Report

On

Lavandin Essential Oil Extraction with Hexane Solvent Recovery

Submitted to

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Executive Summary

The goal of the *Lavandin Essential Oil Extraction with Hexane Solvent Recovery* report is to explore essential oil production methods by designing a lavandin essential oil production plant implementing the solvent extraction method. The design should achieve a product purity with a max value of 25 ppm hexane residue, 15 degree Celsius storage temperature of the oil, and a hexane recovery of at least 98%. Key process assumptions include an oil extraction rate of 22%, and access to 3,700,000 kg/yr of lavandin flowers. If the rate of extraction was halved to 11%, which is within the reported range of extraction rate, the amount of essential oil our plant produces would also half [1]. Since lavandin flowers are our feedstock, a decrease in global production also affects the amount of oil we can produce with the design. Both of these assumptions heavily affect the quantity of oil the plant can produce, and therefore they affects the profitability of this design.

Given these assumptions, the design outlined in this report generates 96 kg/hr of lavandin essential oil, with a hexane presence of 1 ppm and hexane recovery of 99.99%, exceeding process design specifications. The purity of the oil achieved allows for this process to compete in business markets with lavandin essential oils produced with steam distillation, especially because hexane extraction provides greater oil yields for lavandin than steam distillation techniques. Additionally, the proposed process design is flexible to market fluctuations as lavandin flowers can be replaced by jasmine, linden, and others to produce other types of essential oils [2]. From an economic perspective, this lavandin essential oil production design is extremely profitable. It has a 633% rate of return on investment, with a payback period of only 0.2 years.

There are a variety of recommendations to improve the current design. The effect of a more conservative oil yield should be further investigated. The reported yield of lavandin oil extraction was 4-22%, and we assumed the 22% yield as discussed in the Process Assumptions [1]. This has a great effect on the economics of the design, therefore we recommend performing laboratory tests on the oil yield, and analyze economic favorability with the researched yield values.

Another improvement that can be made confronts the lack of historic pricing for the lavandin essential oil. Further investigation of oil pricing data is recommended to establish a more accurate representation of the essential oil product economically. The market analysis can be completed by a consulting firm. Overall, we recommend the design outlined in the *Lavandin Essential Oil Extraction with Hexane Solvent Recovery* report for the production of lavandin essential oil.

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Introduction

For centuries, essential oils derived from plants have been used as a therapeutic agent and in scented products such as perfumes and soaps. One of the most commonly produced essential oils is lavender, which is used in perfume, pharmaceutical, aromatherapy, and specialty foods industries [3]. Typically, steam distillation is used as an extraction method from lavender flowers. Another method used to extract essential oils from flowers and other organic materials is solvent extraction, where the organic matter is added to an organic solvent that separates the oil from the flower. Solvent extraction is a useful method of essential oil extraction because it can operate at lower temperatures than steam distillation, which allows for better preservation of the essential oil properties [4].

This report investigates the utilization and economic viability of a solvent extraction process using hexane as the solvent to extract lavender essential oils. Hexane was chosen due to its availability and price [5]. The species of lavender chosen for this process is hybrid of *lavandula angustifolia* and lavandula latifolia colloquially known as lavandin. Lavandin is cheaper to purchase than true lavender (lavandula angustifolia), and it yields greater amounts of essential oils than true lavender [1].

A preliminary design is outlined in this report. The design specifications are that the mass ratio of hexane to essential oils in the system is 2:1 ensure proper extraction of the oils, and hexane concentration in the final product is 20 ppm or less; the FDA Title 21 regulations for secondary food additives states a value of 25 ppm is the maximum, and was used as a guideline to determine 20 ppm as the target concentration [1]. The proposed design meets these process specifications using desolventizer toasters (modeled as flash drums) and a stripping column, along with various pumps and heat exchangers. The desolventizer toasters vaporize most of the hexane off to reduce the work load of the stripping column, which separates the rest of the hexane from the essential oils. Hexane recovered as a vapor from the desolventizer toasters and the stripping column was condensed and recycled in order to save on operating costs.

The process design was modeled in Aspen Plus, but due to the limitations of the model regarding solid materials, the introduction and disposal of the solid organic matter in the system was not considered. Instead, the model assumed a steady input of liquid essential oils based on an assumed mass yield of 22% [1]. Lavandin essential oil is comprised of over 20 components, however it was assumed for this model that the essential oils were comprised of the four most abundant components. Lastly, the model assumes liquid flow of the essential oils, as it is possible for the oils to form a concrete in the stripping column [1].

Results

Process Specifications

Two key performance variables persist in the Lavender Essential Oils Production process – the purity of lavandin oil product and the recovery of hexane. The process design yields a 99.99% recovery of hexane, with minimal traces of oil components, exceeding design goals of 98% recycle recovery. Performance criteria for product purity of lavandin oil is based on FDA regulation; Title 21 for secondary food additives specifies hexane levels must be under 25ppm [5]. The process design achieves an extremely pure lavandin essential oil, with miniscule hexane presence of 1.07ppm, well below the FDA regulated value. Lastly, the lavandin oil was recommended to be cooled to 10°C for storage [7], however the coldest cooling water stream was specified to be heated to 15°C, so that was decided as the outlet temperature of the lavandin oil product instead, and the process design achieves this temperature.

Process Model Description

The lavandin essential oils production process begins with feeding a hexane stream comprised of recycled and newly fed hexane into a mixing vessel (V-102) containing hybrid *lavandula angustifolia* and *lavandula latifolia* flowers, commonly referred to as lavandin. The hexane separates the lavandin essential oils from the lavandin flower, with a 2:1 mass ratio of hexane to essential oils. The resulting oils are modeled in Aspen with the composition in Table 1.

Table 1 Composition of lavandin essential oil. [1]

Essential Oil Component	Mass Fraction
Cineole	.11
Camphor	.121
Linalool	.439
Linalyl Acetate	.33

From the mixing tank, the stream is pumped to a desolventizer toaster (modeled as heat exchanger E-101 and adiabatic flash drum V-103), which heats the stream to 80 ºC and removes part of the hexane as vapor from the hexane-oil stream. The flash drum bottoms travel to another desolventizer toaster (E-102 and V-104) at 85 ºC. The remaining hexane-oil stream passes through a 14 stage stripping column (T-101), which is the final separation process for the hexane and oil product. The essential oils are collected from the bottom of the column, and the evaporated hexane from the flash drums and column are cooled, mixed, and transported to the hexane storage tank (V-101). Hexane is then transported from the storage tank to the mixing tank, starting the process over again.

BFD

1

PFD

Figure 2 Process Flow Diagram

Process Controls Description

This process is automatically maintained through various control loops. The level of hexane in the system is maintained through a cascade control loop, with a primary flow controller on the inlet hexane feed stream and a secondary flow controller on the recycle stream. The control variable is the hexane recycle flowrate, and the manipulated variable is the inlet hexane feed flowrate. This control loop ensures that a constant amount of hexane is present in the system. The liquid levels in V-101, V-102, V-103, V-104, and T-101 are each controlled with a cascade control loop using a primary level controller and a secondary flow controller to control the outlet liquid flow from the vessel. In these cases, the control variable is the liquid level, and the manipulated variable is the outlet flowrate. These control loops maintain a 10 minute long, 50% full liquid hold-up in each vessel.

The purity of the lavandin essential oils is controlled via a triple cascade control loop with a primary flow controller on the utility condensate stream, a secondary level controller on E-105, and a secondary temperature controller at stage 3. The flow of the condensate is the manipulated variable, and the stage temperature and level of liquid condensate in the tubes of the heat exchanger are the control variables. By manipulating the flow of the condensate, the reboiler duty is manipulated to affect the temperature inside the column, and thus the purity of the product. Stage 3 was chosen because this stage is most affected by changes in temperature (see Figure 4 for the inflection point). E-107 must be a vertical heat exchanger to prevent spaghetti tubes due to thermal expansion.

