-current mode with 2 decanters gives higher removal of water compared to 2 stage and 3 stage extractors over the entire range of S/F (figure 41). Cross-current mode of operation is opted for dehydration unit as it gives maximum removal of water at a lower solvent to feed ratio. However the recovery of lactose is the same in all cases. Since Oleic acid is quantitatively extracted by propane, recovery is the same for all three cases.

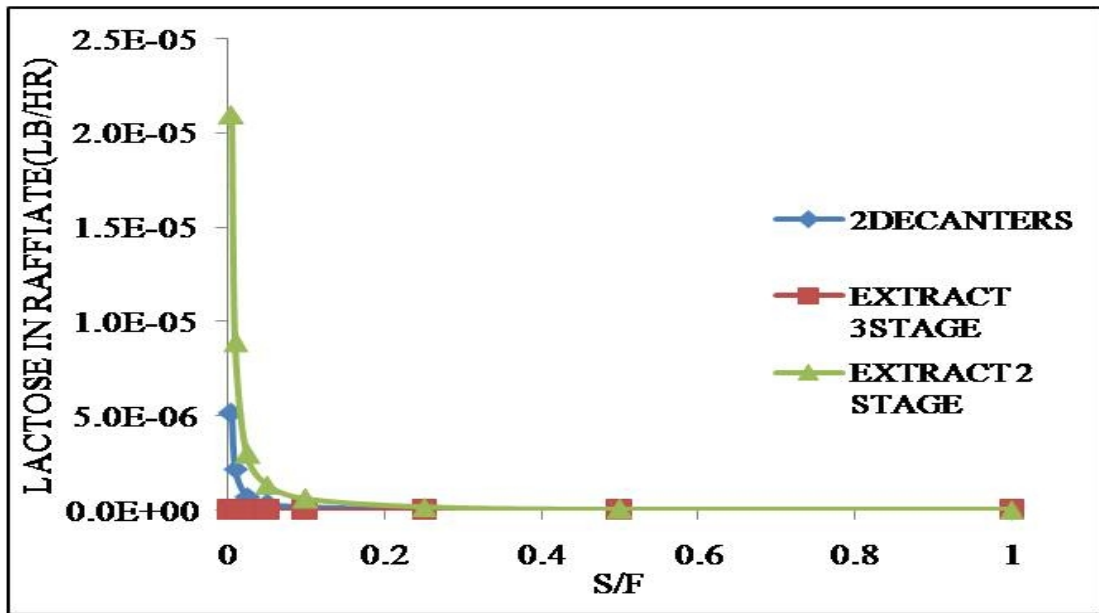


FIGURE 40 LACTOSE RECOVERY FOR DIFFERENT MODES OF OPERATION

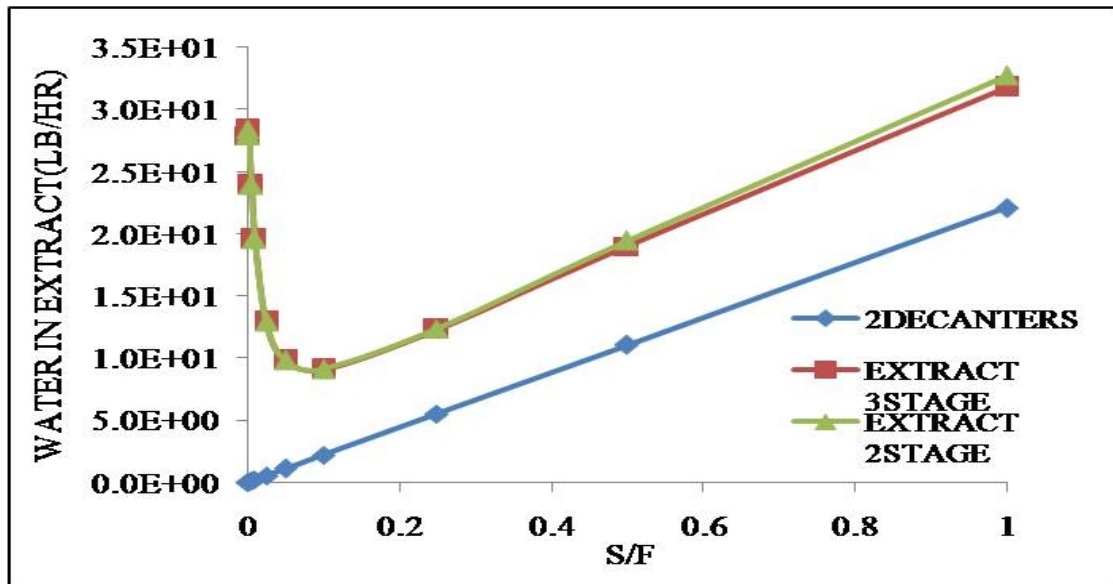


FIGURE 41 WATER REMOVAL FOR DIFFERENT MODES OF OPERATION

Though lactose recovery is higher for S/F_2 of 0.005, the optimum S/F_2 is 0.001 as the variation in amount of lactose in raffinate for different S/F_2 is negligible. As seen from the figures 42 & 43, the optimum combination of S/F s is S/F_1 of 0.001 and S/F_2 of 0.001. Adding decanters in cross-current fashion reduces the solvent consumption.

Sensitivity analysis of S/F combination

Assuming cross-current operation, it is important to find the optimum combination of S/F for the two solvent streams. As observed from the previous cases, the optimum S/F for the first decanter (S/F_1) is in the range of 0.001 to 0.1 and so for this range of (S/F_1), the sensitivity of S/F to second decanter (S/F_2) is analyzed.

From figures 42 and 43 it is observed that though lactose recovery and water removal follow opposite trends with S/F_2 as in earlier cases, they do not vary much with S/F_1 . Irrespective of S/F_1 the optimum S/F_2 is 0.001, since maximum removal of water is obtained at this value (figure 41). S/F_2 values of 0.001 and 0.005 are varied over a wide range of S/F_1 to find the optimum combination of the S/F (insert of figures 42 and 43).

