Modelling Steam Distillation For Essential Oil Production

by

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Author’s Declaration

I declare that this thesis is my own account of my research and contains as its main content work which has not previously been submitted for a degree at any tertiary education institution.

Mitchell Glenister

Abstract

The production of essential oil has remained unchanged for decades. In the face of increased competition from low cost suppliers the requirement to reduce the cost of production is facing many producers. The application of a counter-current system to the steam distillation of essential oils the transition from traditional production methods to more efficient methods was evaluated. Modelling a system using existing mathematical models found in the literature provided a basis for the comparison between the old and the new.

The question of the conversion of operating principles was met with the findings that the system is immature in inception. Further research is required to address the technical issues of introducing and removing plant material from a distillation column without interrupting the flow of steam. Initial bench tests have confirmed the benefits of moving to a non-batch system, this research is in its infancy with many further tests required to cement the findings of this thesis. Substantial portions of this thesis have been redacted or limited details provided, as large sections of the research are of a confidential nature.

Acknowledgements

Firstly, I would like to thank my academic supervisor Professor Parisa Bahri for taking on the role of supervising this thesis and the continuous support that you provided throughout the project. I am forever grateful for your wisdom and advice when dealing with the difficult situations that arise when dealing with external sources.

To the friends and family that I neglected for my studies. I thank you for your patience and understanding. I do intend to be present more often in the future.

A big mention to my fellow students. For all the laughs and conversations, we have had over the years. Hopefully the late nights pay off for us all.

And most importantly of all. My loving girlfriend, Shelby Wilson for putting up with all the nights that I didn’t come home until late, or not at all. I could not have completed this without you.

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List of Abbreviations and Notations

a	Specific Mass Transfer Area	m^{2} kg^{-1}
Gs	Mass flowrate of Steam	kg hr^{-1}
K	Overall Mass Transfer Coefficient	kg oil h^{-1} m^{-2}
Mi	Mass of Inert part of Material	kg
min	Rate of Material Input and Output	kg hr^{-1}
$moil$	Mass of Oil Contained in Material	kg
P^{0}	Partial Pressure of Oil	kPa
s	In Column Steam to Material Ration	kg steam kg^{-1} material
x	Material Oil Mass Fraction	kg oil kg^{-1} material
Xin	Material Input Concentration	kg oil kg^{-1} material
y	Steam Oil Mass Fraction	kg oil kg^{-1} steam
yin	Inlet Steam Oil Mass Fraction	kg oil kg^{-1} steam
α	Proportionality Parameter	kPa^{-1}
ε	Column Voidage	unit less
ρs	Steam Density	kg m^{-3}
Ω (m^{2})	Available Surface Area for Mass Transfer	m^{2}

Chapter 1 Introduction

The rationale for this project is as a response to the Australian Government Rural Industries and Research Corporation's (RIRC) publication "New and Emerging Plant Industries Three-Year RD&E Plan (2015-2018)". The RIRC aims to increase the competitiveness of Australian essential oil producers in the face of increased competition from low cost producers in developing nations, where the cost of labour is substantially lower than Australia's own. Improving production techniques to increase profitability and quality control have been identified within the plan.

To address the needs to improve the current production techniques for essential oil production, a review of the current production methods was required. During the research of essential oil production, the use of batch processes has proven to be in widespread use [1]. A possible solution to improving profitability was identified in the use of a counter-current system.

A comparison between the proposed counter-current system and the existing batch system was required. To do this the basic operating conditions of the two would be explored with a sensitivity analysis of process operating conditions and economic analysis. This is an area that yielded no information in the literature review of this project in relation to essential oil production by steam distillation. Research uncovered references to a small number of non-batch steam distillation systems [1] [2]. These systems are of great commercial value, and such have limited details available due to confidentiality reasons.

The lack of an apparatus to test the theory led to the formation of a computer simulation as a substitute. The validation of such simulations requires external data. Upon receipt of this data comparison can be made based on the production costs of the systems.

Chapter 2 Review of the Literature

2.1 Essential Oil

Essential oils are the fragrant oils extracted from plant materials that produce the characteristic fragrance or essence associated with the plant [3]. Uses for essential oils and other plant extracts range from perfumery and flavouring to their incorporation in pharmaceuticals, including traditional medicine [1]. There is documented use of essential oils in India dating back to 2000 BC, with descriptions of hundreds of aromatic substances and their uses for religious and medicinal purposes [3]. The produced oil, though as close as reasonably possible, is altered in composition to the underlying oil in the plant material. The separation process selected for the oil extraction determines the overall degree of variation.

2.2 Extraction Techniques

As stated in the previous section the method employed to separate the oil into a useable state is a major factor in the composition of the extracted oil. Two of the most widely used techniques are briefly explained in the following sections.

2.2.1 Solvent Extraction

The use of a solvent to extract oil within the solid plant material is a common practice for the processing of delicate materials that are susceptible to heat damage [4]. The oil within the material diffuses into the solvent until the concentration of the oil is in equilibrium between the material and the solvent.

The use of hydrocarbon solvents for essential oil processing is the norm in the studied literature, with hexane being the most frequently mentioned solvent referenced. To isolate the oil from the solvent, the solvent is evaporated [1]. The solvent extracts the oil soluble compounds within the plant material. These include the waxes and resins which produce a semi-solid material known as a concrete.

Separating the oil from the semi-solid concrete involves the dissolution of the concrete into another solvent. Distillation of the new solution is performed with the more volatile compounds evaporating and leaving the residual waxes and resins. The use of the solvents introduces the risk of small traces of the solvent carrying over into the final product [5].

With a trend of environmental and “green” marketing the presence of petroleum based solvents, such as hexane, in the produced goods reduces the appeal to customers [5]. For this reason, solvent extraction was not explored in this report.

2.2.2 Steam Distillation

Steam distillation is the extraction technique that produces majority of essential oil production [1]. Steam is introduced into the bottom of the chamber containing the plant material and moves within the voids of the packed material. The use of steam allows for the extraction of the oils at temperatures lower than their boiling point [1].

The boiling point of some components of essential oils can be significantly higher than the temperature at which they are susceptible to thermal degradation [6]. The use of steam allows for the distillation to be performed at lower temperatures, generally below 100°C. The vapour pressure of the oil components is what allows the oils to be extracted at lower temperatures.

Each component exhibits its own vapour pressure at a given temperature, it is the sum of these partial vapour pressures that make up the system pressure [1]. A mixture will boil when the sum of these vapour pressures is equal to the atmospheric temperature. The implication of this is that the mixture will boil at a temperature lower than the temperature of the pure components. It is for this reason the high boiling temperature components can be extracted at lower temperatures.

A typical steam distillation setup is presented in Figure 2-1. The still section is filled with the solid plant material, which is packed using differing methods based on the feed stock, with steam injected into the bottom section from an external boiler. The steam-oil mixture exiting the still is fed to a condenser. The role of the condenser is to reduce the temperature of the steam-oil mixture until the steam returns to a liquid state.

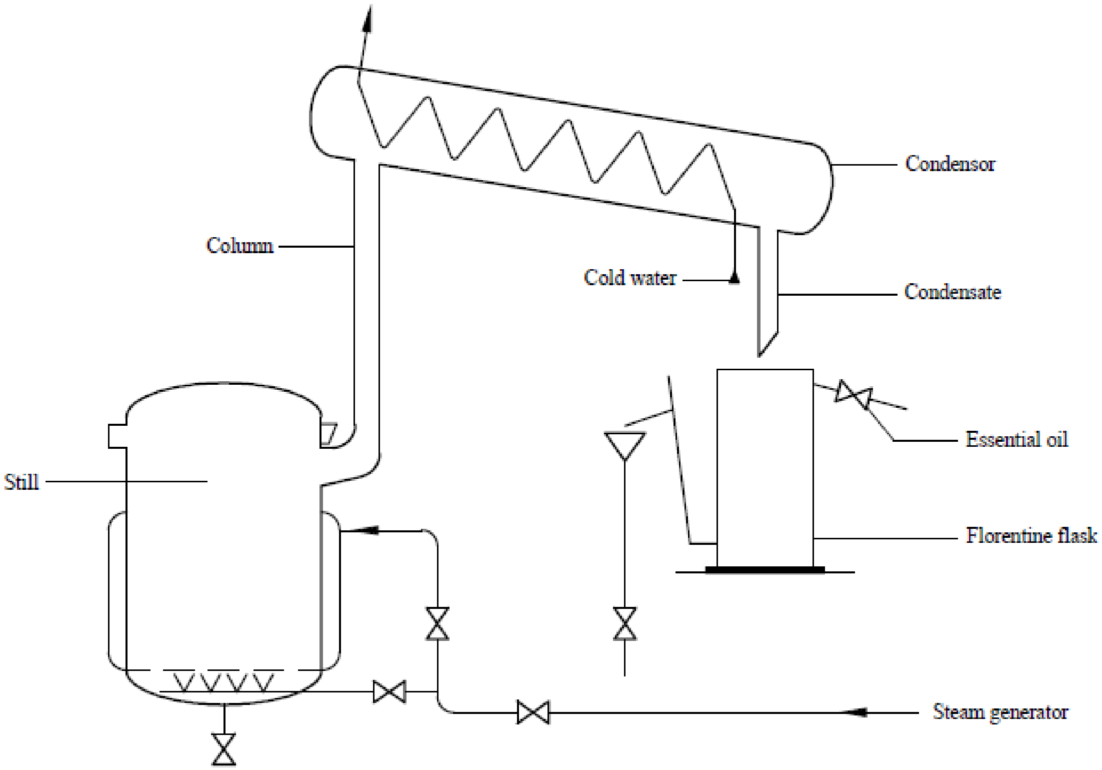


Figure 2-1 Steam Distillation Apparatus for Essential Oil Extraction [3]

Upon exiting the condenser in steam distillation, the oil and condensed steam form two immiscible layers. A separation of the layers is required which is traditionally performed in a Florentine flask. The Florentine flask allows the two layers to be drawn off separately.