Stream Summary

Table 2 Stream table summary for streams 4-10

Stream number	4	5	6	7	8	9	10
Temperature (°C)	30.00	29.90	29.92	80.00	80.00	80.00	85.00
Pressure (bar)	1.00	1.00	1.10	1.10	1.10	1.10	1.10
Molar Vapor Fraction	0.00	0.00	0.00	0.09	0.00	0.00	0.32
Mass Density (kg/m³)	1103.40	758.97	758.95	41.75	719.54	719.54	11.93
Total Mass Flow (kg/hr)	100.00	303.83	303.83	303.83	281.54	281.54	281.54
Hexane (kg/hr)	0.00	199.99	199.99	199.99	177.84	177.84	177.84
Cineole (kg/hr)	11.00	12.76	12.76	12.76	12.71	12.71	12.71
Camphor (kg/hr)	12.10	12.63	12.63	12.63	12.60	12.60	12.60
Linalool (kg/hr)	43.90	45.05	45.05	45.05	45.00	45.00	45.00
Linalyl Acetate (kg/hr)	33.00	33.40	33.40	33.40	33.38	33.38	33.38

Table 3 Stream table summary for streams 11-17

Stream number	11	12	13	14	15	16	17
Temperature (°C)	85.00	85.00	93.50	80.00	60.00	85.00	60.00
Pressure (bar)	1.10	1.10	1.10	1.10	1.00	1.10	1.00
Molar Vapor Fraction	0.00	0.00	1.00	1.00	0.00	1.00	0.00
Mass Density (kg/m³)	764.25	764.25	3.15	3.24	625.60	3.20	626.99
Total Mass Flow (kg/hr)	206.89	206.89	106.89	22.29	22.29	74.64	74.64
Hexane (kg/hr)	104.02	104.02	104.02	22.16	22.16	73.82	73.82
Cineole (kg/hr)	12.43	12.43	1.43	0.05	0.05	0.28	0.28
Camphor (kg/hr)	12.45	12.45	0.35	0.03	0.03	0.15	0.15
Linalool (kg/hr)	44.72	44.72	0.82	0.04	0.04	0.28	0.28
Linalyl Acetate (kg/hr)	33.28	33.28	0.28	0.02	0.02	0.10	0.10

Table 4 Stream table summary for streams 18-25

Stream number	18	19	20	21	22	23	24	25
Temperature (°C)	60.00	60.00	60.00	30.00	30.02	208.78	50.00	15.00
Pressure (bar)	1.00	1.00	1.00	1.00	1.10	1.21	1.00	1.00
Molar Vapor Fraction	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mass Density (kg/m³)	626.67	631.32	629.10	658.12	658.10	897.69	1082.76	1118.61
Total Mass Flow (kg/hr)	96.93	106.89	203.83	203.83	203.83	100.00	100.00	100.00
Hexane (kg/hr)	95.98	104.02	199.99	199.99	199.99	0.00	0.00	0.00
Cineole (kg/hr)	0.33	1.43	1.76	1.76	1.76	11.00	11.00	11.00
Camphor (kg/hr)	0.18	0.35	0.53	0.53	0.53	12.10	12.10	12.10
Linalool (kg/hr)	0.33	0.82	1.15	1.15	1.15	43.90	43.90	43.90
Linalyl Acetate (kg/hr)	0.12	0.28	0.40	0.40	0.40	33.00	33.00	33.00

Streams 1, 2, and 3 are absent in the Aspen model because the inlet hexane feed (stream 1) is significantly small enough to consider it negligible. The compositions of streams 2 and 3 have the same composition as the hexane recycle stream (stream 22).

Utility Summary

Utility costs were obtained from multiple sources. The costs for cooling and chilled water came from Project 2 from Dr. Paskach, and those are as follows: \$12/1000m^3 of cooling water and \$150/1000m^3 of chilled water. Low pressure steam and electricity costs came from Capcost and are as follows: \$1.9E-6 per kJ of energy from low pressure steam, and \$.0775 per kWh of electricity. The cost of high pressure steam came from Turton, and that is \$29.29/1000 kg [6] [7].

Table 5 Utility usage for heat exchangers

Equipment	Utility	Flow Rate (kg/hr)	Cost ($/hr)
E-101	Low Pressure Steam	21.90	0.084
E-102	Low Pressure Steam	13.70	0.052
E-103	Cooling Water	202.01	0.002
E-104	Cooling Water	693.50	0.008
E-105	Cooling Water	1034.52	0.012
E-106	Chilled Water	358.38	0.054
E-107	High Pressure Steam	38.84	1.164
E-108	Cooling Water	925.49	0.011
E-109	Chilled Water	177.72	0.027

Table 6 Utility usage for pumps

Equipment	Utility	Usage (kW)	Cost ($/hr)
PMP-101 A/B	Electricity	0.76	0.059
PMP-102 A/B	Electricity	0.98	0.076
PMP-103 A/B	Electricity	0.96	0.074
PMP-104 A/B	Electricity	0.66	0.051
PMP-105 A/B	Electricity	0.76	0.059

Table 7 Utility costs, usage, and summary

Utility	Cooling Water	Chilled Water	Low Pressure Steam	High Pressure Steam	Electricity
Cost ($/hr)	0.03	0.08	0.14	1.16	0.32
Mass Flow (kg/hr)	2855.52	536.10	35.60	38.84	N/A
Energy Usage (kW/hr)	N/A	N/A	N/A	N/A	4.12
Inlet Temperature (°C)	30.00	5.00	179.94	250.00	N/A
Outlet Temperature (°C)	40.00	15.00	179.94	249.00	N/A
Inlet Pressure (bar)	1.01	1.01	10.01	39.75	N/A
Outlet Pressure (bar)	1.01	1.01	10.01	39.09	N/A
Inlet Vapor Fraction	0.00	0.00	1.00	1.00	N/A
Outlet Vapor Fraction	0.00	0.00	0.00	0.00	N/A

Equipment Summary

Table 8 Heat exchanger equipment table

<u>Heat Exchangers</u>	
E-101	**E-102**
A = 2 m^2	A = 2 m^2
Type: Floating head, shell-and-tube, single pass	Floating head, shell-and-tube, single pass
Shell: Process stream, carbon steel, boiling liquid	Shell: Process stream, stainless steel, boiling liquid
Tube: Utility stream, carbon steel, condensing vapor	Tube: Utility stream, carbon steel, condensing vapor
Q = 44.1 MJ/hr	Q = 27.6 MJ/hr
Maximum pressure rating = 13.4 bar	Maximum pressure rating = 13.4 bar
E-103	**E-104**
A = 2 m^2	A = 2 m^2
Floating head, shell-and-tube, single pass	Floating head, shell-and-tube, single pass
Shell: Process stream, carbon steel, condensing vapor	Shell: Process stream, carbon steel, condensing vapor
Tube: Utility stream, carbon steel, liquid	Tube: Utility stream, carbon steel, liquid
Q = 8.4 MJ/hr	Q = 28.9 MJ/hr
Maximum pressure rating = 4.4 bar	Maximum pressure rating = 4.4 bar
E-105	**E-106**
A = 2 m^2	A = 2 m^2
Floating head, shell-and-tube, single pass	Floating head, shell-and-tube, single pass
Shell: Process stream, carbon steel, condensing vapor	Shell: Process stream, carbon steel, liquid
Tube: Utility stream, carbon steel, liquid	Tube: Utility stream, carbon steel, liquid
Q = 43.2 MJ/hr	Q = 15 MJ/hr
Maximum pressure rating = 4.4 bar	Maximum pressure rating = 4.4 bar
E-107	
A = 1 m^2	A = 2.3 m^2
Kettle reboiler, shell-and-tube, single pass	Floating head, shell-and-tube, single pass
Shell: Process stream, carbon steel, boiling liquid	Shell: Process stream, carbon steel, liquid
Tube: Utility stream, carbon steel, condensing vapor	Tube: Utility stream, carbon steel, liquid
Q = 66.8 MJ/hr	Q = 38.6 MJ/hr
Maximum pressure rating 45.7 bar	Maximum pressure rating = 4.4 bar
E-109	
A = 2 m^2	
Kettle reboiler, shell-and-tube, single pass	
Shell: Process stream, carbon steel, liquid	
Tube: Utility stream, carbon steel, liquid	
Q = 7.4 MJ/hr	
Maximum pressure rating 4.4 bar	