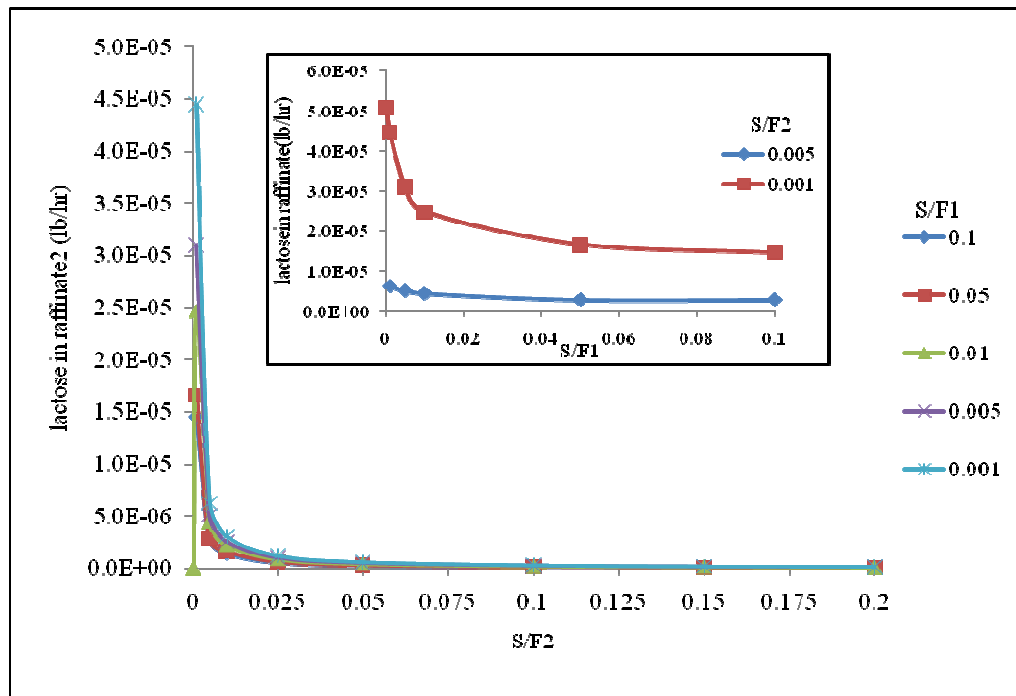


FIGURE 42 LACTOSE REMOVAL FOR SENSITIVITY ANALYSIS OF S/F_2

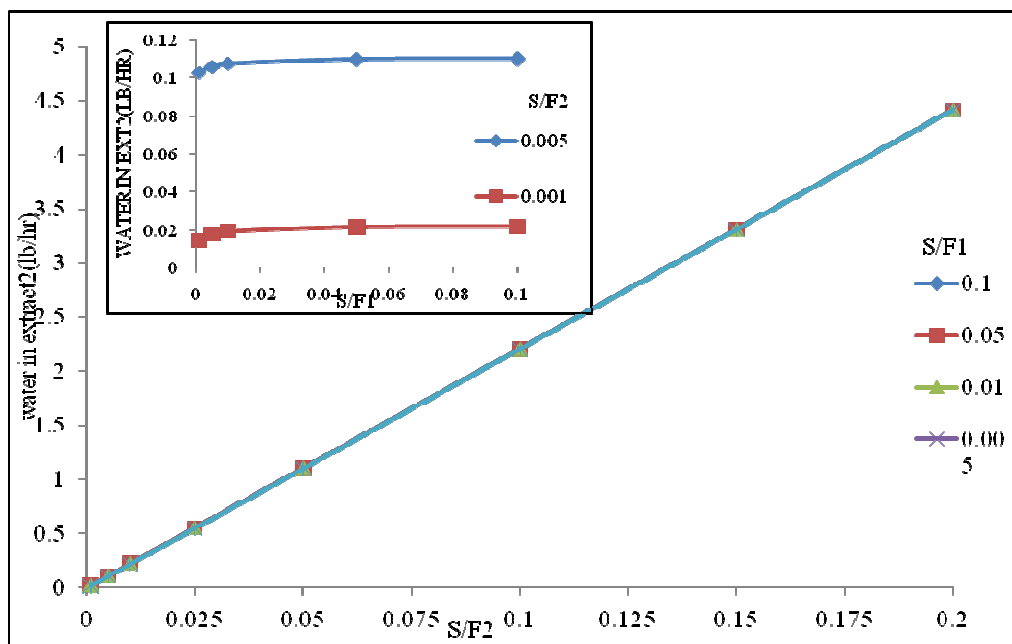


FIGURE 43 WATER REMOVAL FOR SENSITIVITY ANALYSIS OF S/F₂

5.1.4 Base case simulation results

Based on the results of the test cases, a simulation with a milk stream composition as given in figure 26 was run in cross-current mode with two decanters. The solvent to feed ratio of the two propane streams was 0.001. The first decanter was operated at feed conditions while the second decanter (DEC 2) was operated at 30°F and 140 psia based on the temperature sensitivity analysis of case 3. When the mixture of milk and propane was separated in the first decanter, 99.97 % of water came out as raffinate while almost 99.9 % of all fatty acids and lactose came out in the propane extract stream.

Except for lactose all other components of milk are almost insoluble in water (table 4). As fatty acids are much more soluble in propane than in water, the split fraction of fatty acids is very high with 99.9 % of them the propane extract stream.

The raffinate was then mixed with propane and sent to DEC 2. The operating conditions of this decanter was based on the sensitivity analysis shown in figure 44. Water in the

extract stream increases with increase in temperature. As water in extract must be minimized, 30°F was chosen as the operating temperature. Further decrease in temperature increases lactose content in the raffinate which is undesirable. 99.999% separation of water took place in this decanter. RAFF 2 was pure water with little propane and no fatty acids. The extract from DEC 2 contained 0.022 % water and so it was cooled and sent to DEC 3 for further removal of water.

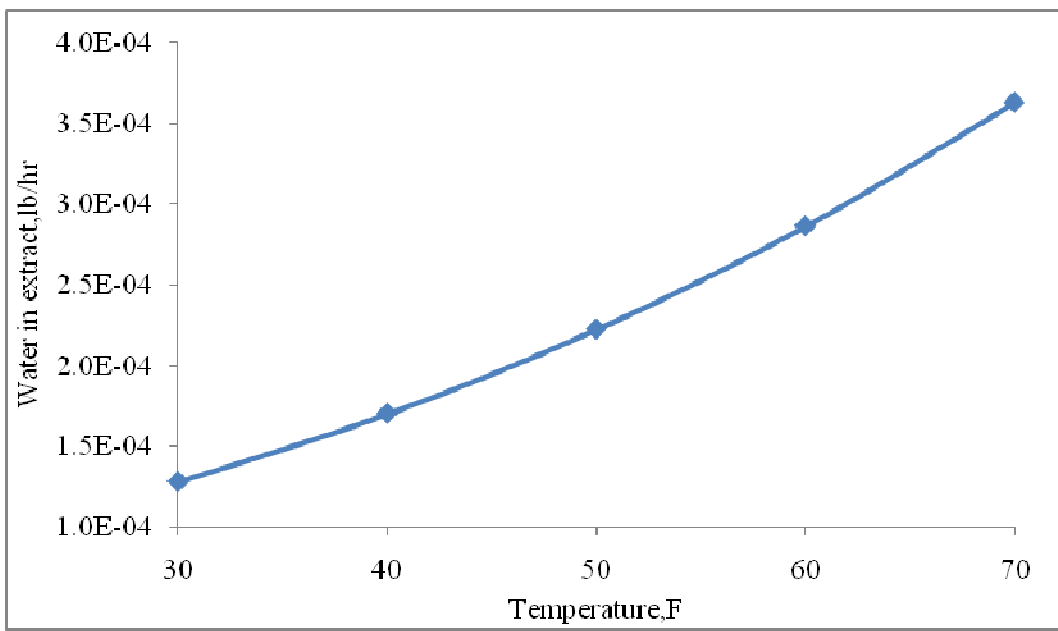


FIGURE 44 TEMPERATURE SENSITIVITY ANALYSIS FOR DECANTER 2

EXT 3, containing no water, was sent to the extraction unit. RAFF 3 was then mixed with RAFF 2 and this mixture was flashed at high temperature in Flash 2. At this temperature and pressure, all of the propane vaporized and liquid water which was 99.9991 % pure came out at the bottom of the flash drum. The vapor stream was compressed for recycling. All the input and output stream compositions are shown in the appendix.

5.2 EXTRACTION UNIT

The extract stream (EXT 3) from the dehydration unit was the input stream for the extraction unit. The input specifications of this unit are shown in table 13. Extraction of fat from anhydrous feed was carried out in cross and counter-current modes. The cross-current mode was modeled using decanter, and the counter-current mode using a counter-current extraction column. The input stream containing anhydrous milk and propane was heated and flashed in a decanter (DEC 4) at 50°F and 140 psia. The operating conditions of this decanter were determined based on the temperature sensitivity analysis shown in figure 45.

Component	Weight %		
	Input stream(EXCT3)	Raffinate(RAFF4)	Extract(EXCT4)
Propane	1.4	0.8	1.9
Lactose	66.7	91.9	43.8
Total Fat	31.9	7.3	54.3

TABLE 13 STREAM COMPOSITION FOR DECANTER 4

The higher the amount of lactose in raffinate the more lactose is removed from the milk fat. With increase in temperature, removal of lactose initially decreases, reaches optimum at 50°F and then starts increasing. Figure 45 with S/F = 0.001 shows that maximum extraction is obtained at 50°F and hence the operating conditions of 50°F and 140 psia. The decanter stream output is given in table 13.

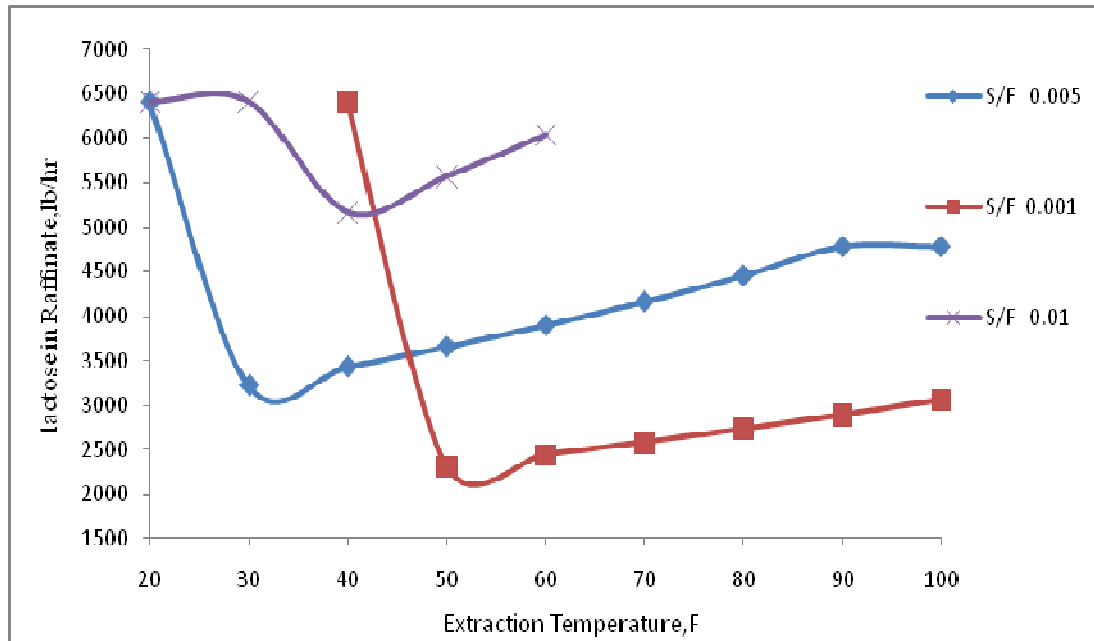


FIGURE 45 TEMPERATURE DEPENDENCE OF EXTRACTION IN DEC 4

The raffinate (RAFF 4) from this decanter was mixed with propane at 70°F and 140 psia with mass flow-rate of 0.1 kg/min. This mixture was then cooled and flashed in DEC 5 at 30°F and 140 psia. Operating conditions of this decanter were determined based on a sensitivity analysis similar to the previous decanter. The raffinate from this decanter was then sent to an extract column with 2 stages operating adiabatically at 60°F and 140 psia in a counter-current mode.