2.3 Modelling of Steam Distillation

Several models have been proposed in the researched literature for steam distillation of essential oils. Each of these models is shown to fit the experimental data to very high degrees. The issue with these approaches is in the application to only the plant material that they are investigating and under strict laboratory conditions. Thus, models developed for specific processes cannot be applied to other processes accurately.

The sterility of the testing, and the various models each closely fitting the experimental data was an area of concern. Selection of a model from the list of possible models was therefore complicated. A decision was made to narrow the possible models to two candidates. The first involved a kinetic model and was presented in the case of the steam distillation of aniseed [7]. The second available model was based on the diffusion of oil into steam with members of the Baccharis genus of plants [8].

Selection of the final model to be used in this thesis was based upon the availability of data. A company was approached for data regarding their oil production; they provided production data and model parameters which resulted in the kinetic model for aniseed oil production to be selected.

2.4 Non-Batch Processing of Essential Oils

Available information for essential oil production that does not rely upon batch processing is severely limited. Several different operators are mentioned as having implemented counter-current systems, these include Texarome in the USA for cedar wood oil production and BIOLANDES in France for dwarf pine oil production [1] [2]. Specific details of the developed processes are unavailable, as the essential oil market is very competitive, and so innovation and processing techniques are not widely shared or reported.

The lack of available details limits the ability to provide cost estimates on the implementation of new counter-current systems. This is due to uncertainty in the method of introducing and removing material to the system with no interruption of steam supply.

Chapter 3 Model Development

This section of the text demonstrates the formation of a working Simulink model to simulate the extraction of essential oils from the non-essential oil portion. Several assumptions have been outlined throughout the process of deriving and implementing the model into Simulink, most of which have eased the implementation greatly. These simplifications have allowed a complex problem to be broken down into manageable parts and assembled into a working simulator that allows for sizing configurations to be tested and analysed. The analyses of altering the parameters of the process is presented within this section, with results contained in the sensitivity analysis sections of this report.

3.1 Batch Steam Distillation Model

Development of a model for the extraction of essential oils in a batch process has been studied by numerous research teams in the past. Each has developed a model that is believed to accurately represent the mode of extraction taking place. The choice of model for this project was based on the availability of data provided by an external source, which for confidentiality reasons will not be presented within this paper. The model was based upon work to develop a kinetic model for the extraction of anethole from Tunisian aniseed [7]. As previously stated the data provided was considered confidential resulting in the model presented to be of a hypothetical essential oil. The benefits of this approach lend themselves to other essential oils, and it is intended that the application of this model development can be easily adapted to various essential oils in the future with minimal model corrections.

The model presented considered the oil to be made up of single pseudo-constituent with the largest component being the modelled oil, which in the case of aniseed is anethole, constitutes 80-94% of the total essential oil of aniseed [7]. The use of a simplified model has its benefits in the model development, a single component leads to a single case of mass transfer, whereas multiple components would lead to several different mass transfer calculations being required. For the purpose of model simplification, and based on the available academic literature,the following list of assumptions were made for the steam extraction model [7];

I. The essential oil is considered as a single component.
II. Constant temperature and pressure along the length of the column.
III. The feedstock was considered to behave like a porous material.
IV. The steam flow has no effect on the movement of the particles of the essential oil-bearing material.
V. The size and shape of the particles is unaffected by the extraction process, they remain spherical and do not change in size throughout the process.
VI. Condensation of steam does not occur in the column.
VII. Diffusion of steam within the pores of the material is negligible.
VIII. Initially, the composition in oil is homogenous.

A linear driving force is the main method for determining the rate of extraction of the oil from the oil bearing material. The use of which plays a major role later in the work of determining the optimal rate of steam flow to the column. To begin the modelling an overall mass balance of the oil within the column was taken to be equal to the change in oil composition of the steam at the boundaries of the column as presented in Equation 3-1.

$$G_s y_{in} - G_s y(t) = \frac{dm_{oil}}{dt}$$

Equation 3-1 Column Overall Oil Mass Balance [7]

Where m_{oil} is the mass of oil contained within the column, y_{in} (kg oil kg^{-1} steam) is the essential oil content of the steam entering the column, y(t) is the essential oil fraction by mass of the outlet steam (kg oil kg^{-1} steam) and G_S (kg hr^{-1}) represents the rate of steam addition to the column. For the batch model the steam entering the column is fresh steam from an external boiler which contained no oil and so was dropped from subsequent equations. Upon dropping the variable y_{in} and a slight rearrangement Equation 3-1 becomes Equation 3-2. Equation 3-2 relates the outlet steam concentration to the rate of oil extraction and the steam flowrate.

$$\boldsymbol{y(t) = -\frac{1}{G_S} * \frac{dm_{oil}}{dt}}$$

Equation 3-2 Fresh Steam Overall Oil Mass Balance [7]

The mass of oil contained within the material is equal to the product of the concentration of the oil, x, (kg oil kg^{-1} material) and the total mass of plant material in the column (kg). Combining the mass of oil within the material and the mass of oil entrained with the steam, the total mass of oil in the column at any given time, t, can be found. The total mass balance of oil in the column is shown in Equation 3-3, with ε, ρ_s and V_c representing the column voidage, density of steam (kg m^{-3}) and the volume of the column (m^3) respectively.

$$m_{oil} = m_{material} x(t) + \varepsilon \rho_s V_c y(t)$$

Equation 3-3 Mass of Oil in Column [7]

The material can be split into two distinct components, the oil portion and an inert portion. The mass of the inert portion is unchanged over the length of the distillation process and is related to the mass of the material by Equation 3-4.

$$M_i = m_{material} \, (1 - x(t))$$

Equation 3-4 Mass of Inert [7]

Substituting $m_{material}$ with M_i and differentiating Equation 3-3 provides the change in the mass of oil in the column, represented by Equation 3-5.

$$dm_{oil} = M_i \frac{dx}{(1 - x)^2} + \varepsilon \rho_s V_c dy$$

Equation 3-5 Mass Balance of Column Oil Content [7]

Considering no net change in the total mass of oil, the mass of oil taken up by the steam is equal to the loss in mass of the material. The final step of the mass balance equations is to substitute the change in the mass of oil in the column with the rate of oil exiting the column with the steam. Combining Equation 3-1 and Equation 3-5 yields the mass balance of oil in the column in terms of the flow of steam through the column and the steam content sent to the condenser of the system.

$$-G_S y dt = M_i \frac{dx}{(1-x)^2} + \varepsilon \rho_s V_c dy$$

Equation 3-6 Mass Balance in Terms of Steam Flow [7]

The presented mass balance equations have so far provided a relationship between the flow of steam in the system and the change in the total oil content of the system. The rate that oil is transferred from the material to the steam is a requirement of the model. The aniseed reference model was presented using an overall mass transfer coefficient, K (kg oil h^{-1} m^{-2}), which relates the rate of oil extraction to the material oil concentration and is shown in Equation 3-7.

$$-\frac{dm_{oil}}{dt} = K\Omega(x - x^*)$$

Equation 3-7 Rate of Extraction [7]

The rate of extraction is dependent on the surface area available for mass transfer, Ω (m^2) and the difference between material oil concentration and a concentration considered to be in equilibrium with steam, x^*. The value for x^* was found to be difficult to directly determine, and was subsequently replaced with a proportionality constant, α (kPa^{-1}) ¸ multiplied by the oil vapour pressure, P^0 (kPa), for a given temperature and pressure [7]. The overall mass transfer coefficient, and proportionality constant were found by combining Equation 3-5 and Equation 3-7 to form and fitting to experimental data [7].

$$\frac{M_i}{(1-x)^2}\frac{dx}{dt} + \varepsilon\rho_s V_c\frac{dy}{dt} = -K\Omega(\mathrm{x} - \alpha P^0)$$

Equation 3-8 Overall Mass Transfer Equation [7]

Dividing both sides of Equation 3-8 by the mass of material in the column converts the available surface area Ω, and the mass of steam in the column to be in terms of the mass of material. These new variables become; the specific mass transfer area a ($m^2\ kg^{-1}$), and the mass of steam in the column becomes a new variable s (kg steam kg^{-1} material). The purpose of this step is to reduce the complexity of the equation by making the coefficient of the dx/dt term approximated to 1 [9]. The final equation, presented in Equation 3-9, with the fitting of experimental data to determine the values for K and α, which was provided by the company that supplied experimental data for this report.

$$\frac{dx}{dt} + s\frac{dy}{dt} = -K\mathrm{a}(\mathrm{x} - \alpha P^0)$$

Equation 3-9 Final Mass Transfer Equation [7]

3.2 Counter-Current Steam Distillation Model

The determination of the counter-current model relied on the equations presented in Chapter 3.1, with the column being separated into several sections of equal volume with the flow of material in the column considered to be in a perfect plug flow. The sections were arrayed in a formation in which the outputs of each section become the inputs of the proceeding section as demonstrated in Figure 3-1. The material depleted of oil is removed at or near the point of steam entry to the column, this ensures the material on exit has minimal residual oil. The mass of the inert portion of the input and output streams of material are equal, ensuring no net gain of inert in the plug flow reactor model.