Table 9 Pump, tower, and vessel equipment table

<u>Pumps</u>	
P-101 A/B Centrifugal/electric drive Carbon Steel Power = 0.76 kW Efficiency = 75% Pressure out = 2.1 bar	**P-102 A/B** Centrifugal/electric drive Stainless Steel Power = 0.98 kW Efficiency = 75% Pressure out = 2.1 bar
P-103 A/B Centrifugal/electric drive Carbon Steel Power = 0.96 kW Efficiency = 75% Pressure out = 2.1 bar	**P-104 A/B** Centrifugal/electric drive Carbon Steel Power = 0.66 kW Efficiency = 75% Pressure out = 2.1 bar
P-105 A/B Centrifugal/electric drive Carbon Steel Power = 0.76 kW Efficiency = 75% Pressure out = 2.1 bar	
<u>Towers</u>	
T-101 Hexane Stripping Column Carbon Steel 14 Stages including reboiler 0.6096 m spacing per tray Column height = 9.7 m Diameter = 3.2 m Maximum pressure rating = 4.4 bar	
<u>Vessels</u>	
V-101 Horizontal Hexane Holding Tank Carbon Steel Length = 0.39 m Diameter = 0.13 m Maximum pressure rating = 4.4 bar	**V-102** Horizontal Hexane and Essential Oil Mixing Tank Carbon Steel Length = 0.52 m Diameter = 0.17 m Maximum pressure rating = 4.4 bar
V-103 Vertical Desolventizer Toaster Stainless Steel Length = 0.50 m Diameter = 0.17 m Maximum pressure rating = 4.4 bar	**V-104** Vertical Desolventizer Toaster Carbon Steel Length = 0.34 m Diameter = 0.11 m Maximum pressure rating = 4.4 bar

The process setup was decided based on an existing setup for solvent extraction [4]. The stripping column (T-101) was rigorously designed in Aspen. Though the hydraulic plots provided by Aspen show that the column can run safely for its calculated diameter of 0.124 m, the ratio between the column height and column diameter is 78.4:1, much higher than the ratio of 3:1 recommended in Turton as Rule 4 in Table 11.6 [7]. The diameter of the column was adjusted to match the recommended ratio in order to ensure column integrity will be sustained. The number of stages was determined through an optimization process for purity versus equipment cost, and afterwards was adjusted to ensure an extremely low concentration of hexane in the product stream in order to provide a safety buffer in the event of a process upset and to provide a more competitive product (see Process Optimization, page). The inlet stage was chosen to be stage 1 in order to maximize the time for mass transfer to occur. The hydraulic plots are provided in Figure 3, and the temperature and composition profiles for the stripping column are provided in Figure 4 and Figure 5.

All of the heat exchangers were designed and sized using Aspen EDR, and all but the column reboiler (E-107) were determined to be floating head exchangers in order to handle thermal expansion of the materials passing through the shell. E-107 is a kettle reboiler oriented vertically so the product composition control strategy can function properly. The utility streams are all on the tube side of each exchanger and the process streams are on the shell side of each exchanger, either to ensure the exchanger can deal with the thermal expansion or contraction of the process stream, or to reduce equipment costs by having the higher pressure utility stream run through the tubes. All the chosen material of construction for all process equipment is carbon steel, as all materials are compatible with it and because it is a sturdy, safe material for the high pressure sections of the process. The maximum operating pressures for the process equipment were determined using Rule 2 in Table 11.7. Further heuristics used for sizing the pumps, stripping column, and the process vessels are located in the Appendix.

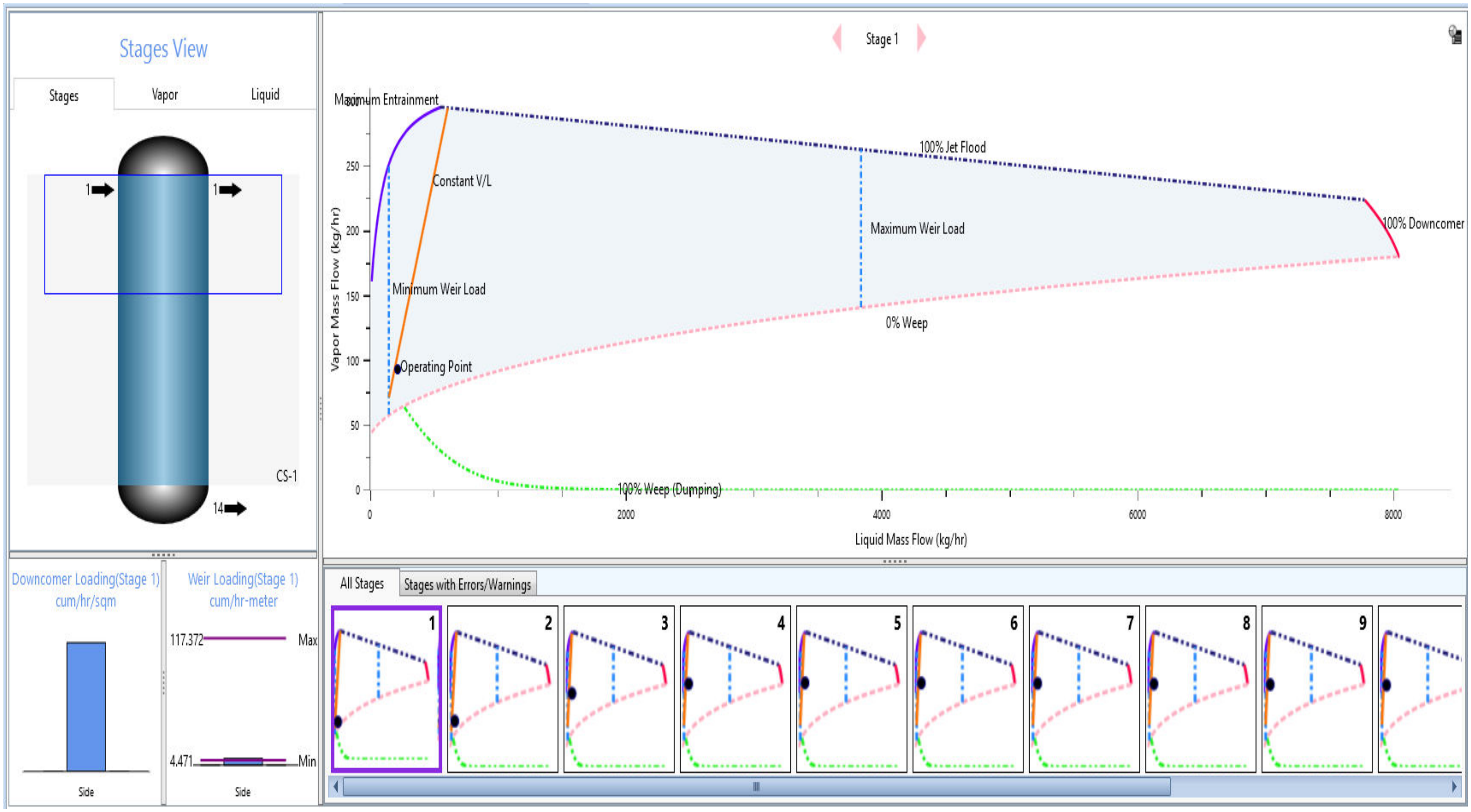

Figure 3: Hydraulic plots for the stripping column.