Propane with mass flow-rate 25 kg/min was sent from the bottom of the tower at the operating conditions. To determine the operating conditions of this column, temperature and stage sensitivity analyses were carried out.

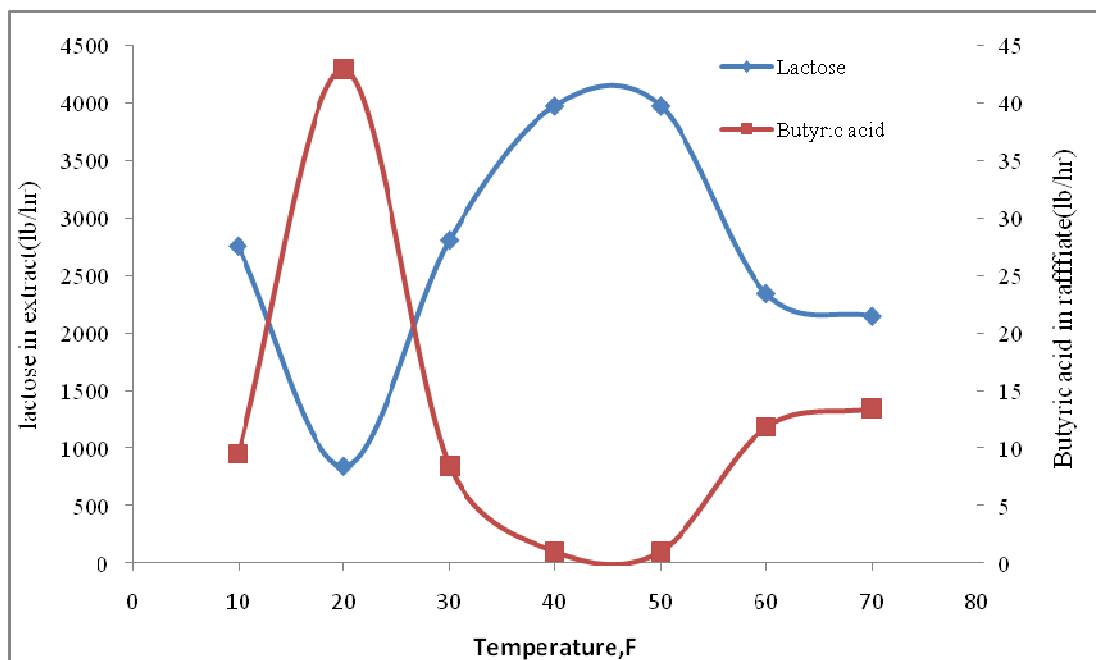


FIGURE 46 TEMPERATURE SENSITIVITY ANALYSIS AT 60PSIA FOR EXTRACT

Figure 46 shows the effect of temperature on the extract with 2 stages operating at 140 psia. Along with lactose, fatty acids tend to come out in the raffinate stream. To represent these fatty acids, removal of butyric acid is plotted as it has the highest mass fraction in the input fat stream. Removal of lactose should be maximized while that of butyric acid minimized. As seen in the figure, removal of lactose and butyric acid follow opposite trends. As temperature increases, lactose removal first decreases then increases and then decreases again. Maximum removal of lactose is observed at 20°F but butyric acid removal is also maximized at this temperature. To optimize both the conditions, the operating temperature is selected as 60°F.

The number of stages in the column was determined from the analysis shown in figure 47. At 10°F and 140 psia, the extraction was carried out with different number of stages. Removal of lactose is inversely proportional to the number of stages. It is observed that the extract with the minimum number of stages gives maximum separation.

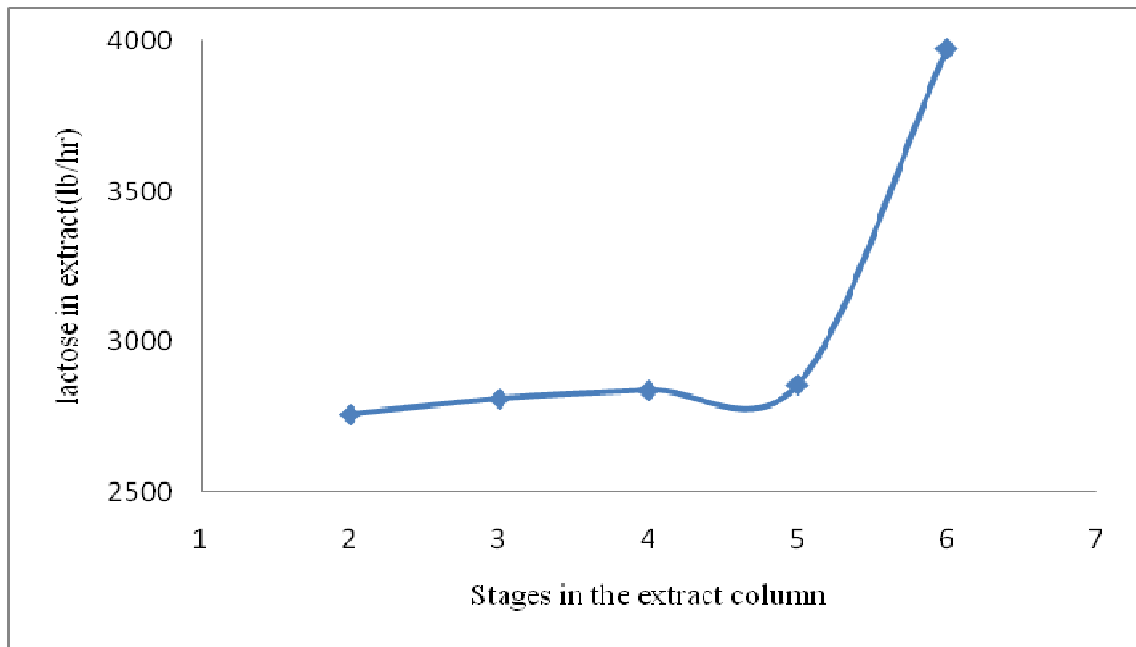


FIGURE 47 EFFECT OF NUMBER OF STAGES IN EXTRACT

To determine the effect of pressure on the extraction, a pressure sensitivity analysis was carried out. The variation of percent removal of lactose with pressure is found to be negligible as shown in figure 48. The extraction process is independent of pressure. Hence the extract was operated with 2 stages at 60°F and 140 psia. The stream composition of the column is given in table 14.

Component	Weight %		
	Input stream(RAFF5)	Raffinate(BTM1)	Extract(TOD6)
Propane	0.8	18.8	53.3
Lactose	92.3	79.2	42.1
Total Fat	6.9	2	4.6

TABLE 14 STREAM COMPOSITION FOR EXTRACTOR

The raffinate stream, lean in fat was sent for further extraction to two more decanters operating at 70°F and 30°F respectively. The operating conditions of DEC 6 and DEC 7 were determined based on temperature analysis carried out similar to DEC 4. The raffinate streams from DEC 6, DEC 7 and EXTRCT were all mixed to give fat free milk.

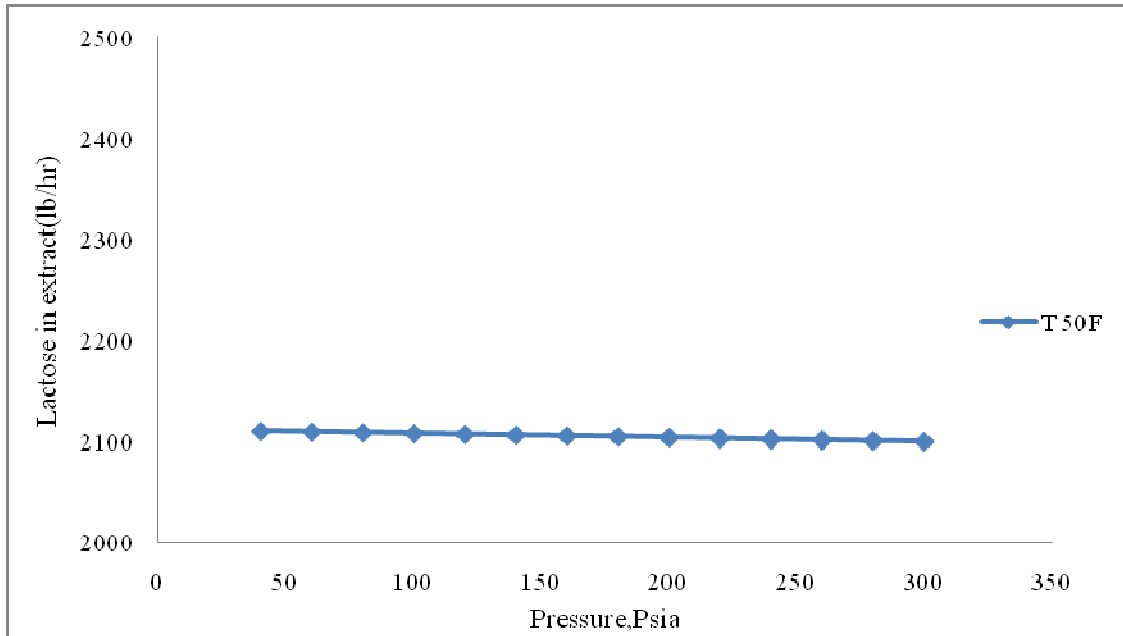


FIGURE 48 PRESSURE SENSITIVITY ANALYSIS FOR DEC 4

The output stream from the extraction process, MIX5 contains 2.8 % total fat (table 15).