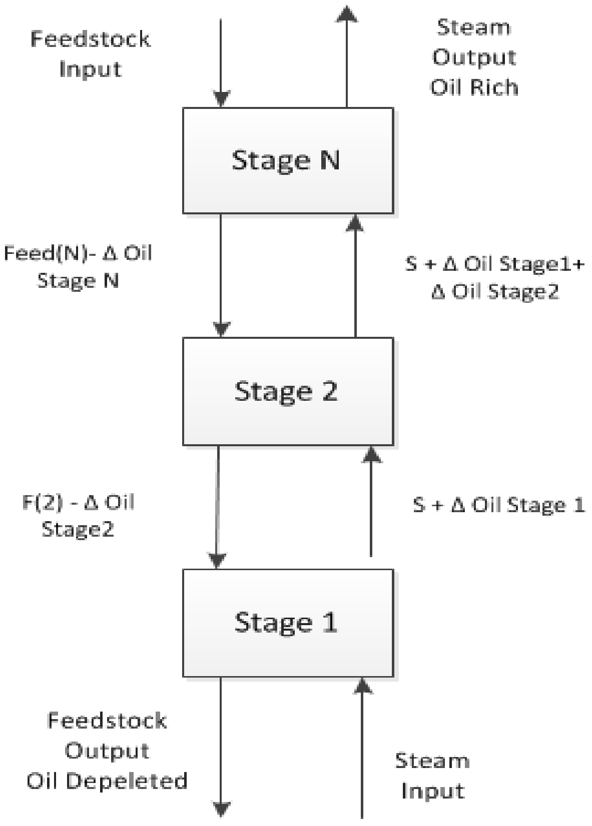


Figure 3-1 Flow Chart of Model Operations [10]

Employing Equation 3-1 with the $G_S * y_{in}$ term no longer removed for all sections, barring the steam entry point, the change in each sections oil content is equal to the change in the steam oil concentration at the boundaries of the section. The concentration of oil in the steam can be found at the boundaries of the section through Equation 3-10 [7].

$$y(t) = -\frac{1}{G_S} * \frac{dm_{oil}}{dt} + y_{in}$$

Equation 3-10 Steam Oil Concentration for Sections

The mass of oil extracted from the column is calculated using the change in the steam oil concentration between the inlet port of fresh steam and the outlet of the final stage, which is at the entry point of fresh material to the column.

To account for the constant input and output of material to the system, two new variables are added to the batch model in Equation 3-9. The new variables are to complete the mass balance of the system at steady state. The new variables m_{in} (kg hr^{-1}) and x_{in} (kg oil kg^{-1} material) are the mass of material and concentration of input material respectively [11]. The overall mass balance at steady state for the counter-current system is presented in Equation 3-11.

$$\frac{dx}{dt} + s\frac{dy}{dt} = -K\mathrm{a}(\mathrm{x} - \alpha P^0) + m_{in}\, x_{in}$$

Equation 3-11 Counter-current Overall Mass Balance [11]

3.3 Economic Modelling

Prices of essential oils are extremely variable, and can be very volatile. A recent example of the volatility of the market is the average price for peppermint oil, in the period between 1994 and 2006 the average price steadily declined from US$42/kg to a low of US$12/kg. The steady downward trend abruptly reversed at the beginning of 2006 and in the two-year period ending 2008 the price had risen approximately 600% to US$72/kg [1]. The variability of the oils is evident in market price differences between differing oils. Orange oil which is a by-product of the fruit juice industry is produced in copious quantities and is readily available, the price is evidently very low for essential oils at approximately US$1.80/kg [12].

Production of essential oils is a labour-intensive operation. This has seen the movement of essential oil production from high-cost areas such as France to low cost areas, predominantly China and India [3]. To produce oils in the higher cost areas, the extra cost of production must be accounted for by either an increase in efficiency or a higher output. The inclusion of estimated production outputs for the batch and counter-current models has been used to evaluate the viability of moving away from a batch processing system.

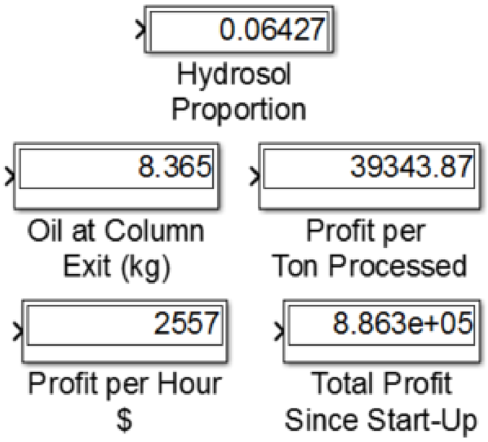
Incorporated into the model is a value for the fixed hourly overhead cost to run the facility. Overheads have been included to provide a comparison between operating in a high cost area to a low-cost area and the effect on the operating conditions of the plant. Steam production is the other major cost included in the model, with the assumption that a production facility will be retrofitting existing facilities to accommodate a counter-current capability. The assumption of retrofitting an existing facility is due to envisioned technical difficulties associated with developing a true counter-current system.

Revenue from the sales of produced oil was the only income stream available from all essential oils investigated. Some essential oils have secondary products which provide a dual income stream potentially increasing the viability of the project [3]. However, as the oil considered for this study is considered a hypothetical oil, the assumption of the oil being the only product of value produced allows the oil to encapsulate a greater number of actual oils. The breakdown of operating costs and revenue streams is provided in Table 3-1 based on hourly production figures generated.

Table 3-1 Revenue and Cost Breakdown Per Hour

Revenue Generated by Oil Sales	Price per kg * Rate of production
Cost of Steam	Gs * Unit Price Steam ($/Ton)
Fixed Overhead Cost	Cost per hour

The variables presented in Table 3-1 were incorporated into the Simulink model to allow for an indication of the effect of running conditions on the profitability of a plant. The outputs from Simulink are shown in Figure 3-2, an extra value is presented which is the production of hydrosol. Hydrosol is the water-soluble fractions of the essential oil which form a solution with the condensed steam [1]. The formation of hydrosol was presented as a major contributor to the loss of oil in steam distillations of essential oils. For the distillation of aniseed previously presented, the hydrosol comprised 25% of the extracted oil [7].



0.06427
Hydrosol Proportion

8.365
Oil at Column Exit (kg)

39343.87
Profit per Ton Processed

2557
Profit per Hour $

8.863e+05
Total Profit Since Start-Up

Figure 3-2 Simulink Output of Current Profit Output from Model

The objective of the economic analysis is to determine the operating point that will provide the highest profit per unit processed. Figure 3-3 indicates the relationship between the pursuit of high yield and the required hours to achieve the target yield.

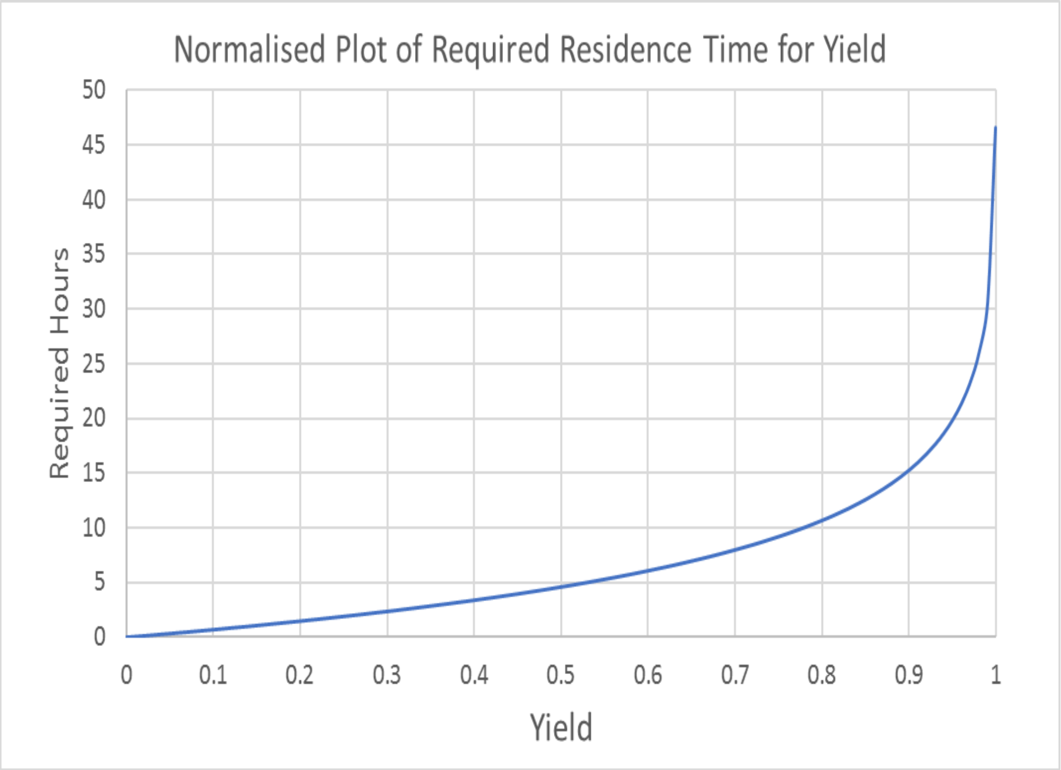


Figure 3-3 Asymptotic Curve of Yield

It is the avoidance of achieving a yield from the fed material results in increased generation of oil, but at rates that do not cover the cost to produce the extra oil. It should however be reiterated that the oil in this paper, is a hypothetical oil containing only a single component. To extract a complete essential oil, the distillation must be under the specific conditions that will generate the complete oil.