Column Profiles

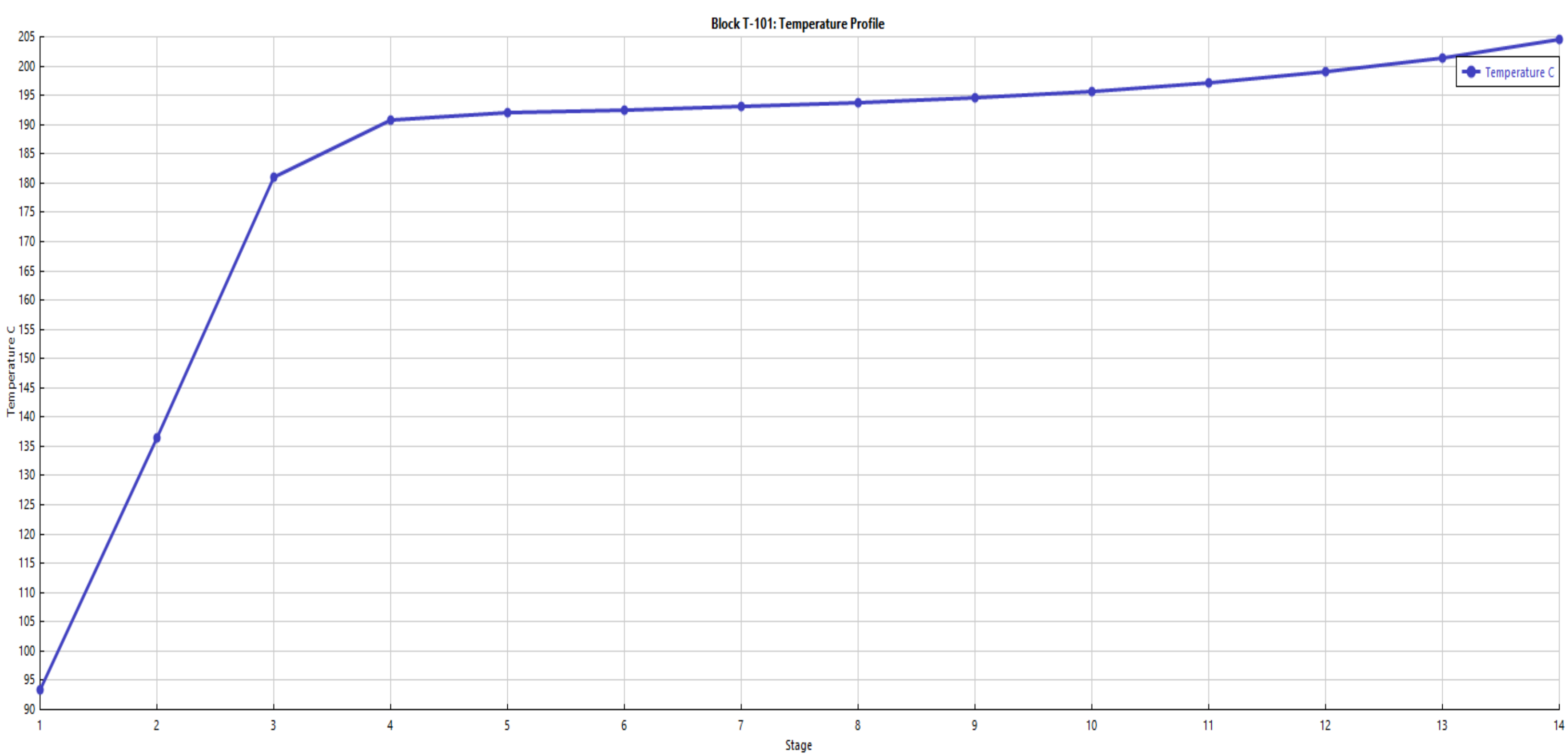

Figure 4: Temperature profile of the stripping column.

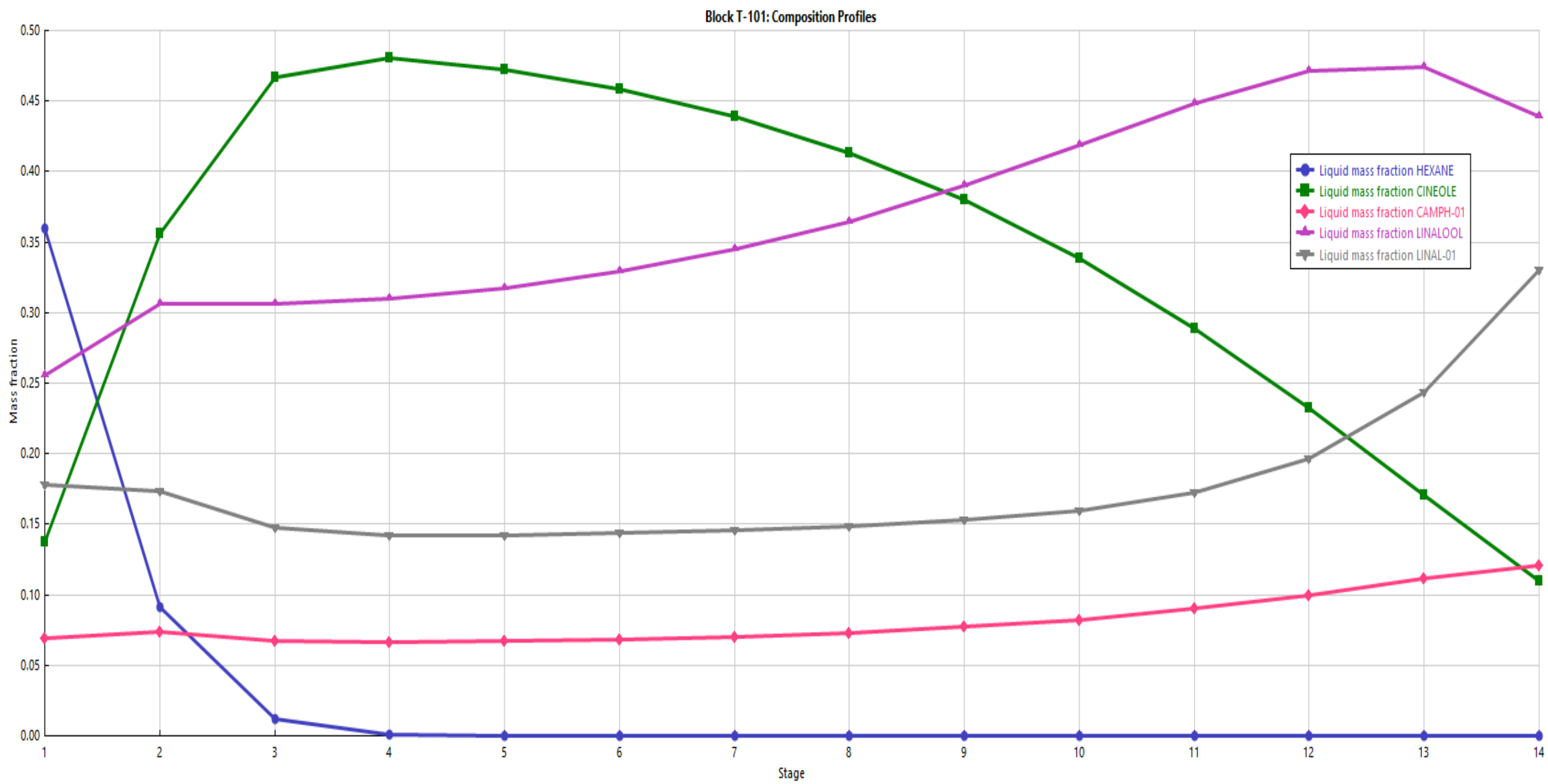

Figure 5: Composition profile of the stripping column.

Discussion

Process Assumptions

There are 48 species of lavender with hundreds of different genotypes and each of these lavender species has a specific chemical composition. It was assumed for the Lavender Essential Oils Production process that the species used is *L. angustifolia x L. latifolia* specifically, also known as lavandin [1]. Using this assumption, the major chemical components of lavandin, shown in Table 1, were modeled in ASPEN.

One aspect of lavandin that was not modeled was the solid flower itself. We did not account for plant down-time during routine maintenance when the lavandin flower would need to be replaced, nor costs associated with the disposal and removal of the flower. We also assumed a proper flow of oil in all parts of the process, and did not account for potential lavandin concretes being produced. Concretes are produced during solvent extraction, and can be considered a 'side reaction.' Concretes have different use cases than the essential oil, and require additional processes to produce *absolutes*, which are a valuable ingredient in perfume [1]. By assuming these concretes were not produced, we did not account for the processes required for the refinement of lavandin concretes. Without the lack-of-concretes assumption, we would have had to implement the collection of the concretes after the hexane-oil separation column. The concretes require cooling, filtering, and additional ethyl alcohol extraction to produce the absolute product [1].

Other assumptions related to the feed stock of lavandin leaves. The country of Bulgaria produces 19,000,000 kg/yr of flowers according to 2015 reports [8]. We assumed that we could purchase 20% of the crop – 3,800,000 kg/yr. With an extraction rate of 22%, we'd be able to produce 836,000 kg/yr of oil [1]. These assumptions were the basis for our oil production rate of 100kg oil/hr – a conservative estimate.

The rate of extraction was assumed to be 22%, which is the high estimate for oil yields. If the lowest estimate of 4% was assumed instead, we'd only be able to produce 174,800kg of oil [1]. Access to lavandin crop is also very important to our process design. Even though lavender is one of the most common essential oil-bearing crops in the world, global flower production can vary drastically due to weather and long-standing diseases and pests [8]. If lavandin production in Bulgaria decreased by only 10% in a given year (sustaining the assumption of a 20% stake), the amount of oil we'd be able to produce would be 865,2610 kg/yr. This would cause oil production design specifications to decrease from 100kg/hr to 97kg/hr, and would create cascading effects in economic results as well. Therefore, the economics of our design relies heavily on the assumption of 22% oil extraction and the availability of lavandin flowers in the global market.