Component	Weight %		
	Input stream(EXCT3)	Raffinate(MIX5)	Extract(MIX6)
Propane	1.4	20.1	30.2
Lactose	66.7	77.1	39.2
Total Fat	31.9	2.8	30.6

TABLE 15 STREAM COMPOSITION FOR EXTRACTION UNIT

5.3 DESOLVENTIZING UNIT

The raffinate from the extraction process containing 20.1 % propane was desolventized in this section by flash separation at 200°F and 60 psia. Figure 49 shows the temperature and pressure sensitivity analyses.

The percent of propane removal and lactose removal are plotted for wide temperature range of 50°F to 400°F. Propane removal is directly proportional to temperature. Maximum solvent removal is obtained at the highest temperature but the upper limit is

determined by the stability of lactose. Temperatures higher than 200°F cause browning and isomerization reactions, which affect the nutritional values and product quality.

Propane removal increases with a decrease in pressure, as shown in figure 51.

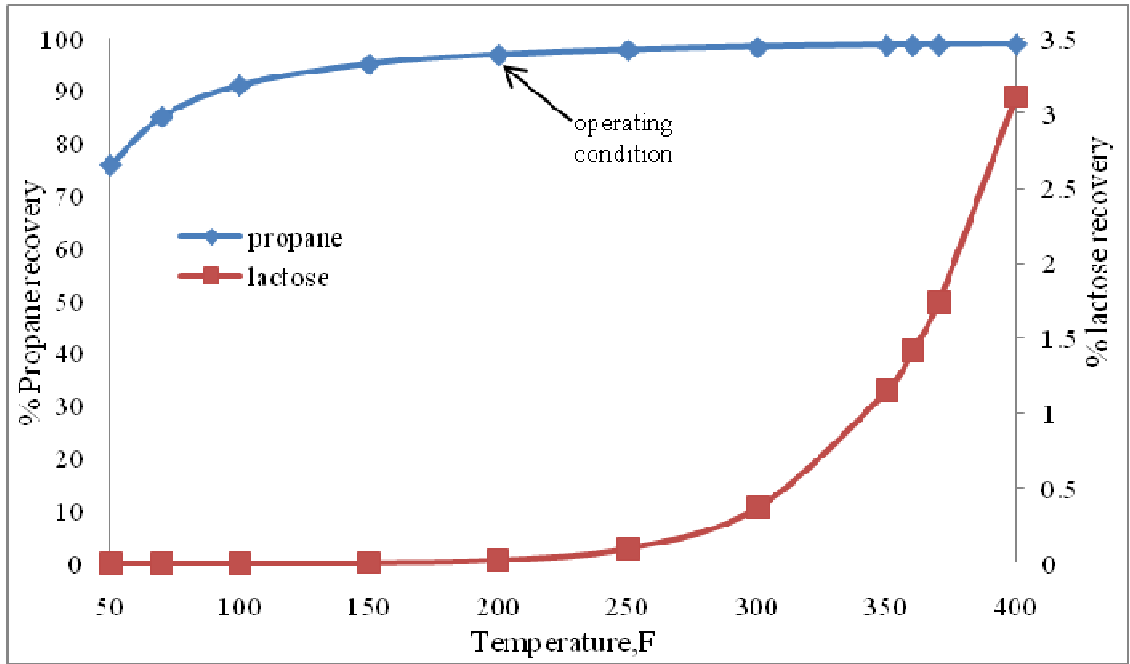


FIGURE 49 TEMPERATURE EFFECT ON FLASH 2

The fat lean milk stream (NOFAT) had 0.8 % propane and 95.8 % lactose. The composition of the input and output streams is given in table 16.

component	Weight%		
	Input(MIX5)	Vapor(RECPROP1)	Liquid(NOFAT)
Propane	20.1	99.8	0.8
Lactose	77.1	828 PPM	95.8
Total Fat	2.8	~ 0.0 2	3.4

TABLE 16 STREAM COMPOSITION FOR DESOLVENTIZING UNIT

5.4 SOLVENT RECOVERY UNIT

The specifications of the input stream (MIX 6) to the solvent recovery unit are shown in table 17. It contains 30.2 % propane. When the depressurized fat rich stream was flashed at 270°F, 97.4 % propane vaporized. The effect of temperature and pressure on the evaporation is shown in the figures 50 ad 51. Along with propane, some of the fatty acids also started to vaporize. To increase the extract purity, removal of fatty acids should be minimized while maximizing the removal of propane.

Removal of propane and caproic acid are observed to determine the operating conditions. Caproic acid is chosen to represent the entire suit of fatty acids due to its high volatility. At 60 psia, removal of both propane and caproic acid increased with increase in temperature. At 270°F, 98.5 % removal of propane and 30.8 % removal of caproic acid were obtained. Further increase in temperature causes instability in the milk fat. Higher temperature stimulates oxidation reaction and causes deterioration and coagulation.

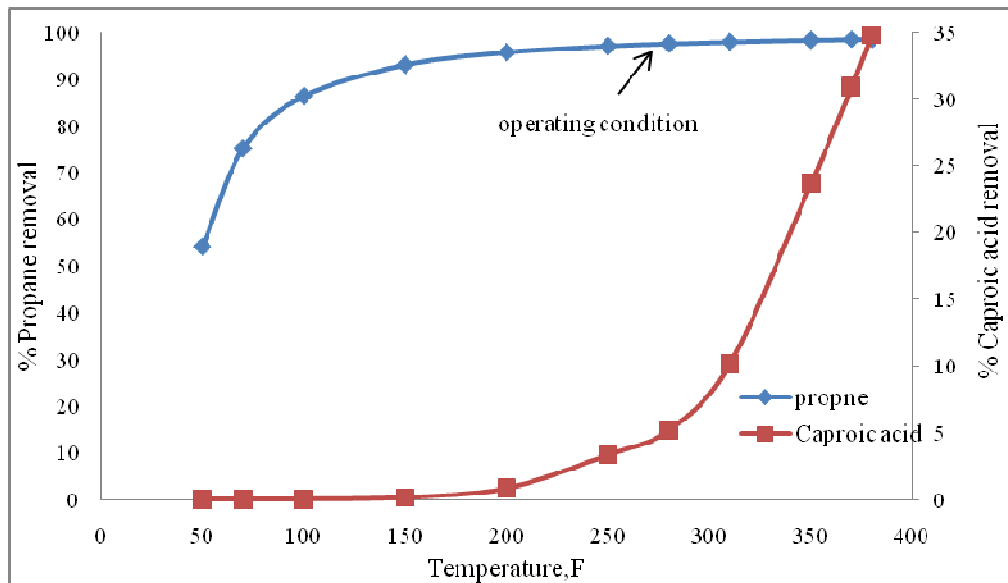


FIGURE 50 TEMPERATURE SENSITIVITY ANALYSIS OF FLASH 4

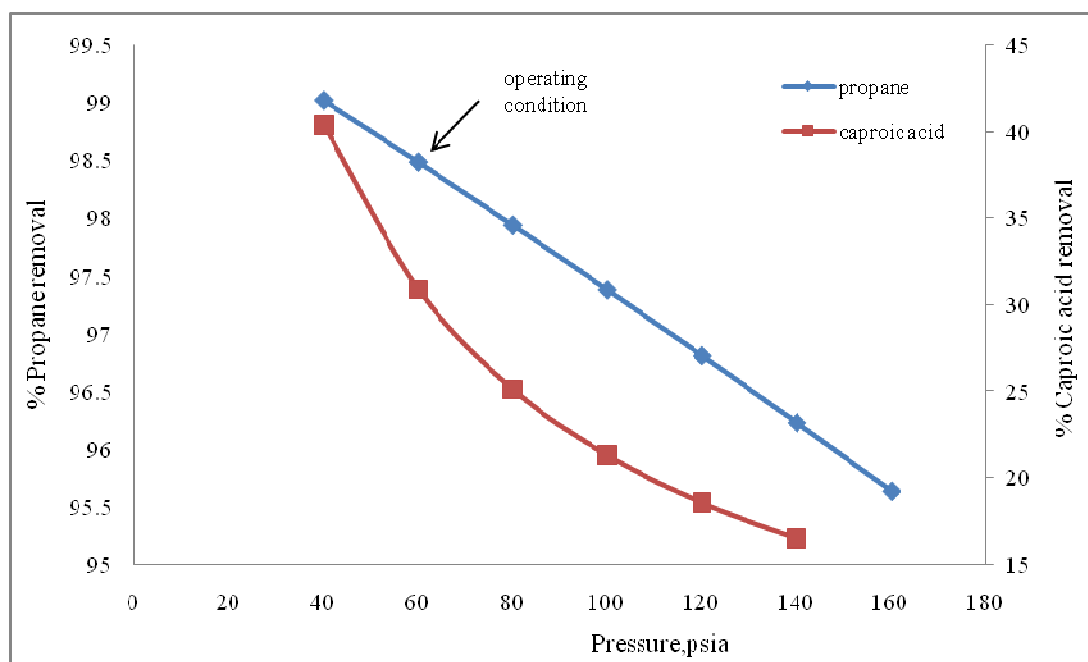


FIGURE 51 PRESSURE SENSITIVITY ANALYSIS OF FLASH 4

At 270°F, the effect of pressure on the evaporation was examined. As seen in figure 51, propane removal and caproic acid removal decreases as the pressure increases. The optimum condition is found to be 60 psia which gives 98.5 % propane removal and 30.8 % caproic removal. Hence it can be seen that high temperature and low pressure are suitable to obtain pure extract.