Modelling the extraction of essential oils in the past has focused on the mode of extraction of oil from the bearer material. Moving away from this approach to follow the economics of the extraction is a key feature in determining the optimal running conditions of the process. The ability to model the point at which the operation of a batch system is a powerful tool, for every hour that the system is operating there is a cost associated with the production of steam, the payment of wages and other factors. The inverse of this is also true, for every hour that the system is not producing oil there is a loss of opportunity and overheads continue, this is a fundamental weakness of a batch process.

Chapter 4 Simulation Results

4.1 Model Initial Testing

Initial model validation started with testing the model with the parameters initially provided by the external company against the provided data. The model outputs were presented to a representative of the company in which it was noted that the oil content in the steam was out of range. A typical value for the steam oil concentration was provided, and was incorporated into the model as a limiting factor. The concentration of the steam at differing heights within the column was recorded to ensure that the modelling of the limit of steam concentration was operating as intended. A generic model output is included in Figure 4-1.

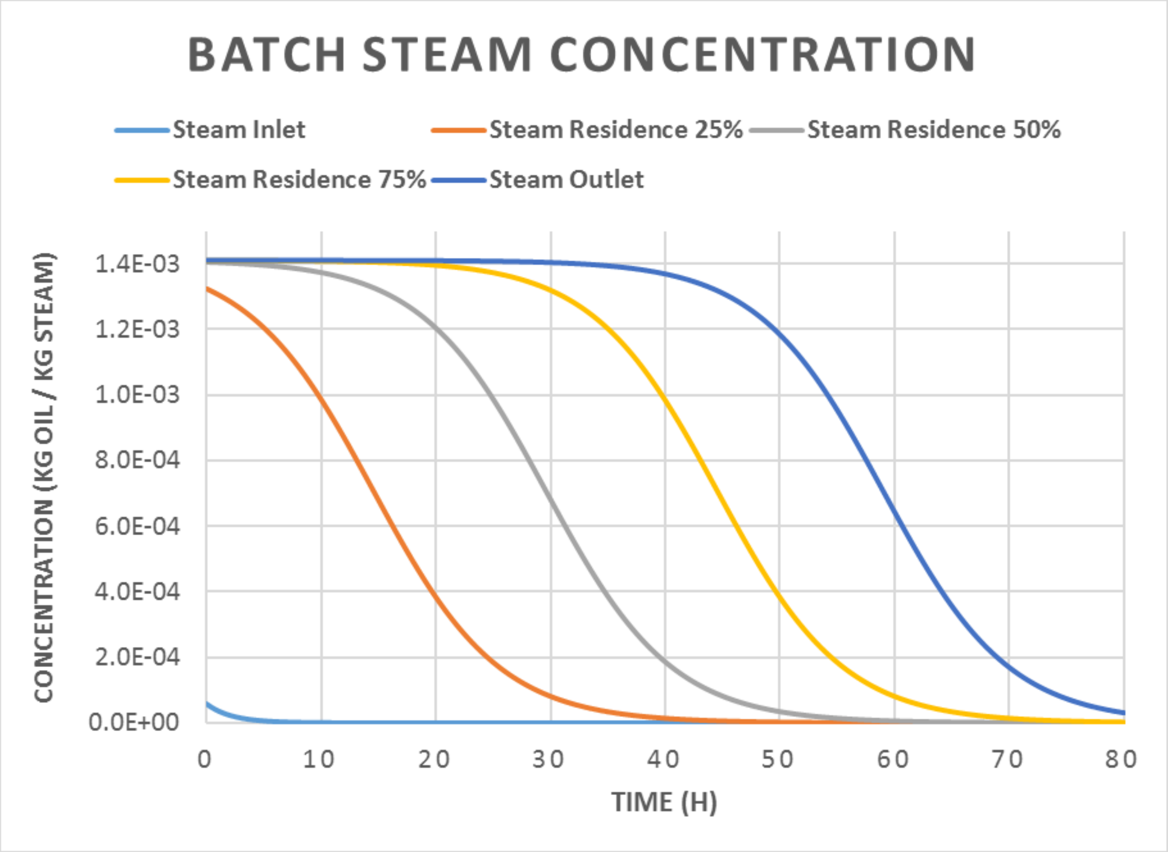


Figure 4-1 Generic Model Steam Concentration Output of Batch System

The profile shown in Figure 4-1 shows the point of saturation of steam moving towards the top of the column, or towards the steam outlet over time. The outlet steam concentration is shown to start reducing at the 40-hour mark in Figure 4-1, at this point it would be prudent to start evaluating the viability of continuing the extraction. The simulated concentration of the oil in material for the same simulation is presented in Figure 4-2. The concentration of the material at the top of the column is shown to be depleting in the hours before the steam outlet concentration begins to decline.

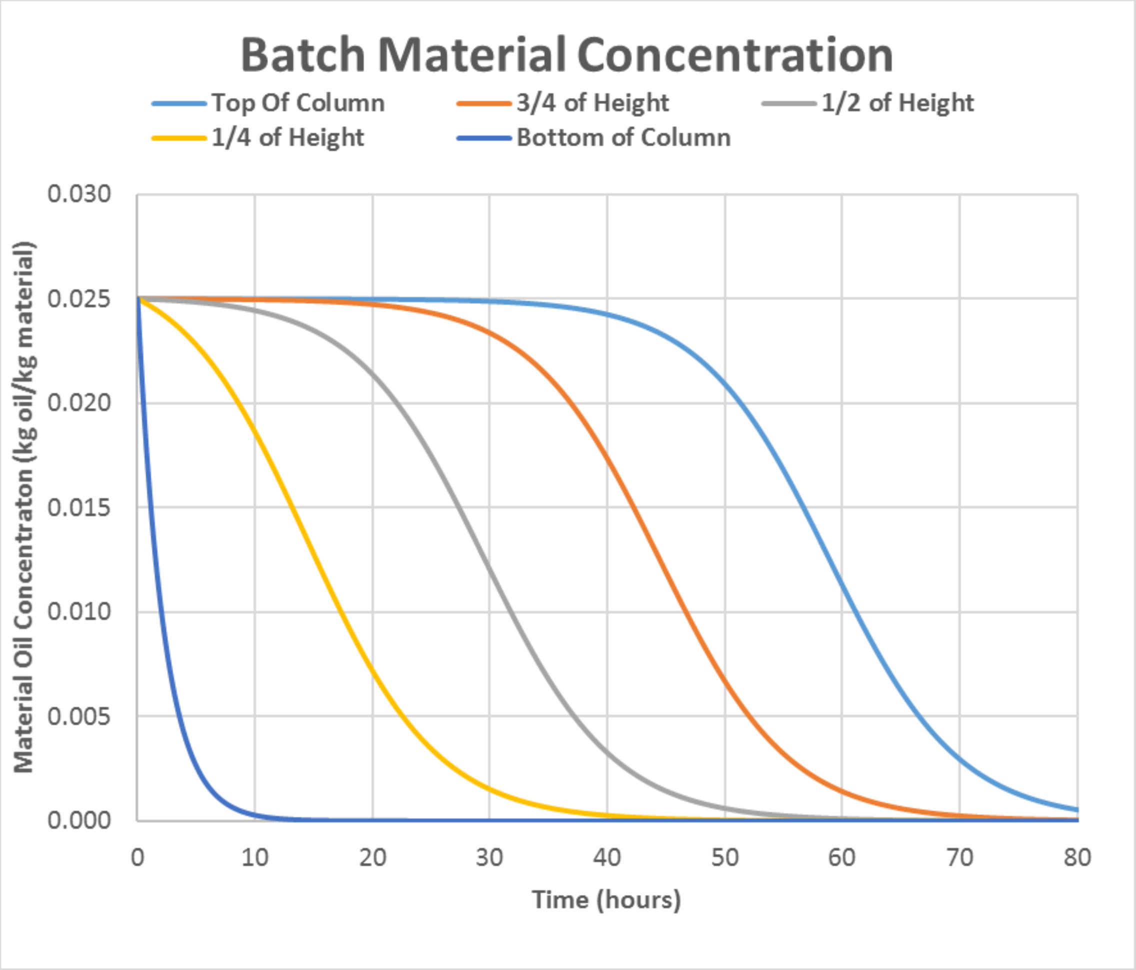


Figure 4-2 Generic Model Steam Concentration Output of Batch System

4.2 Sensitivity Analysis

A sensitivity analysis is a measure of the effect of uncertainty in the inputs of a model on the outputs of the model. The use of sensitivity analyses allows for greater understanding of the effect of parameter changes within the model and the magnitude of changes in the inputs and the outcome of the change throughout the whole system. In this model, the main objective of sensitivity analysis was performed to determine the viability of using the steam outlet concentration after the condenser to accurately represent the oil content of the system.