Thermodynamic Package Justification

Initially, interactions for both systems were investigated using the azeotrope search analysis feature in Aspen to evaluate what kinds of models should be used for the simulation. The azeotrope search, performed at a pressure of 1 bar, yielded no azeotropes for the system as seen in Figure 3. No azeotrope was found in the system at the given pressure, and no further pressure changes and temperature variations were necessary to achieve specifications.

AZEOTROPE SEARCH REPORT

Physical Property Model: NRTL **Valid Phase:** VAP-LIQ

Mixture Investigated For Azeotropes At A Pressure Of 1 BAR

Comp ID	Component Name	Classification	Temperature
HEXANE	N-HEXANE	Unstable node	68.31 C
CINEOLE	1,8-CINEOLE	Saddle	176.10 C
CAMPH-01	CAMPHOR	Saddle	206.82 C
LINALOOL	C10H18O	Saddle	197.57 C
LINAL-01	LINALYL-ACETATE	Stable node	220.40 C

No Azeotropes Were Found

Figure 6 Azeotrope search analysis for Lavandin oil components and Hexane

Different thermodynamic packages were intended to be modeled for the two binary mixtures of Linalool/Hexane and Linalyl acetate/Hexane, in order to determine the model that most closely predicted the behavior of the mixtures in the stripping column. Experimental data in the NIST database was not available for neither of the oil component interactions with Hexane, which resulted in no possible comparison of theoretical data from binary analysis with different models. Further experiments to obtain data for the lavandin system will greatly improve the accuracy of the process when compared to specific models.

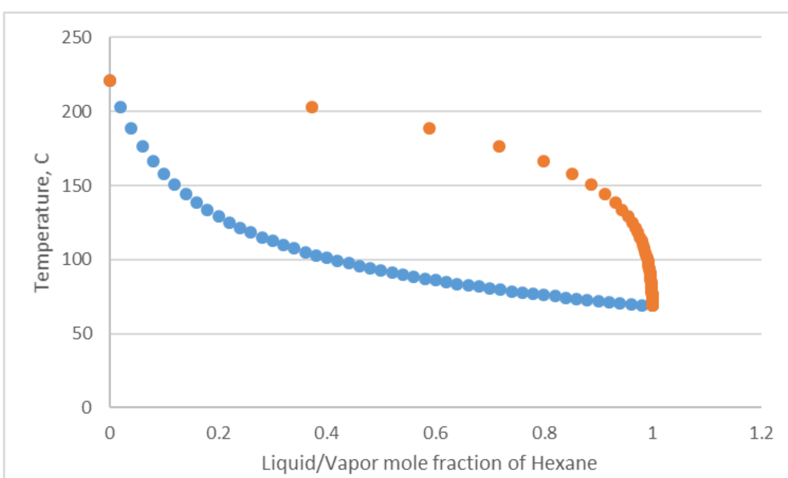

Figure 7: Txy diagram for Hexane and Linalool interactions using the NRTL model.

When considering the selection of thermodynamic models, NRTL was chosen due to the presence of liquid-liquid interactions in the system as well as the use of activity coefficients to predict interactions between the compounds [9] Figure 4 was obtained using the NRTL model for Hexane and Linalyl acetate. Figure 5was obtained from theoretical data with the same model, NRTL, for Hexane and Linalool.

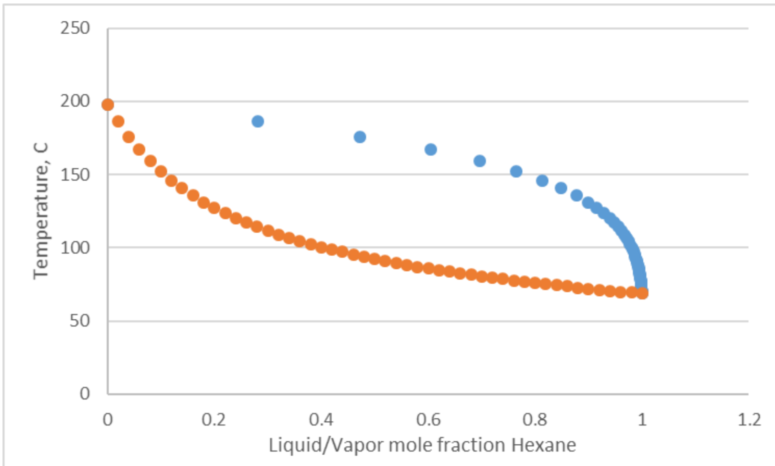

Figure 8 Txy diagram for Hexane and Linalyl acetate interactions using the NRTL model.

Experimental data obtained from thermodynamic experiment for the major oil components should be compared to the data in the previous figures to verify the accuracy of the model used in the simulation. Other potential options for models include UNIQUAC and UNIFAC, which would be potentially helpful in adding binary coefficient parameter interactions.

Plant Optimization

The given plant design includes a Capital Cost vs Product Purity optimization. When analyzing the plant design, the separation column was the largest capital cost, wherefore optimizing the column capital cost would have the largest impact on the plant as a whole. The manipulated design variable for the optimization was therefore the number of column stages. The economic objective function contains the measurement of product purity, given in parts-per-million of hexane present in the oil product. The primary constraint of the product purity is also a performance variable, where hexane presence in the essential oil must be under 25ppm. The overall function for measuring Hexane ppm vs column stages is given by the exponential function $y = 5 * 10^6 * e^{-1.106x}$, seen in Figure 6. This function is an accurate representation of the data, with $R^2 = 0.9911$. Applying the FDA mandated hexane value of 25ppm to our function yields a value of 11.04 stages.

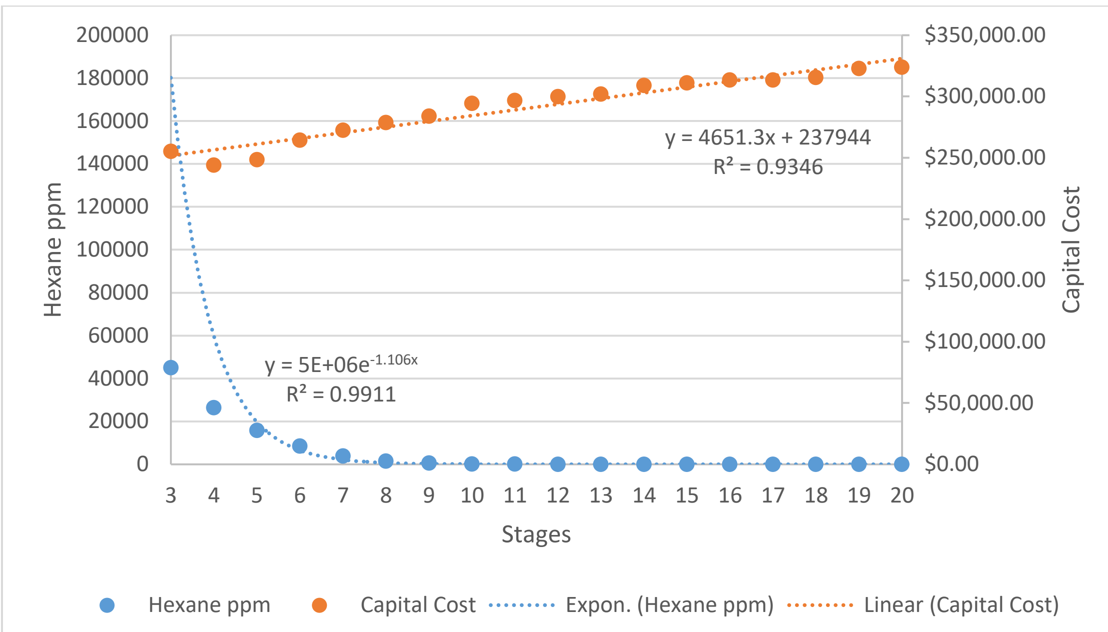

Figure 9 Oil product purity, measured in hexane parts-per-million, and capital cost of a column with 3-20 total stages.

However, when looking closer at the data for 11 Stages in Figure 7, the value for Hexane ppm is closer to 40 instead of 25. Therefore, a new function specifically for stages 11 through 15 was calculated to be $y = 3 * 10^7 * e^{-1.225*x}$ which is shown in Figure 7. This function has an even more accurate representation of data, with $R^2 = 1$. Applying the hexane condition to this updated equation yields 11.43 stages, which is rounded up to 12. Therefore, implementing twelve stages achieves the minimum FDA regulated lavender essential oil purity of 25ppm hexane.