component	Weight%		
	Input(MIX6)	Vapor(RECPROP2)	Liquid(MILKFAT)
propane	30.2	99.6	1.1
lactose	39.2	44PPM	55.7
Total Fat	30.6	0.4	43.2

TABLE 17 STREAM COMPOSITION FOR SOLVENT RECOVERY UNIT

The vapor stream with 98.6 % propane and 0.9 % fat was sent to a second flash after being cooled to 150°F. In this flash, the input stream was de-vaporized and the fatty acids were separated as a liquid stream. The outlet stream composition is given in table 17. The

recovered solvent stream was mixed with the solvent stream recovered from the other units and also recycled. This solvent stream was 99.6 % pure (table 17).

CHAPTER VI

CONCLUSIONS AND RECOMENDATIONS

The lipid extraction process using propane is advantageous over other method using other solvents. Low solvent flow rate, high purity end product and suppressed hazardous operating conditions are some of the advantages. Use of propane for milk extraction has not been studied in great detail and no design information is available in the literature. Therefore the main objective of this work was to check the feasibility of propane for liquid –liquid extraction in Aspen PlusTM.

Aspen PlusTM was used to generate the process models and simulations. The design parameters were obtained based on the sensitivity analyses carried out for each unit. The operating conditions were set to provide maximum extraction yield with maximum product purity and solvent recovery. Modeling liquid-liquid extraction using Aspen PlusTM was also explained in detail.

6.1 CONCLUSIONS

1. The simulations indicate that most extractions can be run at ambient temperature.

2. High temperatures give higher yields with the upper limit determined by the stability of milk components. The pressure does not have any effect on the extraction but it is important in order to keep the propane solvent in the liquid state.
3. Besides temperature, extraction depends largely on solvent flow rate and mode of operation.
4. Dehydration processes give high yield in cross-current extraction mode modeled with two decanters. Further addition of decanters would lead to increased costs with no significant benefit.
5. In the dehydration process removal of 99.98 % water is achieved with a solvent to feed ratio of 0.001. Higher solvent flow rates provide no major benefit to product quality but result in higher cost.
6. Fat content of anhydrous milk was reduced from 32 % to 3.4 % in this extraction process.
7. The Desolventizing unit is a single flash unit operated at 200°F and 60 psia. Addition of another flash unit has no significant impact on either the solvent purity (99.6 %) or the product purity (95.6 %). High product purity is obtained at higher temperature but further increase in temperature causes lactose degradation.
8. Recovery of solvent increases with an increase in temperature. It can be carried out at 270°F and 160 psia to obtain 99.6 % pure solvent.

6.2 FUTURE WORK

The following factors can be considered to improve the current work

1. This model considers only fatty acids as lipid content of milk due to lack of thermodynamic data of triglycerides and phospholipids. A database can be developed with properties of compounds calculated from group contributions. Including all the compounds would help in predicting accurate results.
2. Binary interaction parameters can be regressed from experimental data. Multi component liquid-liquid equilibria would result in much more reliable results using available binary interaction parameters. Parameters regressed from multi-component data is essential if more accuracy is required.
3. This model does not consider solids present in milk such as nutrients, proteins and ash. Solvent extraction can be used to model the fractionation of milk including these solids.
4. No experimental data is available in the literature to be compared with the obtained results. Experiments can be carried out to generate data for model validation.
5. The milk fat stream still contains 55.4 % lactose. Crystallization can be considered to separate lactose from fat.

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APPENDIX: SIMULATION RESULTS

Table A-1 Stream summary for Decanter 1 in Dehydration unit

STREAM ID	MILK	PROPANE	MIX1	FEED	EXT1	RAFF1
Temperature F	70	70	101.2849	70.0000	70	70
Pressure psia	140	140	140.0000	140.0000	140	140
Vapor Frac	0	0	0.0000	0.0000	0	0
Enthalpy kcal/kg	-3596.9825	-657.73	-3594.0462	-3594.8506	1202.241	-3787.339
Total Flow kg/hr	60000.0007	60.00	60060.0007	60060.0007	4160.695	55899.306
COMPONENTS:	KG/HR					
LACTOSE	2765.36484	0	2765.3648	2765.3648	2765.352	0.013
WATER	55908.4631	0	55908.4631	55908.4631	12.271	55896.192
PROPANE	0	60.00	60.0000	60.0000	56.905	3.10E+00
OLEIC-01	45.0874702	0	45.0875	45.0875	45.087	8.75E-17
CAPROIC	45.0874702	0	45.0875	45.0875	45.087	9.41E-06
PALMITIC	539.246144	0	539.2461	539.2461	539.246	1.05E-15
STEQARIC	219.425688	0	219.4257	219.4257	219.426	4.26E-16
MYRISTIC	178.546382	0	178.5464	178.5464	178.546	3.46E-16
LINOLEIC	72.1399524	0	72.1400	72.1400	72.140	1.40E-16
LAURIC	46.2898028	0	46.2898	46.2898	46.290	8.98E-17
BUTYRIC	45.0874702	0	45.0875	45.0875	45.081	6.62E-03
LINOLENI	45.0874702	0	45.0875	45.0875	45.087	8.75E-17
CAPRIC	45.0874702	0	45.0875	45.0875	45.087	3.93E-14
CAPRYLIC	45.0874702	0	45.0875	45.0875	45.087	4.19E-09
COMPONENTS:	MASS FRAC					
LACTOSE	0.04608941	0	0.0460	0.0460	0.665	2.25E-07
WATER	0.93180771	0	0.9309	0.9309	0.003	0.99994
PROPANE	0	1	0.0010	0.0010	0.014	5.54E-05
OLEIC-01	0.00075145	0	0.0008	0.0008	0.011	1.57E-21
CAPROIC	0.00075145	0	0.0008	0.0008	0.011	1.68E-10
PALMITIC	0.00898743	0	0.0090	0.0090	0.130	1.87E-20
STEQARIC	0.00365709	0	0.0037	0.0037	0.053	7.62E-21
MYRISTIC	0.00297577	0	0.0030	0.0030	0.043	6.20E-21
LINOLEIC	0.00120233	0	0.0012	0.0012	0.017	2.50E-21
LAURIC	0.00077149	0	0.0008	0.0008	0.011	1.61E-21
BUTYRIC	0.00075145	0	0.0008	0.0008	0.011	1.18E-07
LINOLENI	0.00075145	0	0.0008	0.0008	0.011	1.57E-21
CAPRIC	0.00075145	0	0.0008	0.0008	0.011	7.03E-19
CAPRYLIC	0.00075145	0	0.0008	0.0008	0.011	7.50E-14