Another aspect of the sensitivity analysis was to find the oil concentration in the material that became the point at which operation transitioned from generating revenue to cover the overhead costs and steam usage of the plant. At this point it would no longer be viable to extract additional oil from the material as the revenue generated would be less than the cost of running the facility.

4.2.1 Steam Rate Comparison

The rate of steam addition to the column plays an integral role in determining the rate of extraction. A comparison between two steam flow rates in the batch system, one at 200kg/hr and the other at 250 kg/hr, are shown in Figure 4-3 and Figure 4-4 respectively. For the batch process the rate of steam addition plays a key role in determining the length of the total distillation time. With an increase in steam rate, the upper layers of material are subjected to unsaturated steam at an earlier stage in the process allowing for transfer of oil in the material to the flowing steam.

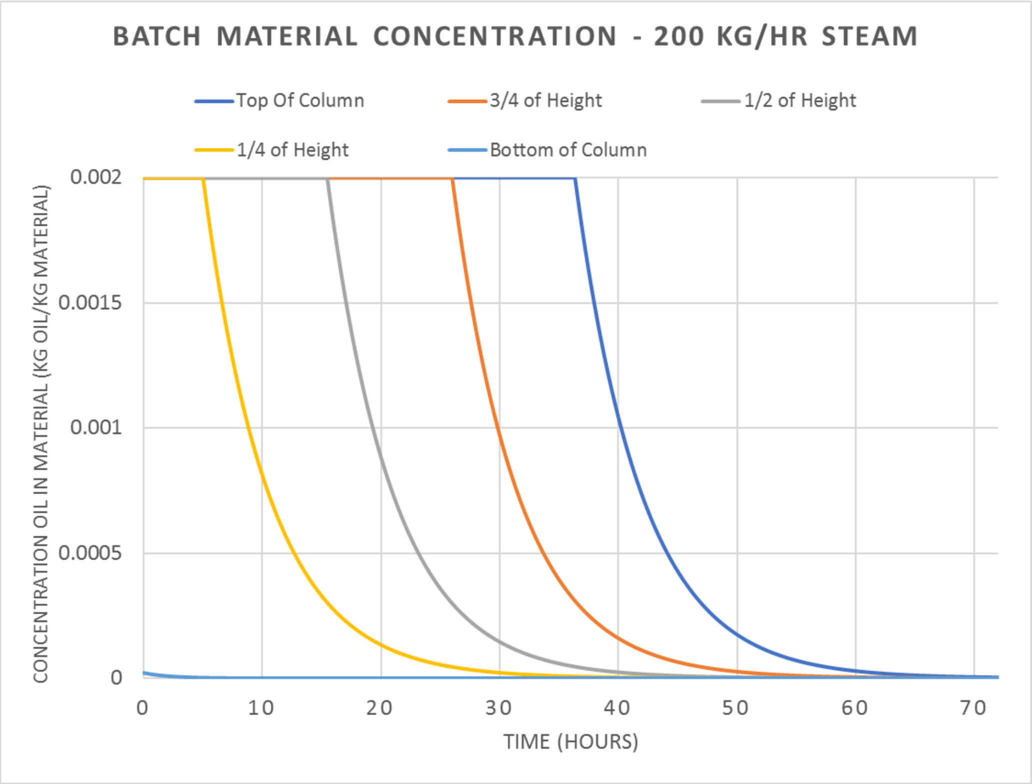


Figure 4-3 Material Concentration with 200 kg/hr Steam Flow

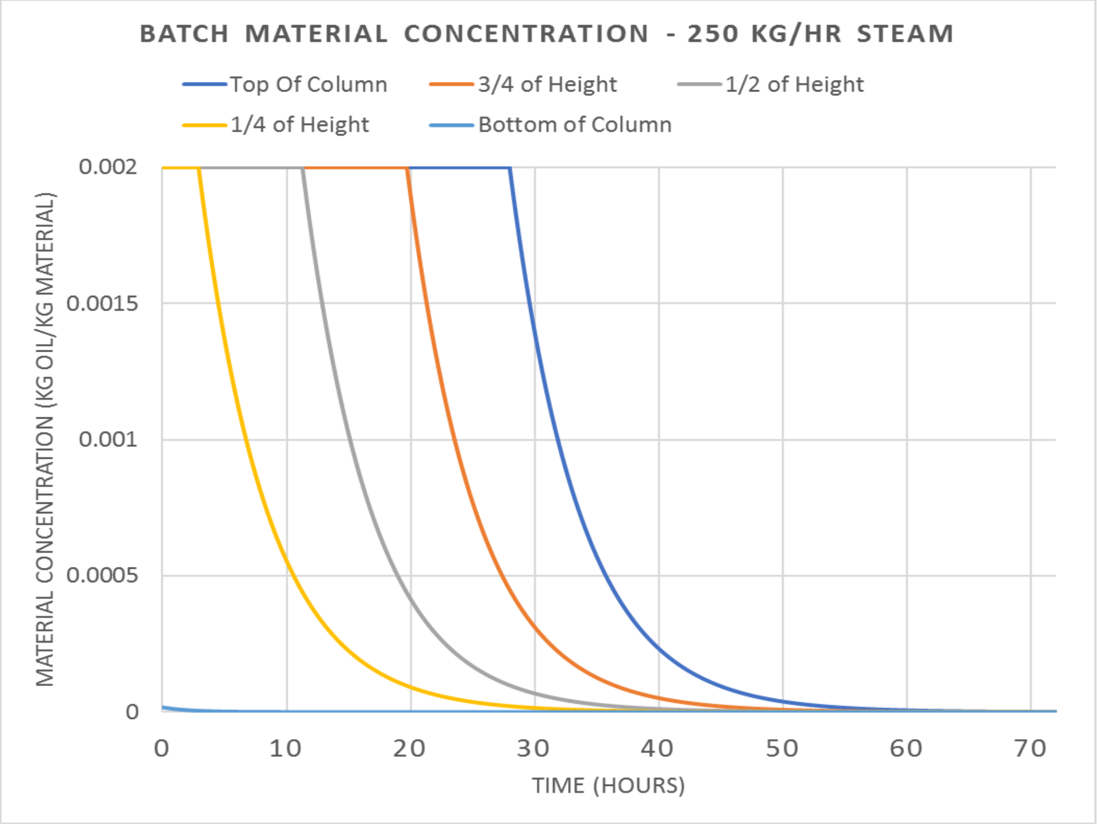


Figure 4-4 Material Concentration with 250 kg/hr Steam Flow

An increase in the steam rate from 200 kg/hr to 250 kg/hr at the 10-hour mark of the batch distillation is shown in Figure 4-5. The plot shows that the outlet steam concentration remains unchanged in the hours following the change in input steam rate. This was originally considered to be a possible source of monitoring the process, with the model disproving that measuring the steam concentration would provide an insight into the conditions within the vessel from an early stage. The ramifications of which were further realised during the trials of the counter-current model. Figure 4-5 also indicates that the concentration of the steam at the 25% of residence time drops rapidly. This may happen in a real system to a degree but would be unmeasurable, in a similar manner to sensing the concentration on every stage in a trayed distillation column [13].

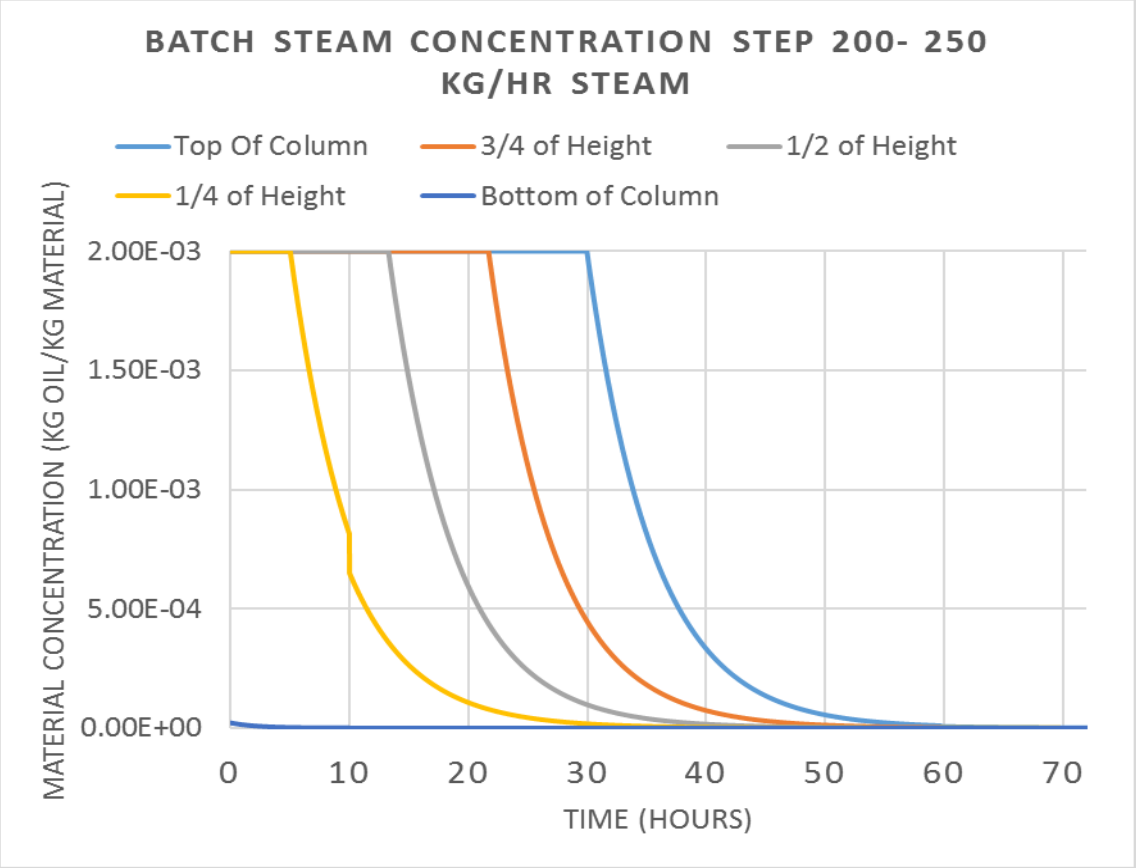


Figure 4-5 Steam Concentration Response to Increase in Steam Rate from 200-250 kg/hr