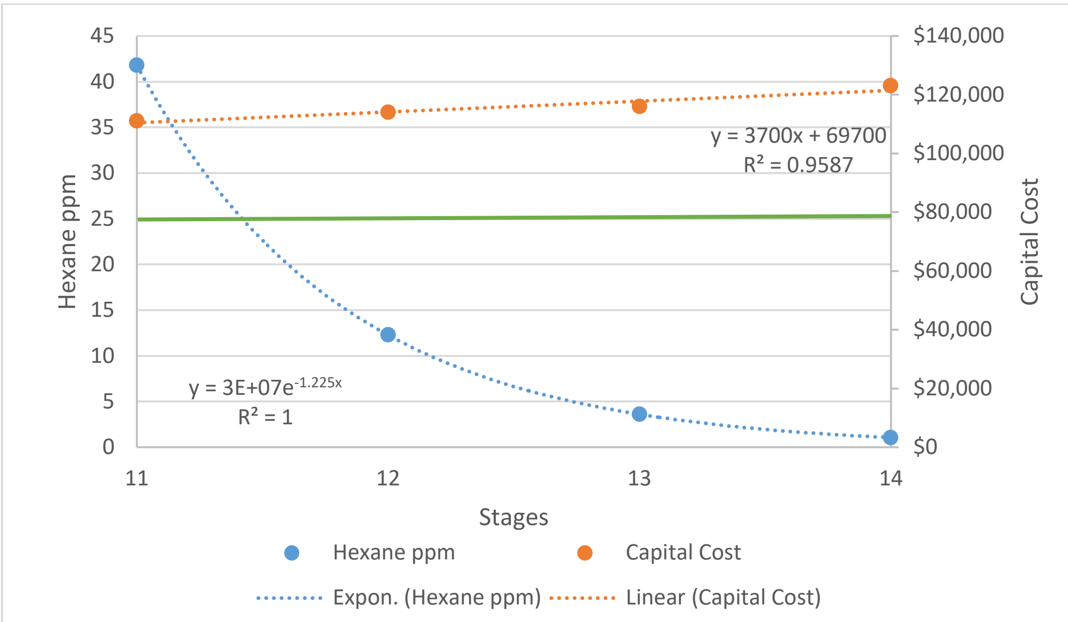

Figure 10: Oil product purity, measured in hexane parts-per-million, and capital cost of a column with 11-14 total stages.

The capital costs of the columns that meet product purity specifications are shown in Table 10. While implementing a twelve stage column would put our design within purity specifications, there is still a significant presence of hexane in the product. Evaluating the capital cost equation calculated in Figure 7, $y = 3700x + 69700$, we see that the column capital costs are relatively similar. Therefore, we decided to implement a fourteen stage column instead of twelve. Fourteen stages achieves hexane values of 1ppm, which is a much more conservative value. Additionally from a marketability perspective, the less hexane present in the product essential oil, the more marketable it will be.

Table 10 Column capital costs for column stages that are within product purity design specifications

Stage	Column Capital Cost (USD)	Hexane ppm
11	111,000	41.79246
12	114,000	12.27717
13	116,000	3.598097
14	123,000	1.056206

Utility Usage and Economics
Operating Costs

Several assumptions are used when completing the analysis of the operating costs. The cost of utilities are assumed to be the values from Table 11. The values were taken from Aspen, Turton, or was provided to us by Dr. Paskach. The cost of lavender flowers was found from Bulk Apothecary [10]. The price is the most realistic since they are providing large quantities causing the price to lower. Pump efficiencies are assumed to be 75%. The pumps we have are small and will not need to generate much pressure, so a higher value from Turton was taken as the efficiency of the pumps [7].
The utility and material costs are shown below in Table 11. The usage of each unit op is shown in Appendix A.

Table 11 Utility and material costs.

	High Pressure Steam	Low Pressure Steam	Electricity	Chilled Water	Cooling Water	Lavender
Cost per unit	\$17.43/GJ	\$1.9/GJ	\$0.06/kWh	\$150/1000 m^3	\$12/1000 m^3	\$12.56/kg

The largest operating cost besides raw material that is needed for the extraction of lavandin oil is the cost of operating labor. The yearly cost for operators was calculated using CAPCOST. The number of operators required is based on the amount and type of equipment. The labor costs, $105,800 associated with this project are over 10 times the utility costs, $14,517. Future analysis on market studies are important because if the throughput of the plant is increased, larger equipment can be purchased which will not increase the operating labor costs.

Utility usage and costing rates were taken from Aspen. The operating costs per hour were calculated by dividing the yearly operating labor by the number of hours in operation. The cost per kilogram is calculated by taking the hourly costs divided by the production in kilograms per hour. The values can be seen in Table 12 below.

Table 12: Estimated operating costs.

Utility ID	Costing rate	
	\$/hr	\$/kg
Cooling Water	.11	0.0011
Electricity	.33	0.0033
Operators	12.71	.1271
Lavender	1256	12.56
Steam	1.3	.013
Total	\$1270.45	\$12.70

The plant is assumed to be in operation for 8,322 hours a year, and produces 100 kilograms of lavandin oil per hour. The plant is assumed to produce 832,200 kilograms of oil per year. The selling cost of oil is assumed to be $37.82 per kilogram. The total cost per kilogram of oil is calculated by taking the COMd divided by the total amount of oil produced per year. This comes out to be a cost of $16.18 per kilogram of oil produced. The taxable income per kilogram is found by subtracting the selling price by the cost per kilogram. The taxable income per kilogram is $21.64. For the year the plant is estimated to earn $18,008,808 in taxable income. The price per kilogram is higher because CAPCOST uses multiplying values for operating costs. This value is higher than the expected total, as this does not include maintenance costs, lab costs, supervisory or clerical labor, controls, permits, etc.

Capital Costs

CAPCOST was used for estimations of the capital costs for the equipment used. A CEPCI value of 581 was used. CAPCOST does not estimate pipe or installation costs, but allows to see if the project is worth pursuing at all. CAPCOST calculates data on economic return. The cost of land is assumed to be $250,000. Other assumptions used are that the plant will take one year to build, and the sixth tenth rule applies to the purchased equipment cost, bare module cost, and grass roots cost for equipment too small for CAPCOST. Lastly, there is assumed to be no salvage cost.
Since the taxable income is approximately $21 million, a marginal tax rate of 42% is used in CAPCOST. For equipment that was too small for CAPCOST, the sixth tenth rule was used to calculate the purchased equipment cost, bare module cost, and grass roots cost for equipment. The sixth tenth rule is shown from Equation 1:

$$\mathrm{Cp},2 = \mathrm{Cp},1\left(\frac{S2}{S1}\right)^{0.6} \tag{1}$$

Where: $C_{P,i}$ = cost of the equipment i
S_i = size of the equipment i

Since the size of the equipment is smaller it will produce a lower cost than estimated by CAPCOST. The cost of the equipment could be higher than estimated, so it will need to be looked into by talking to vendors.

The total module cost estimation is $1,930,000 and the grass roots cost is estimated at $1,640,000. These can be seen in Table 13.

Table 13: Purchase costs, bare module costs, and grass roots costs.

Equipment	Equipment costs ($)	Bare module cost ($)	Grass root cost ($)
E-101	11,079	36,702	61,678
E-102	11,079	36,702	61,678
E-103	11,079	36,436	61,298
E-104	11,079	36,436	61,298
E-105	11,079	36,436	61,298
E-106	11,079	36,436	61,298
E-107	7,912	26,626	44,460
E-108	12,048	39,623	66,660
E-109	11,079	36,436	61,298
PMP-101 A/B	6,081	24,258	38,423
PMP-102 A/B	7,084	28,255	44,754
PMP-103 A/B	6,997	27,908	44,204
PMP-104 A/B	5,588	22,289	35,304
PMP-105 A/B	6,081	24,258	38,423
T-101	179,000	577,000	890,000
V-101	238	717	1,208
V-102	394	1,187	2,000
V-103	395	1,610	2,697
V-104	181	738	1,237
Totals	310,000	1,030,000	1,640,000

Table 14 shows the non-discounted profitability criteria. The cumulative cash position is the sum of all the cash flows. The rate of return on investment is the percentage that the cash flows can be discounted, so the net present value is zero. The payback period is the amount of years until the initial investment is paid back.