Table A-2 Stream summary for Decanter 2 in Dehydration unit

STREAM ID	PROP2	MIX2	TOD2	EXT2	RAFF2	MIX3
Temperature F	70.00	69.85	29.87	30.00	30.00	30.00
Pressure psia	140.00	140.00	140.00	140.00	140.00	140.00
Vapor Frac	0.00	0.00	0.00	0.00	0.00	0.00
Enthalpy kcal/kg	-657.73	-3783.98	-3802.43	-672.41	-3805.08	-3805.08
Total Flow kg/hr	60.00	55959.31	55959.31	47.28	55912.02	55924.29
COMPONENTS	KG/HR					
LACTOSE	0	1.26E-02	1.26E-02	1.26E-02	7.34E-07	9.11E-07
WATER	0	5.59E+04	5.59E+04	2.31E-03	5.59E+04	5.59E+04
PROPANE	60.00	6.31E+01	6.31E+01	4.73E+01	1.58E+01	1.58E+01
OLEIC-01	0	8.75E-17	8.75E-17	0.00E+00	0.00E+00	5.41E-26
CAPROIC	0	9.41E-06	9.41E-06	9.41E-06	1.23E-11	1.57E-10
PALMITIC	0	1.05E-15	1.05E-15	0.00E+00	0.00E+00	1.60E-23
STEQARIC	0	4.26E-16	4.26E-16	0.00E+00	0.00E+00	1.02E-24
MYRISTIC	0	3.46E-16	3.46E-16	0.00E+00	0.00E+00	3.84E-23
LINOLEIC	0	1.40E-16	1.40E-16	0.00E+00	0.00E+00	1.38E-25
LAURIC	0	8.98E-17	8.98E-17	0.00E+00	0.00E+00	8.27E-23
BUTYRIC	0	6.62E-03	6.62E-03	6.60E-03	1.38E-05	1.41E-05
LINOLENI	0	8.75E-17	8.75E-17	0.00E+00	0.00E+00	5.10E-26
CAPRIC	0	3.93E-14	3.93E-14	0.00E+00	0.00E+00	3.62E-20
CAPRYLIC	0	4.19E-09	4.19E-09	4.19E-09	7.48E-19	2.08E-14
COMPONENTS:	MASSFRAC					
LACTOSE	0	2.25E-07	2.25E-07	2.66E-04	1.31E-11	1.63E-11
WATER	0	9.99E-01	9.99E-01	4.88E-05	1.00E+00	1.00E+00
PROPANE	1	1.13E-03	1.13E-03	1.00E+00	2.83E-04	2.83E-04
OLEIC-01	0	1.56E-21	1.56E-21	0.00E+00	0.00E+00	9.67E-31
CAPROIC	0	1.68E-10	1.68E-10	1.99E-07	2.20E-16	2.80E-15
PALMITIC	0	1.87E-20	1.87E-20	0.00E+00	0.00E+00	2.85E-28
STEQARIC	0	7.61E-21	7.61E-21	0.00E+00	0.00E+00	1.83E-29
MYRISTIC	0	6.19E-21	6.19E-21	0.00E+00	0.00E+00	6.86E-28
LINOLEIC	0	2.50E-21	2.50E-21	0.00E+00	0.00E+00	2.46E-30
LAURIC	0	1.61E-21	1.61E-21	0.00E+00	0.00E+00	1.48E-27
BUTYRIC	0	1.18E-07	1.18E-07	1.40E-04	2.47E-10	2.52E-10
LINOLENI	0	1.56E-21	1.56E-21	0.00E+00	0.00E+00	9.12E-31
CAPRIC	0	7.02E-19	7.02E-19	0.00E+00	0.00E+00	6.47E-25
CAPRYLIC	0	7.49E-14	7.49E-14	8.87E-11	1.34E-23	3.71E-19

Table A-3 Stream summary for Decanter 3 and Flash 1 in Dehydration unit

STREAM ID	TOF1	VAP1	WATER	TOD3	EXT3	RAFF3
Temperature F	348	348	348	29.8719551	30	30
Pressure psia	140	140	140	140	140	140
Vapor Frac	0.0064742	1	0	0	0	0
Enthalpy kcal/kg	-3639.17	-3035.86	-3643.20	-3802.43	-1203.35	-3805.87
Total Flow kg/hr	55924.29	371.06	55553.23	55959.31	4148.43	12.26
COMPONENT	KG/HR					
LACTOSE	9.11E-07	8.23E-09	8.63E-07	1.26E-02	2.77E+03	1.77E-07
WATER	5.59E+04	3.56E+02	5.56E+04	5.59E+04	8.37E-03	1.23E+01
PROPANE	1.58E+01	1.53E+01	5.24E-01	6.31E+01	5.69E+01	4.27E-04
OLEIC-01	5.41E-26	0.00E+00	0.00E+00	8.75E-17	4.51E+01	5.41E-26
CAPROIC	1.57E-10	0.00E+00	0.00E+00	9.41E-06	4.51E+01	1.44E-10
PALMITIC	1.60E-23	0.00E+00	0.00E+00	1.05E-15	5.39E+02	1.60E-23
STEARIC	1.02E-24	0.00E+00	0.00E+00	4.26E-16	2.19E+02	1.02E-24
MYRISTIC	3.84E-23	0.00E+00	0.00E+00	3.46E-16	1.79E+02	3.84E-23
LINOLEIC	1.38E-25	0.00E+00	0.00E+00	1.40E-16	7.21E+01	1.38E-25
LAURIC	8.27E-23	0.00E+00	0.00E+00	8.98E-17	4.63E+01	8.27E-23
BUTYRIC	1.41E-05	6.72E-06	1.15E-05	6.62E-03	4.51E+01	2.65E-07
LINOLENI	5.10E-26	0.00E+00	0.00E+00	8.75E-17	4.51E+01	5.10E-26
CAPRIC	3.62E-20	0.00E+00	0.00E+00	3.93E-14	4.51E+01	3.62E-20
CAPRYLIC	2.08E-14	0.00E+00	0.00E+00	4.19E-09	4.51E+01	2.07E-14
COMPONENT	MASSFRAC					
LACTOSE	1.63E-11	2.22E-11	1.55E-11	2.25E-07	0.66660192	1.44E-08
WATER	0.99971685	0.95873622	0.99999057	0.99887214	2.02E-06	0.99996518
PROPANE	0.00028314	0.04126376	9.43E-06	0.00112751	0.0137171	3.48E-05
OLEIC-01	9.67E-31	0	0	1.56E-21	0.01086855	4.41E-27
CAPROIC	2.80E-15	0	0	1.68E-10	0.01086855	1.18E-11
PALMITIC	2.85E-28	0	0	1.87E-20	0.12998797	1.30E-24
STEQARIC	1.83E-29	0	0	7.61E-21	0.05289365	8.35E-26
MYRISTIC	6.86E-28	0	0	6.19E-21	0.04303949	3.13E-24
LINOLEIC	2.46E-30	0	0	2.50E-21	0.01738969	1.12E-26
LAURIC	1.48E-27	0	0	1.61E-21	0.01115838	6.74E-24
BUTYRIC	2.52E-10	1.81E-08	2.07E-10	1.18E-07	0.01086696	2.16E-08
LINOLENI	9.12E-31	0	0	1.56E-21	0.01086855	4.16E-27
CAPRIC	6.47E-25	0	0	7.02E-19	0.01086855	2.95E-21
CAPRYLIC	3.71E-19	0	0	7.49E-14	0.01086855	1.69E-15

Table A-4 Stream summary for Decanters 4 & 5 in Extraction unit

STREAM ID	TOD4	EXT4	RAFF4	PROP3	TOD5	EXT5	RAFF5
Temperature F	49.6671284	50	50	70	30	3.00E+01	3.00E+01
Pressure psia	140	140	140	140	140	1.40E+02	1.40E+02
Vapor Frac	0	0	0	0	0	0.00E+00	0.00E+00
Enthalpy kcal/kg	-1199.82	-1052.24	-1363.05	657.73	-1367.01	-1038.11	-1369.28
Total Flow kg/hr	4148.43	2179.57	1968.86	0.06	1968.92	15.82	1953.09
COMPONENTS	KG/HR						
LACTOSE	2765.35	955.47	1809.88	0.00	1809.88	6.49E+00	1.80E+03
WATER	0.01	0.00	0.00	0.00	0.00	1.20E-05	3.59E-03
PROPANE	56.90	41.06	15.85	0.06	15.91	3.12E-01	1.56E+01
OLEIC-01	45.09	42.83	2.26	0.00	2.26	3.35E-01	1.92E+00
CAPROIC	45.09	22.03	23.06	0.00	23.06	1.52E-01	2.29E+01
PALMITIC	539.25	513.97	25.27	0.00	25.27	4.05E+00	2.12E+01
STEQARIC	219.43	215.15	4.27	0.00	4.27	1.46E+00	2.82E+00
MYRISTIC	178.55	163.60	14.95	0.00	14.95	1.32E+00	1.36E+01
LINOLEIC	72.14	68.14	4.00	0.00	4.00	5.35E-01	3.46E+00
LAURIC	46.29	37.75	8.54	0.00	8.54	2.94E-01	8.25E+00
BUTYRIC	45.08	20.24	24.84	0.00	24.84	1.37E-01	2.47E+01
LINOLENI	45.09	41.94	3.14	0.00	3.14	3.32E-01	2.81E+00
CAPRIC	45.09	31.91	13.17	0.00	13.17	2.39E-01	1.29E+01
CAPRYLIC	45.09	25.47	19.62	0.00	19.62	1.81E-01	1.94E+01
COMPONENTS:	MASSFRAC						
LACTOSE	6.67E-01	4.38E-01	9.19E-01	0	9.19E-01	4.10E-01	9.23E-01
WATER	2.02E-06	2.19E-06	1.83E-06	0	1.83E-06	7.58E-07	1.84E-06
PROPANE	1.37E-02	1.88E-02	8.05E-03	1	8.08E-03	1.97E-02	7.98E-03
OLEIC-01	1.09E-02	1.97E-02	1.15E-03	0	1.15E-03	2.12E-02	9.84E-04
CAPROIC	1.09E-02	1.01E-02	1.17E-02	0	1.17E-02	9.59E-03	1.17E-02
PALMITIC	1.30E-01	2.36E-01	1.28E-02	0	1.28E-02	2.56E-01	1.09E-02
STEQARIC	5.29E-02	9.87E-02	2.17E-03	0	2.17E-03	9.20E-02	1.44E-03
MYRISTIC	4.30E-02	7.51E-02	7.59E-03	0	7.59E-03	8.31E-02	6.98E-03
LINOLEIC	1.74E-02	3.13E-02	2.03E-03	0	2.03E-03	3.38E-02	1.77E-03
LAURIC	1.12E-02	1.73E-02	4.34E-03	0	4.34E-03	1.86E-02	4.22E-03
BUTYRIC	1.09E-02	9.29E-03	1.26E-02	0	1.26E-02	8.68E-03	1.26E-02
LINOLENI	1.09E-02	1.92E-02	1.60E-03	0	1.60E-03	2.10E-02	1.44E-03
CAPRIC	1.09E-02	1.46E-02	6.69E-03	0	6.69E-03	1.51E-02	6.62E-03
CAPRYLIC	1.09E-02	1.17E-02	9.97E-03	0	9.97E-03	1.14E-02	9.95E-03