Initially a Coriolis mass flow meter to measure the concentration of oil in the steam upon exiting the condenser was hypothesised to be a suitable instrument for determination of the steam oil content. Taking the density at the outlet of the condenser rather than as a vapour was projected to be the best location for a steam density measurement, as the higher density of liquid water, in relation to steam, reduces the uncertainty in the reading.

The rationale for Coriolis flow meters was in their ability to measure density of the fluid, providing the mass flow of the process fluid [14]. The dismissal of Coriolis as an option is due to the low flow rates envisioned for the process and the small change in density that the oil contributes. The maximum accuracy that could be readily found was of an Endress and Hauser manufactured device, which had an accuracy down to 0.2g L^{-1}, which gives a 10% uncertainty in the concentration of steam if equilibrium is at 2.0g L^{-1} [15]

4.2.2 Material Input Concentration

The rate of extraction is dictated by two factors in the model, the availability of steam not yet saturated with oil traversing past the oil laden material and the concentration of the oil in the material. As presented in Equation 3-7, the greater the difference between the current concentration and the equilibrium concentration, x^* in Equation 3-7, the greater the rate of mass transfer. Figure 4-6 shows the effect of differing oil concentrations over a 50-hour period, at the end of the period the concentration of oil in material for each trial is approaching the equilibrium point with steam.

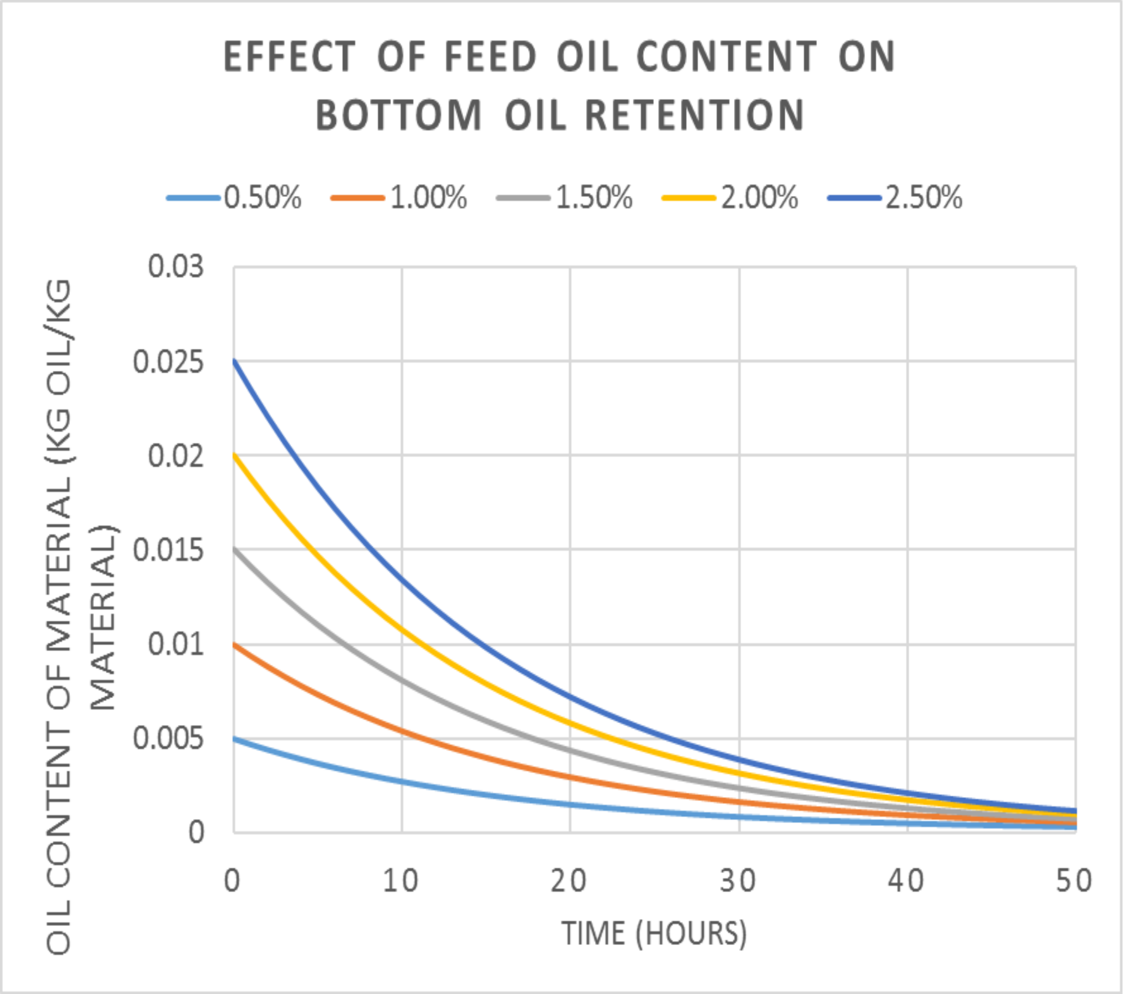


Figure 4-6 Comparison of 5 Different Initial Oil Concentrations Over Time

To further illustrate the effect of feed concentration on the oil content over time, Figure 4-7 is included to illustrate the mass of oil extracted for a 2-ton batch of material with no limit to the amount of steam supplied to the column. In a non-theoretical situation, the flow rate of steam to the column required to achieve this outcome would result in an oil of inferior quality due to degradation of heat sensitive compounds or charring depending on the severity [1].

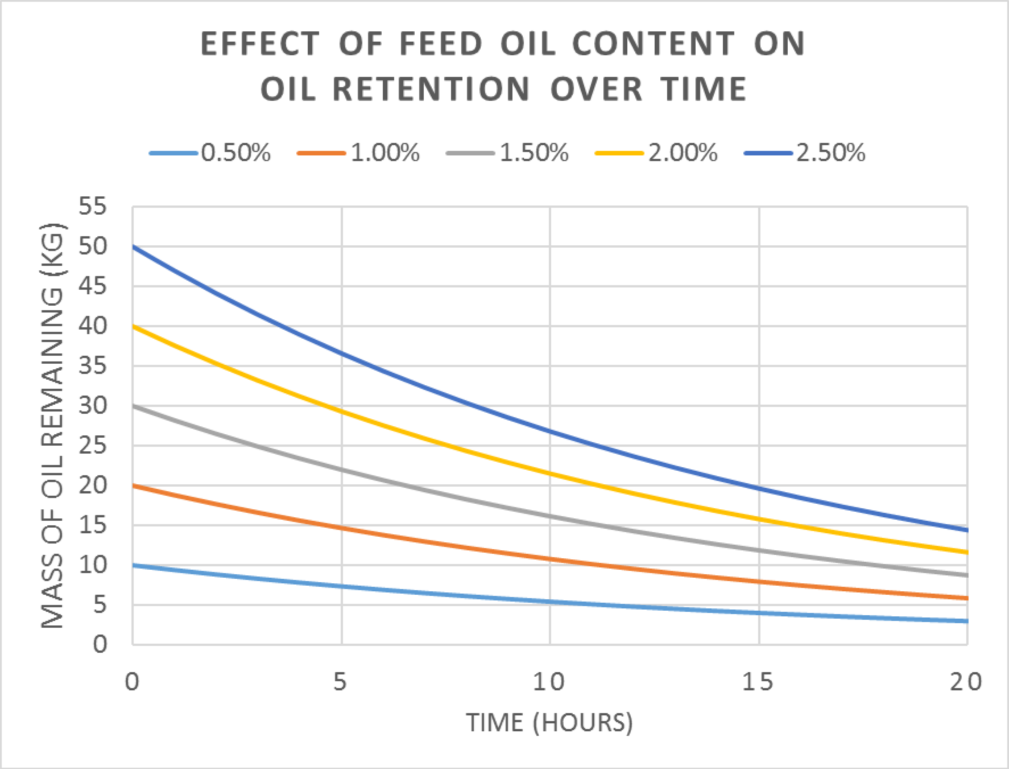


Figure 4-7 Mass of Oil Remaining Over 20 Hour Period of 5 Starting Concentrations

The oil concentration for the batch system is typically known before the process begins for operations on a factory scale [1]. With the starting concentration known, the number of hours to process the batch can be calculated assuming ideal conditions. For ideal conditions, sufficient steam is required to ensure saturation occurs at the exit of the column. The calculation is provided in Equation 4-1 [16].

$$x(t) = x_0 e^{-Kat}$$

Equation 4-1 Characteristic equation of A Batch Reactor [16]

4.2.3 Partial Pressure of Essential Oil

Partial pressure plays a dual role in the model: firstly, it represents the maximum value that the steam oil concentration can take on, and secondly it is used to determine the equilibrium point between the steam and the material. The vapour pressure of the oil is temperature dependent, and with the assumption that the temperature and pressure of the system is fixed for the length of the column at all steam flow rates, the effect of differing vapour pressures and the resultant effect on steam requirements and time for extraction can therefore be analysed. The mass transfer rate for anethole for aniseed was found to increase sevenfold with an increase in system pressure from 140kpa to 200kpa [7].