Table 14: Non-discounted profitability criteria.

Cumulative Cash Position (millions)	155.75
Rate of Return on Investment	633.03%
Payback Period (years)	0.2

Table 15 shows the discounted profitability criteria. The net present value is the present value of all of the cash flows over the span of the project. The span of our project is 15 years, and the cash flows are discounted at 3%. The discounted cash flow rate of return, shows the return after the cash flows have been discounted. The discounted payback period is the amount of years until the initial investment is paid off while discounting the cash flows.

Table 15: Discounted profitability criteria.

Net Present Value (millions)	119.60
Discounted Cash Flow Rate of Return	280.17%
Discounted Payback Period (years)	0.2

The cash flow diagram is seen in Figure 11. The cash flow diagram shows the cumulative cash position for each year. There is assumed to be a single investment made.

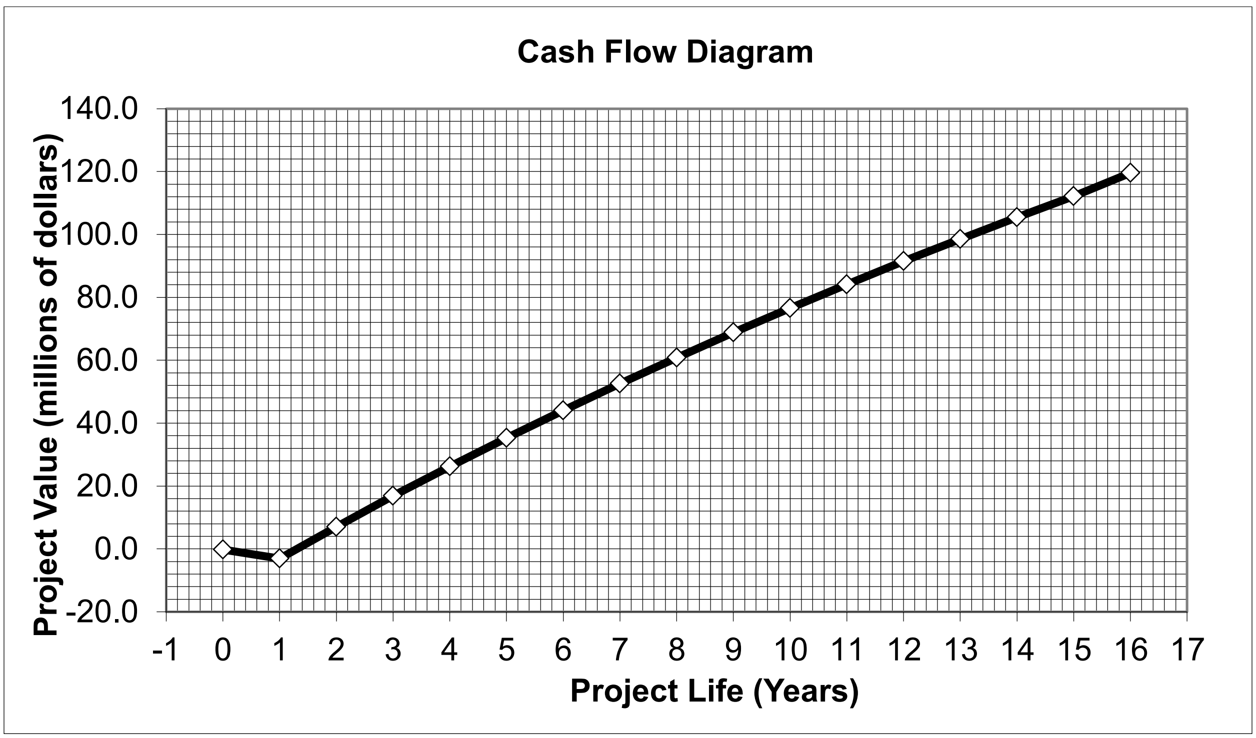

Figure 11: Cash flow diagram.

The cash flows CAPCOST calculated are provided in Appendix C. To account for the variability of costing, a Monte Carlo simulation was run. The high values of costing typically ranges from 10% to 30%, but for the raw material price a value of 100% is used. There is not much historical data for the cost of lavender flowers. Since there is not much data, the variability is very high. The low end values will range from 10% to 20%. The high and low end are expected to be at these values while a class 3 economic evaluation as provided by Dr. Paskach [6]. The ranges for the product and the raw materials are supposed to be determined with triangle plots, but there was not enough data on them to produce a triangle plot. Since there was not information readily available, a consulting firm should be contacted to perform a more in depth market analysis. The high and low values for the product were calculated by using the current price of lavandin oil and taking the percentage of when it was the lowest for the low end, and the high end uses plus 20% because it is a class 3 evaluation. A Monte Carlo simulation will find probabilities of economic values, as it performs the analysis at different values based on high and low ends many times. The values of the high and low ends are shown in Table 16.

Table 16 Monte Carlo variabilities

	Lower Limit	Upper Limit	Base Value
FCIL	-20%	30%	$ 1,640,320
Price of Product	-20%	20%	$ 31,473,804
Working Capital	-50%	10%	$ 1,220,000
Income Tax Rate*	-20%	20%	42%
Interest Rate*	-10%	20%	3%
Raw Material Price	-20%	150%	$ 10,452,432
Salvage Value	-80%	20%	0

The Monte Carlo simulation was performed on the discounted rate of return and the discounted payback period. The Monte Carlo simulations for the discounted rate of return and the discounted payback period are located in Appendix C.

The Monte Carlo simulation for the discounted cash flow rate of return shows that the low is 4% and the high is 397%. The hurdle rate for the project is 15%. When comparing the hurdle rate to the discounted cash flow rate of return if the discounted cash flow rate of return is greater than the hurdle rate the project is acceptable. The Monte Carlo simulation shows that the cumulative times that it fell below 15% was 24 times out of 986. The project is under the hurdle rate 2.4% of the time. Being such a low value the probability of having an acceptable project is very high.

The Monte Carlo simulation for the discounted payback period shows that the low is .1 years and the high value is 8.8 years. The lower the discounted payback period the faster the project is paid off. The first bin of the simulation goes to 1 year, and 937 out of 986 of the simulations fell in bin 1. This shows that 95% of the simulations had a discounted payback period of a year or less. The simulation shows that 99% percent of the simulations are paid off by at least 1.8 years.

Safety

In order to ensure safe operation of this process design multiple precautions were implemented. The automated process control system strategies previously described ensure operation levels for the process are met and extraneous conditions are noted. Because of the highly flammable and toxic compounds used and the temperature used in the process (250 C), alarms attached to each controller are extremely necessary to notify operators if the controlled parameter exceeds a specified range, and adjust parameters in order to avoid dangerous temperature and pressure values that could lead to leakage and explosive conditions. Overall, the lavandin extraction is an unpressurized process, using 1-1.1 bar for all unit operations. Pressure relief systems seem necessary in the stripping column, where HPS is used for the reboiler. Use of carbon steel in the process ensures a good chemical compatibility for Hexane and essential oil components [11].

Hexane presents the most safety concerns in the process. With a NFPA fire rating of 3, Hexane is a highly flammable compound that can be easily ignited at normal conditions. Due to this extreme flammability, equipment should be electrically grounded to avoid static charges. Camphor, Eucalyptol, Linalyl acetate and Linalool have a NFPA fire rating of 2, and are slightly less flammable than Hexane. Only non-sparking tools should be available to operators to use. In case a fire develops, vessels containing Hexane should be cooled down with water and the entire process should be shut down to avoid possibilities of flashback (since this compound is used in a recycle stream). Hexane presents a flammability range of 1.1%-7.5% by volume. Proper ventilation is necessary to avoid these limits and prevent further vapor explosions and dangerous carbon oxide levels in the area (Hazardous decomposition of Linalyl acetate and Linalool) [12] [13]. Dry carbon dioxide, dry chemicals or foam should be available on site for firefighting procedures [14]. Water can be used to knock off vapors however direct water spray should be avoided as it could result in spreading of fire in the area [15].

Hexane is highly irritative to the skin, eyes and respiratory tract. Direct exposure to Hexane and Linalyl acetate causes chronic nerve damage, central nervous depression and peripheral neuropathy hence NIOSH regulations for exposure limits (10 hours for 50 ppm) should be closely followed for operator safety. Proper personal equipment should be used by operators at all times. Camphor, Eucalyptol and Linalool have a NFPA health rating of 2. Skin, eye and respiratory tract irritation can occur, therefore Eye wash stations and showers need to be accessible in case of exposure to these compounds [12] [16] [17] [18].