Table A-5 Stream summary for extractor and Decanter 6 in Extraction unit

STREAM ID	TOEXT1	BTM1	TOH4	TOD6	RAFF6	EXT6
Temperature F	60	63.121016 2	62.674846 3	69.798065 2	70	70
Pressure psia	140	140	140	140	140	140
Vapor Frac	0	0	0	0	0	0
Enthalpy kcal/kg	-1363.91	-1252.14	-986.13	-984.24	-1226.02	-973.36
Total Flow kg/hr	1953.09	942.90	2510.20	2510.20	107.45	2402.75
COMPONENTS	KG/HR					
LACTOSE	1803.39	746.70	1056.69	1056.69	80.66	976.03
WATER	0.00	0.00	0.00	0.00	0.00	0.00
PROPANE	15.59	177.66	1337.93	1337.93	22.98	1314.95
OLEIC-01	1.92	0.06	1.86	1.86	0.03	1.83
CAPROIC	22.91	5.27	17.64	17.64	0.86	16.78
PALMITIC	21.22	0.46	20.76	20.76	0.31	20.46
STEQARIC	2.82	0.02	2.79	2.79	0.03	2.77
MYRISTIC	13.63	0.52	13.11	13.11	0.25	12.86
LINOLEIC	3.46	0.11	3.35	3.35	0.06	3.29
LAURIC	8.25	0.73	7.52	7.52	0.22	7.30
BUTYRIC	24.70	5.45	19.25	19.25	0.91	18.34
LINOLENI	2.81	0.13	2.68	2.68	0.06	2.63
CAPRIC	12.94	1.76	11.18	11.18	0.40	10.78
CAPRYLIC	19.44	4.01	15.43	15.43	0.70	14.73
COMPONENTS:	MASSFRA					
LACTOSE	9.23E-01	7.92E-01	4.21E-01	4.21E-01	7.51E-01	4.06E-01
WATER	1.84E-06	2.40E-06	5.28E-07	5.28E-07	1.47E-06	4.86E-07
PROPANE	7.98E-03	1.88E-01	5.33E-01	5.33E-01	2.14E-01	5.47E-01
OLEIC-01	9.84E-04	6.11E-05	7.43E-04	7.43E-04	2.95E-04	7.63E-04
CAPROIC	1.17E-02	5.59E-03	7.03E-03	7.03E-03	7.98E-03	6.98E-03
PALMITIC	1.09E-02	4.91E-04	8.27E-03	8.27E-03	2.84E-03	8.51E-03
STEQARIC	1.44E-03	2.55E-05	1.11E-03	1.11E-03	2.51E-04	1.15E-03
MYRISTIC	6.98E-03	5.54E-04	5.22E-03	5.22E-03	2.32E-03	5.35E-03
LINOLEIC	1.77E-03	1.20E-04	1.33E-03	1.33E-03	5.52E-04	1.37E-03
LAURIC	4.22E-03	7.75E-04	3.00E-03	3.00E-03	2.00E-03	3.04E-03
BUTYRIC	1.26E-02	5.78E-03	7.67E-03	7.67E-03	8.44E-03	7.63E-03
LINOLENI	1.44E-03	1.34E-04	1.07E-03	1.07E-03	5.15E-04	1.09E-03
CAPRIC	6.62E-03	1.86E-03	4.45E-03	4.45E-03	3.73E-03	4.49E-03
CAPRYLIC	9.95E-03	4.25E-03	6.15E-03	6.15E-03	6.54E-03	6.13E-03

Table A-5 Stream summary for Decanter 7 in Extraction unit

STREAM ID	TOD7	EXT7	RAFF7	MIX5
Temperature F	100	100	100	73.247596
Pressure psia	200	140	140	140
Vapor Frac	0	0	0	0
Enthalpy kcal/kg	-965.02	-919.38	-1227.14	-1243.82
Total Flow kg/hr	2402.75	2047.08	355.66	1406.01
COMPONENTS	KG/HR			
LACTOSE	976.03	705.64	270.39	1097.75
WATER	0.00	0.00	0.00	0.00
PROPANE	1314.95	1243.08	71.87	272.51
OLEIC-01	1.83	1.71	0.12	0.21
CAPROIC	16.78	13.87	2.90	9.03
PALMITIC	20.46	19.28	1.17	1.94
STEQARIC	2.77	2.66	0.10	0.15
MYRISTIC	12.86	11.91	0.95	1.72
LINOLEIC	3.29	3.06	0.23	0.40
LAURIC	7.30	6.50	0.80	1.75
BUTYRIC	18.34	15.33	3.01	9.37
LINOLENI	2.63	2.41	0.22	0.40
CAPRIC	10.78	9.33	1.45	3.60
CAPRYLIC	14.73	12.28	2.45	7.16
COMPONENTS:	MASSFRAC			
LACTOSE	4.06E-01	3.45E-01	7.60E-01	7.81E-01
WATER	4.86E-07	3.78E-07	1.11E-06	2.01E-06
PROPANE	5.47E-01	6.07E-01	2.02E-01	1.94E-01
OLEIC-01	7.63E-04	8.35E-04	3.48E-04	1.52E-04
CAPROIC	6.98E-03	6.78E-03	8.16E-03	6.43E-03
PALMITIC	8.51E-03	9.42E-03	3.30E-03	1.38E-03
STEQARIC	1.15E-03	1.30E-03	2.91E-04	1.10E-04
MYRISTIC	5.35E-03	5.82E-03	2.67E-03	1.22E-03
LINOLEIC	1.37E-03	1.49E-03	6.47E-04	2.86E-04
LAURIC	3.04E-03	3.18E-03	2.25E-03	1.24E-03
BUTYRIC	7.63E-03	7.49E-03	8.46E-03	6.66E-03
LINOLENI	1.09E-03	1.18E-03	6.08E-04	2.83E-04
CAPRIC	4.49E-03	4.56E-03	4.07E-03	2.56E-03
CAPRYLIC	6.13E-03	6.00E-03	6.88E-03	5.09E-03

Table A-5 Stream summary for Desolventization unit

STREAM ID	TOH6	TOF2	NOFATMIL	VAP2	TOF3	RECPROP	LQD3
Temperature F	32.1935992	270	270	270	1.25E+02	8.00E+01	8.00E+01
Pressure psia	60	60	60	60	6.00E+01	6.00E+01	6.00E+01
Vapor Frac	0.30	0.63	0.00	1.00	1.00	1.00	0.00
Enthalpy kcal/kg	-1243.85	-1185.44	-1341.86	-529.20	-569.05	-569.15	-1320.22
Total Flow kg/hr	1406.01	1406.01	1135.37	270.64	270.64	266.73	3.91
COMPONENTS	KG/HR						
LACTOSE	1097.75	1097.75	1095.96	1.80	1.80	0.00	1.79
WATER	0.00	0.00	0.00	0.00	0.00	0.00	0.00
PROPANE	272.51	272.51	5.56	266.95	266.95	266.64	0.32
OLEIC-01	0.21	0.21	0.21	0.00	0.00	0.00	0.00
CAPROIC	9.03	9.03	8.67	0.37	0.37	0.00	0.36
PALMITIC	1.94	1.94	1.94	0.00	0.00	0.00	0.00
STEQARIC	0.15	0.15	0.15	0.00	0.00	0.00	0.00
MYRISTIC	1.72	1.72	1.72	0.00	0.00	0.00	0.00
LINOLEIC	0.40	0.40	0.40	0.00	0.00	0.00	0.00
LAURIC	1.75	1.75	1.74	0.01	0.01	0.00	0.01
BUTYRIC	9.37	9.37	7.98	1.38	1.38	0.08	1.30
LINOLENI	0.40	0.40	0.40	0.00	0.00	0.00	0.00
CAPRIC	3.60	3.60	3.58	0.02	0.02	0.00	0.02
CAPRYLIC	7.16	7.16	7.06	0.10	0.10	0.00	0.10
COMPONENTS:	MASSFRAC						
LACTOSE	7.81E-01	7.81E-01	9.65E-01	6.63E-03	6.63E-03	1.30E-06	4.59E-01
WATER	2.01E-06	2.01E-06	3.73E-08	1.03E-05	1.03E-05	1.04E-05	1.76E-07
PROPANE	1.94E-01	1.94E-01	4.90E-03	9.86E-01	9.86E-01	1.00E+00	8.08E-02
OLEIC-01	1.52E-04	1.52E-04	1.88E-04	2.98E-07	2.98E-07	6.71E-14	2.06E-05
CAPROIC	6.43E-03	6.43E-03	7.63E-03	1.35E-03	1.35E-03	7.68E-06	9.32E-02
PALMITIC	1.38E-03	1.38E-03	1.71E-03	8.01E-06	8.01E-06	2.26E-11	5.54E-04
STEQARIC	1.10E-04	1.10E-04	1.36E-04	4.74E-07	4.74E-07	5.27E-13	3.28E-05
MYRISTIC	1.22E-03	1.22E-03	1.51E-03	1.28E-05	1.28E-05	1.24E-10	8.82E-04
LINOLEIC	2.86E-04	2.86E-04	3.54E-04	6.77E-07	6.77E-07	2.10E-13	4.68E-05
LAURIC	1.24E-03	1.24E-03	1.53E-03	1.94E-05	1.94E-05	5.94E-10	1.34E-03
BUTYRIC	6.66E-03	6.66E-03	7.03E-03	5.12E-03	5.12E-03	3.15E-04	3.32E-01
LINOLENI	2.83E-04	2.83E-04	3.51E-04	4.34E-07	4.34E-07	7.44E-14	3.00E-05
CAPRIC	2.56E-03	2.56E-03	3.15E-03	8.74E-05	8.74E-05	1.52E-08	6.04E-03
CAPRYLIC	5.09E-03	5.09E-03	6.22E-03	3.75E-04	3.75E-04	3.13E-07	2.59E-02