A sevenfold increase in the overall mass transfer coefficient significantly increases the attractiveness of increasing the system pressure. Increasing the system pressure for a steam system results in higher system temperatures [17]. The ideology of increasing temperatures is counter to the selection of steam distillation as the method of extraction, with most essential oils being susceptible to degradation from heightened temperatures [6]

Manipulation of the saturation limit of oil in steam for the model, with all other variables unchanged is shown in Figure 4-8. The time between starting the process and the outlet steam reducing from the upper concentration limit for increased solubility of oil exhibits a significant decrease in elapsed time. A rise in the maximum vapour pressure of the oil at the operating conditions is the manipulated variable for the sensitivity analysis of oil saturation limits.

The steam requirements to achieve a target level of extraction are heavily dependent on the vapour pressure of the oil. Figure 4-8 exhibits this; the different saturation limits were subjected to the same flow rate of steam, 200 kg hr^{-1}, as discussed earlier the time at which steam is no longer saturated on exit. The steam requirements are shown to be inversely proportional to the vapour pressure of the oil. Steam requirements are inversely related to vapour pressure.

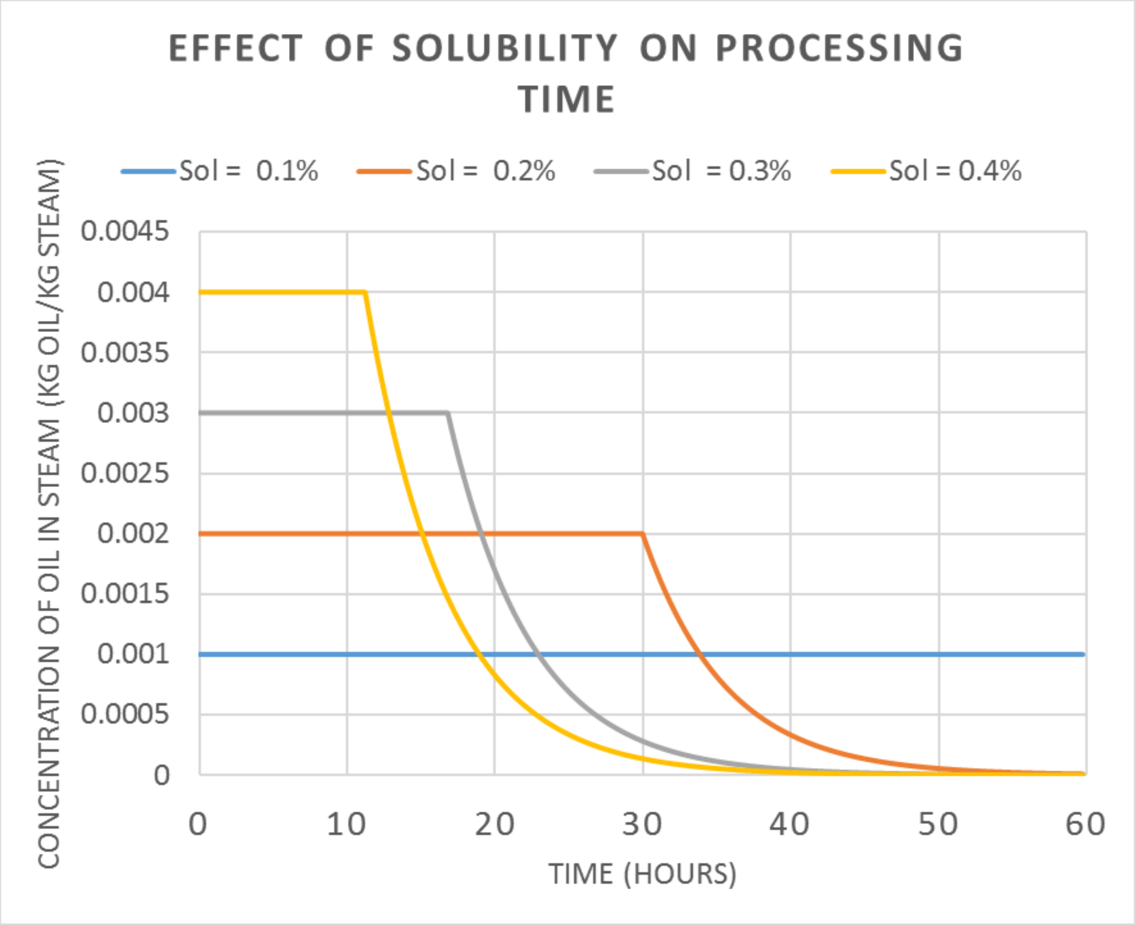


Figure 4-8 Effect on Processing Time of Differing Oil Solubilities in Steam

4.2.4 Feed Rate of Material

The application of material feed rate to the batch model does not exist, due to the process being characterised by the lack of a continual flow of fresh material to the system. Feed rate plays a major role in the operation of the counter-current model, because if the system is unbalanced between the mass of oil added and removed there will be a net change in the column oil content. The rate of material feed governs the residence time of the counter-current model, for a given flowrate the residence time, τ (hr), can be found through Equation 4-2.

$$\tau = \frac{V_c \rho_{material}}{m_{in}}$$

Equation 4-2 Residence Time for Counter-current System

Determination of the residence time of the system can allow for the determination of the outlet material concentration at steady state, provided the steam rate can remove oil throughout the column. For a system at steady state, the mass of oil entering the system with fresh material is equal to the rate of extraction, the left side of Equation 3-11 is equal to zero at steady state, the rate of addition of oil is therefore equal to the rate of extraction. This relationship is presented in Equation 4-3.

$$\frac{dm_{oil-in}}{dt} = Kax(t) - \alpha \mathrm{Ka} P^0$$

Equation 4-3 Counter-current Oil Balance [11]

The effect of differing the feed rate of material to the system creates concentration gradients within the column. Figure 4-9 indicates the various concentration profiles generated by the model for the same steam and concentration feeds. The feed rates of 20kg/hr of fresh material reaches the equilibrium point at the highest section in the column, with the concentrations increasing with increasing feed rates. The characteristic equation for a plug flow reactor is presented in with x_0 representing the material inlet concentration (kg oil kg^{-1} material) [16].

$x(t) = x_0 e^{-Kat}$

$$x(t) = x_0 e^{-Kat}$$

Equation 4-4 Characteristic equation of A Plug Flow Reactor [16]

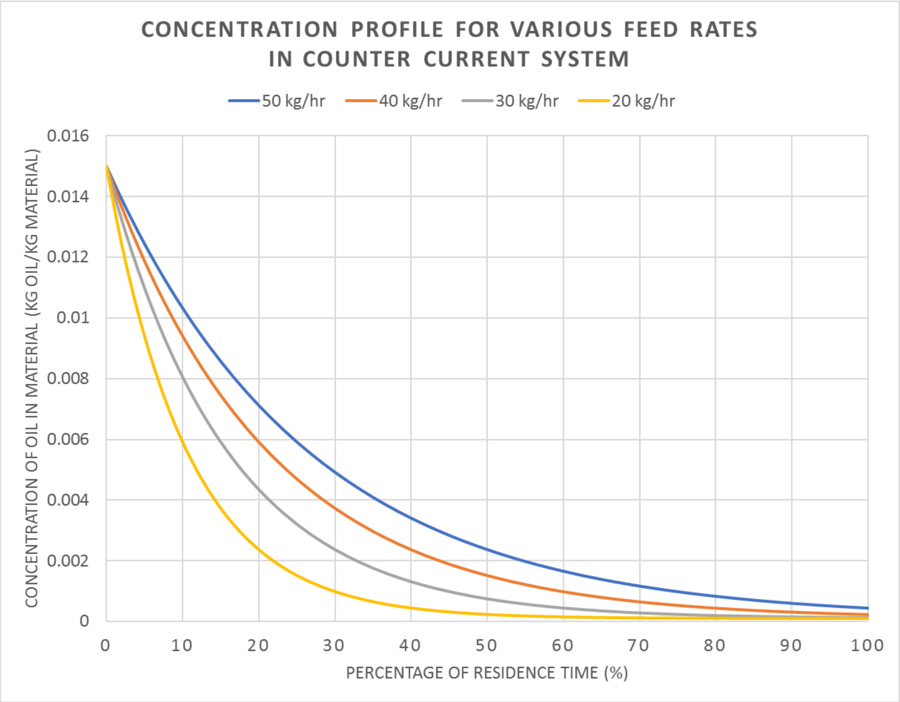


Figure 4-9 Concentration Profiles of Various Feedrates

Using Equation 4-4 the concentration at any point in the reactor can be found, substituting the residence time into the value of t. This allows the outlet concentration to be determined in a plug flow reactor and the concentration in a batch system for any given time after the start of production [16]. Residence time values for the data in Figure 4-9 are presented in Table 4-1. As the concentration of oil in the material approaches equilibrium with steam the rate of extraction drops significantly.