A HAZID analysis was performed to better understand the risks associated with the oil components and Hexane. When considering all the risks involved in the project, shown in Table 17, Hexane is the compound that causes most threats for the plant design.

Table 17: List of threats, hazardous events and possible consequences for every compound in the Lavandin oil extraction process.

Compound	Threats	Hazardous events	Consequences
	Toxicity	Toxic releases	Chronic illness: nerve damage/CNS depresion
	Extreme Flammability	Fire/Explosion	Reprouctory disease
Hexane	Explosivity	Flashback reactions	Dermatitis
	Spillage	Inhalation/skin contact	Property loss
	Health hazards	Environmental: Volatilization/Adsorption	Operator fatalities
Eucalyptol	Health hazards	Skin contact	Skin irritation
	Flamability	Fire	Property loss
	Inhalation hazard	Inhalation	Fatalities/Injuries
	Flammability	Fire	Property loss
Camphor	Aquatic toxicity	Release to water stream	Toxic to aquatic environments
	Health hazard	Skin contact	Skin Irritation/Sensitation
	Explosivity	Explosion	Toxic combustion products release
	Inhalation hazard	Inhalation	Repiratory tract irritation
	Health hazards	Skin contact	Skin irritation
Linalool	Spillage	Eye contact	Eye irritation
	Flammability	Combustion	Hazardous decomposition products: Carbon Oxides
	Health hazards	Inhalation	Repiratory tract irritation
Linalyl Acetate	Flammability	Skin contact	Severe skin irritation
	Spillage	Eye contact	Eye irritation
		Combustion	Aspiration: CNS Depression
			Hazardous decomposition products: Carbon Oxides

A bow tie plot was done in Figure 9 with references in Table 18 to account for loss of Hexane containment in the design. Three major threats are taken into account in this process. Hexane vessel overflow, heat exchanger tube rupture and flooding of stripping column could all result in spillage of Hexane. Barriers implemented to control these issues include the use of LIC/FIC cascade controls and alarm systems, appropriate heuristic considerations for the stripping column and heat exchanger materials. Recovery measures intended to mitigate the risk include proper use of PPE of operators, safety training and emergency plan for shut down to protect operators and plant personnel. Major consequences posed by Hexane are risk of fire/explosion, property loss and injuries/fatalities regarding Hexane exposure.

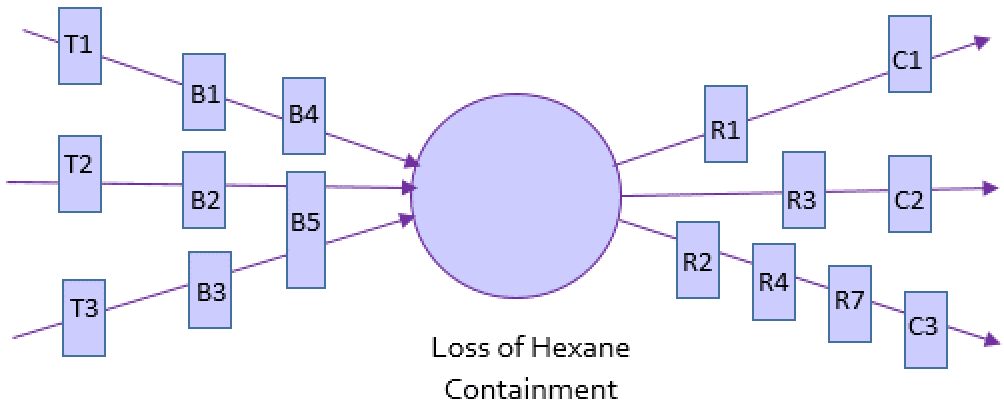

Figure 12: HAZID bowtie plot for Loss of Hexane containment.

Table 18: HAZID bowtie plot references of threats, barriers, recovery measures and consequences.

Threats	
T1	Hexane vessel overflow
T2	Heat exchanger tube rupture
T3	Flooding of stripping column
Barriers	
B1	LIC/FIC Control Systems
B2	Proper MOC for Hexane (Carbon Steel)
B3	Allowable flooding percentage set
B4	Heuristic equipment oversize
B5	Alarm systems
Recovery Measures	
R1	Immediate clean up/ inert material availability
R2	Shut down
R3	PPE use at all times
R4	Safety training
R5	Emergency Plan
Consequences	
C1	Fire and Explosion
C2	Major property loss
C3	Operator Injuries and Fatalities

Safety measures need to be considered in case a spillage occurs. Respirators should be readily available in the area to avoid chemical exposure. Inert materials (vermiculite, sand or earth) need to be used for clean up immediately after the spill occurs [14] . Immediate cleanup of spills should take place to ensure operators are not exposed to the compounds.

Environmental Issues

Environmental issues need to be considered seriously in any chemical process. Hexane is a readily biodegradable compound, and is not expected to be adsorbed in sediments. Due to its toxicity to aquatic organisms, this component should not be released directly to the environment [14] . Hexane is recycled throughout the system, which eliminates the environmental risk associated with this compound in an independent product stream. Hexane from a spillage that is recovered in any incident, or that comes into contact with operators and cannot be recycled safely in the system should be contained in a separate vessel. EPA regulations under the Clean Water Act allow for a concentration of 796 ppm. Any Hexane from a spillage that is released to water should be evaluated to have an equal or lower concentration than this standard [19].

Due to the use of water only for utilities, there is no waste water stream produced from the system. Condensate from the High Pressure Steam used in the T-101 reboiler is produced. Further options to recycle this condensate in the process as part of water utilities could be evaluated to reduce water waste and potential utility costs.

Eucalyptol, Camphor, Linalool and Linalyl acetate all readily volatilize from water sources. Linalool is a biodegradable compound in both terrestrial and aquatic conditions. Camphor and Linalyl acetate present slow biodegradation and the former one is expected to adsorb into suspended solids and sediments [20] [21]. Due to the slow rates of degradation of previous compounds and no available data for Eucalyptol, no oil components should be released in water as a cautionary measure. If oil components are released into water, further removal techniques as activated sludge should be implemented to minimize effects [22].

Conclusions and Recommendations

We recommend this process design for the production of lavandin essential oil. The given design produces 96 kg/hr of lavandin essential oil, while exceeding process specifications for the hexane recycle stream achieving 99.99% hexane recovery. Additionally, the design minimizes the residue of hexane in the final oil product. Hexane presence regulated by the US Food and Drug Administration sets a value of 25 ppm hexane in essential oils using hexane in solvent extraction. The proposed design achieves 1.07 ppm hexane in the lavandin oil product.

The purity of the oil achieved allows for this process to compete in business markets with lavandin essential oils produced with steam distillation, especially because hexane extraction provides greater oil yields for lavandin than steam distillation techniques. Additionally, the process design is flexible to market fluctuations as lavandin flowers can be replaced by jasmine, linden, and others to produce other types of essential oils [2]. The implementation of hexane extraction introduces safety and environmental measures related to hexane use, however our research finds that risks are manageable given that proper safety and environmental measures are employed.

From an economic perspective, this lavandin essential oil production design is extremely profitable. It has a 633% rate of return on investment, with a payback period of only 0.2 years. Equipment in the process is relatively small, which allows for lower capital costs and smaller costs for equipment replacement and maintenance.

There are a variety of recommendations to improve the current design. Firstly, we recommend investigating other components of the lavandin oil that were not considered in the process design. These components would be very small in comparison, but are worth investigating to produce a more complete model. Another recommendation to create a more accurate model would be to implement the production of absolutes from lavandin concretes that would be formed during the extraction process. Also, the solid flowers could be modeled in ASPEN and their maintenance and waste disposal could be added to the economics of the design.

Additionally, the effects of a more conservative oil yield could be further investigated. The reported yield of lavandin oil extraction was 4-22%, and we assumed the 22% yield as discussed in the Process Assumptions [1]. This has a great effect on the economics of the design, therefore we recommend performing laboratory tests on the oil yield, and run the economics with the discovered yield.
Another improvement that can be made confronts the lack of historic pricing for the lavandin essential oil. Further investigation of oil pricing data is recommended to establish a more accurate representation of the essential oil product economically. The market analysis can be completed by a consulting firm.

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