Table A-5 Stream summary for Flash 4 in Solvent recovery unit

STREAM ID	MIX6	TOH7	TOF4	VAP4	LQD4
Temperature F	79.64	41.81	270.00	270.00	270.00
Pressure psia	140.00	60.00	60.00	60.00	60.00
Vapor Frac	0.00	0.24	0.72	1.00	0.00
Enthalpy kcal/kg	-987.89	-987.93	-914.04	-528.91	-1079.23
Total Flow kg/hr	4226.66	4226.66	4226.66	1268.70	2957.94
COMPONENTS	KG/HR				
LACTOSE	1661.11	1661.11	1661.11	6.90	1654.22
WATER	0.01	0.01	0.01	0.01	0.00
PROPANE	1284.14	1284.14	1284.14	1251.41	32.74
OLEIC-01	44.54	44.54	44.54	0.00	44.54
CAPROIC	35.90	35.90	35.90	1.94	33.96
PALMITIC	533.25	533.25	533.25	0.11	533.15
STEQARIC	217.82	217.82	217.82	0.02	217.80
MYRISTIC	175.51	175.51	175.51	0.09	175.42
LINOLEIC	71.20	71.20	71.20	0.01	71.20
LAURIC	44.25	44.25	44.25	0.06	44.18
BUTYRIC	35.58	35.58	35.58	7.37	28.19
LINOLENI	44.36	44.36	44.36	0.00	44.35
CAPRIC	41.24	41.24	41.24	0.20	41.04
CAPRYLIC	37.75	37.75	37.75	0.59	37.16
COMPONENTS:	MASSFRAC				
LACTOSE	3.93E-01	3.93E-01	3.93E-01	5.44E-03	5.59E-01
WATER	1.31E-06	1.31E-06	1.31E-06	4.32E-06	1.86E-08
PROPANE	3.04E-01	3.04E-01	3.04E-01	9.86E-01	1.11E-02
OLEIC-01	1.05E-02	1.05E-02	1.05E-02	2.22E-06	1.51E-02
CAPROIC	8.49E-03	8.49E-03	8.49E-03	1.53E-03	1.15E-02
PALMITIC	1.26E-01	1.26E-01	1.26E-01	8.41E-05	1.80E-01
STEQARIC	5.15E-02	5.15E-02	5.15E-02	1.41E-05	7.36E-02
MYRISTIC	4.15E-02	4.15E-02	4.15E-02	7.44E-05	5.93E-02
LINOLEIC	1.68E-02	1.68E-02	1.68E-02	4.56E-06	2.41E-02
LAURIC	1.05E-02	1.05E-02	1.05E-02	5.11E-05	1.49E-02
BUTYRIC	8.42E-03	8.42E-03	8.42E-03	5.81E-03	9.53E-03
LINOLENI	1.05E-02	1.05E-02	1.05E-02	2.16E-06	1.50E-02
CAPRIC	9.76E-03	9.76E-03	9.76E-03	1.57E-04	1.39E-02
CAPRYLIC	8.93E-03	8.93E-03	8.93E-03	4.65E-04	1.26E-02

Table A-5 Stream summary for Flash 5 in solvent recovery unit

STREAM ID	TOF5	RECPROP2	LQD5	MILKFAT
Temperature F	130.00	150.00	150.00	270.00
Pressure psia	60.00	60.00	60.00	60.00
Vapor Frac	1.00	1.00	0.00	0.00
Enthalpy kcal/kg	-566.44	-554.77	-1304.79	-1080.20
Total Flow kg/hr	1268.70	1255.88	12.82	1135.37
COMPONENTS	KG/HR			
LACTOSE	6.90	0.05	6.84	1095.96
WATER	0.01	0.01	0.00	0.00
PROPANE	1251.41	1251.01	0.40	5.56
OLEIC-01	0.00	0.00	0.00	0.21
CAPROIC	1.94	0.33	1.61	8.67
PALMITIC	0.11	0.00	0.11	1.94
STEQARIC	0.02	0.00	0.02	0.15
MYRISTIC	0.09	0.00	0.09	1.72
LINOLEIC	0.01	0.00	0.01	0.40
LAURIC	0.06	0.00	0.06	1.74
BUTYRIC	7.37	4.46	2.91	7.98
LINOLENI	0.00	0.00	0.00	0.40
CAPRIC	0.20	0.00	0.20	3.58
CAPRYLIC	0.59	0.02	0.57	7.06
COMPONENTS:	MASSFRAC			
LACTOSE	5.44E-03	4.35E-05	5.34E-01	9.65E-01
WATER	4.32E-06	4.36E-06	7.96E-08	3.73E-08
PROPANE	9.86E-01	9.96E-01	3.11E-02	4.90E-03
OLEIC-01	2.22E-06	1.34E-10	2.20E-04	1.88E-04
CAPROIC	1.53E-03	2.63E-04	1.25E-01	7.63E-03
PALMITIC	8.41E-05	3.32E-08	8.32E-03	1.71E-03
STEQARIC	1.41E-05	2.58E-09	1.40E-03	1.36E-04
MYRISTIC	7.44E-05	8.26E-08	7.36E-03	1.51E-03
LINOLEIC	4.56E-06	3.63E-10	4.51E-04	3.54E-04
LAURIC	5.11E-05	1.45E-07	5.04E-03	1.53E-03
BUTYRIC	5.81E-03	3.55E-03	2.27E-01	7.03E-03
LINOLENI	2.16E-06	1.05E-10	2.14E-04	3.51E-04
CAPRIC	1.57E-04	1.79E-06	1.54E-02	3.15E-03
CAPRYLIC	4.65E-04	1.89E-05	4.42E-02	6.22E-03

VITA

Harita Byluppala

Candidate for the Degree of

Master of Science

Thesis: PROCESS DESIGN AND SIMULATION FOR MILK FAT EXTRACTION
USING LIQUID PROPANE

Major Field: Chemical Engineering

Biographical:

Education: Completed the requirements for the Master of Science in Chemical Engineering at Oklahoma State University, Stillwater, Oklahoma in December, 2010.

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Worked as Teaching Assistant for Thermodynamics, EN GR 2213 in fall 2009.

Worked as Teaching Assistant for Legal Environment of Business, ENGR 5103 in spring 2009.

Name: Harita Byluppala

Date of Degree: December 2010

Institution: Oklahoma State University

Location: Stillwater, Oklahoma

Title of Study: PROCESS DESIGN AND SIMULATION FOR MILK FAT
EXTRACTION USING LIQUID PROPANE

Pages in Study: 95

Candidate for the Degree of Master of Science

Major Field: Chemical Engineering

Scope and Method of Study:

Numerous studies have been conducted to increase the utilization of milk by fractionating the fat. This work examines the use of liquid propane for extraction of lipids from bovine milk. Aspen PlusTM, a commercial process simulator was used to model liquid-liquid extraction of milk fat. The design parameters were obtained based on sensitivity analyses carried out for each unit. The operating conditions were set to provide maximum extraction yield with maximum product purity and solvent recovery.

Findings and Conclusions:

Propane is a feasible solvent for milk fat extraction at sub critical conditions. The process models show that 90 wt % lipid can be extracted from milk using liquid propane at ambient temperature. Pressure has no effect on the extraction process. Results of dehydration step indicate that 99.9 wt % water can be removed from milk using liquid propane. Complete separation of lactose from milk fat cannot be achieved using propane. The procedure to model a liquid-liquid extraction process using Aspen PlusTM was explained in detail.

ADVISER'S APPROVAL: Dr. A. JOHANNES