Table 4-1 Residence Time and Outlet Data for Varying Mass Flow Rates

Mass Flow Rate of Material Feed ($kg\ hr^{-1}$)	Residence Time in Column (Hours)	Outlet Material Concentration ($kg\ oil\ kg^{-1}\ material$)
20	100	0.000106
30	66.67	0.000133
40	50	0.000241
50	40	0.000452

Chapter 5 Economic Analysis Results

The results of the economics benefits for operating a batch or counter-current system are presented in this section. A key area explored is the cost to process a ton of material, a major factor in establishing a cost competitive industry, with a reduction in the processing cost of the raw materials, the unit price for oil produced is reduced leading to a possible competitive advantage.

5.1 Cost-Revenue Imbalance

The example presented in Table 4-1 indicates the degree by which the rate of change of the mass of oil changes throughout the length of the column in the counter-current model and the batch model at long residence times. Comparison between the feed rates of 20 and 30 kg/hr of material fed to the reactor, are shown as a 20% increase in material oil content at the exit of the column. Placing a monetary value on the difference between the two provides a comparison for the difference between increased yield and the increased expenditure to achieve that yield. The values for oil revenue, steam cost and fixed over-head cost are presented in Table 5-1.

Table 5-1 Hypothetical Costs for Operation of Plant

Revenue - Unit Price of Oil ($/kg)	$5000
Steam Cost- Unit Price of Steam Generation ($/ton)	$45
Fixed Overhead Costs ($/hr)	$200

Table 5-2 shows the difference in the cost of gaining additional yield versus the generated revenue from the venture. Although an extreme example it highlights the goal of maximising the profit generated rather than the yield obtained from the material. The fixed overhead costs are the main source of the difference between the profits of the two material feed rates.

Table 5-2 Generated Revenue and Cost to Process 1 Ton of Material

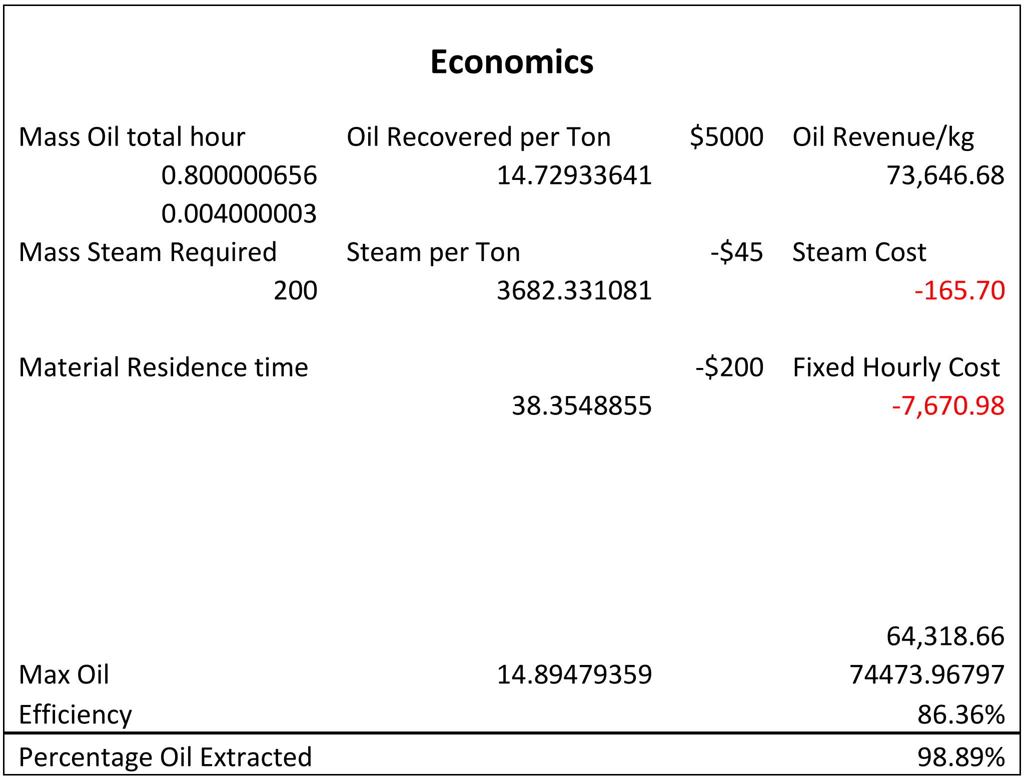
Parameter	Material Flow = 20 kg/hr	Material Flow = 30 kg/hr
Fixed Overhead Costs	-\$10,000	-\$6,667
Steam Consumed *CS	-\$4,500	-\$3,000
Revenue from Extracted Oil	\$74,470	\$74,335
Profit	\$59,970	\$64,670

The values generated in Table 5-2, were generated with an unlimited steam flow rate available. This was described earlier in the sensitivity analysis section as being a situation not recommended as the increased steam through the column would lead to an increase in temperature and ultimately a decrease in the quality of oil produced [6].

5.2 Determination of Ideal Feed Rate

To determine the maximum feed rate of material to the column, an Excel spreadsheet was developed. The Solver add-in was used to determine the maximum rate of profit that could be generated with limitations on the steam input to the column. The output for limit of 200 kg hr^{-1} of steam is shown in Table 5-3, with the parameters set in Table 5-4.

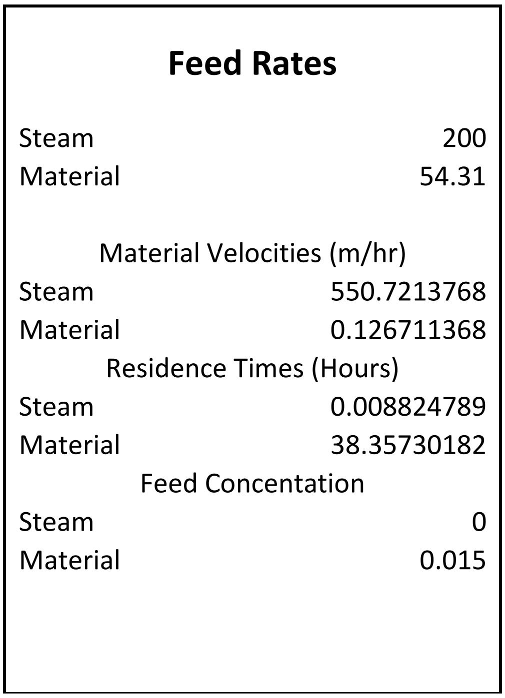
Table 5-3 Output from Optimization Excel Spreadsheet on basis of 1 Ton at 1.5% Oil



Economics

Mass Oil total hour	Oil Recovered per Ton	$5000	Oil Revenue/kg
0.800000656	14.72933641		73,646.68
0.004000003			
Mass Steam Required	Steam per Ton	-$45	Steam Cost
200	3682.331081		-165.70
Material Residence time		-$200	Fixed Hourly Cost
	38.3548855		-7,670.98
			64,318.66
Max Oil	14.89479359		74473.96797
Efficiency			86.36%
Percentage Oil Extracted			98.89%

Table 5-4 Run Condition Input for Optimization Spreadsheet



Feed Rates	
Steam	200
Material	54.31
Material Velocities (m/hr)	
Steam	550.7213768
Material	0.126711368
Residence Times (Hours)	
Steam	0.008824789
Material	38.35730182
Feed Concentation	
Steam	0
Material	0.015

The optimization spreadsheet employed a discrete model of the system with each step calculated by means of Equation 3-11. The cell specifying the material oil concentration was the most important factor in the optimization spreadsheet. The role of the spreadsheet was to generate the material feed rate that would see the steam achieve saturation at the exit of the column.

Ensuring the steam is on the verge or having just reached saturation at the exit of the column allows the material within to be subject to mass transfer for the entire length of the column. This was explored in the sensitivity analysis of the project. Consequently, this ensures that Equation 4-4 will reliably predict the material exit concentration.

For the Solver function, the objective was to maximise the cell labelled; Efficiency. The purpose of the cell is to quantify generated revenue per ton of material processed in relation to the maximum revenue that could be generated. The maximum generated value did not include the costs associated with the extraction of the oil, resulting in the efficiency being both a measure of the revenue generated and the relative cost to generate that revenue. For the data in Table 5-4 the efficiency is stated as 86%. This indicates that the running conditions will result in 86% of the available revenue being converted into profit.

5.3 Batch Model with Lower Overheads

The overheads included in the model heavily influence the point at which the operation becomes unprofitable. As the oil content in the material declines the rate at which it is extracted decreases proportionally. The point at which the revenue trend and cost trend intersect in Figure 5-1, at the 72-hour mark, is with a fixed operating cost of $200 an hour. In comparison to Figure 5-2, the lower cost area can operate until the 100-hour mark without generating a loss.

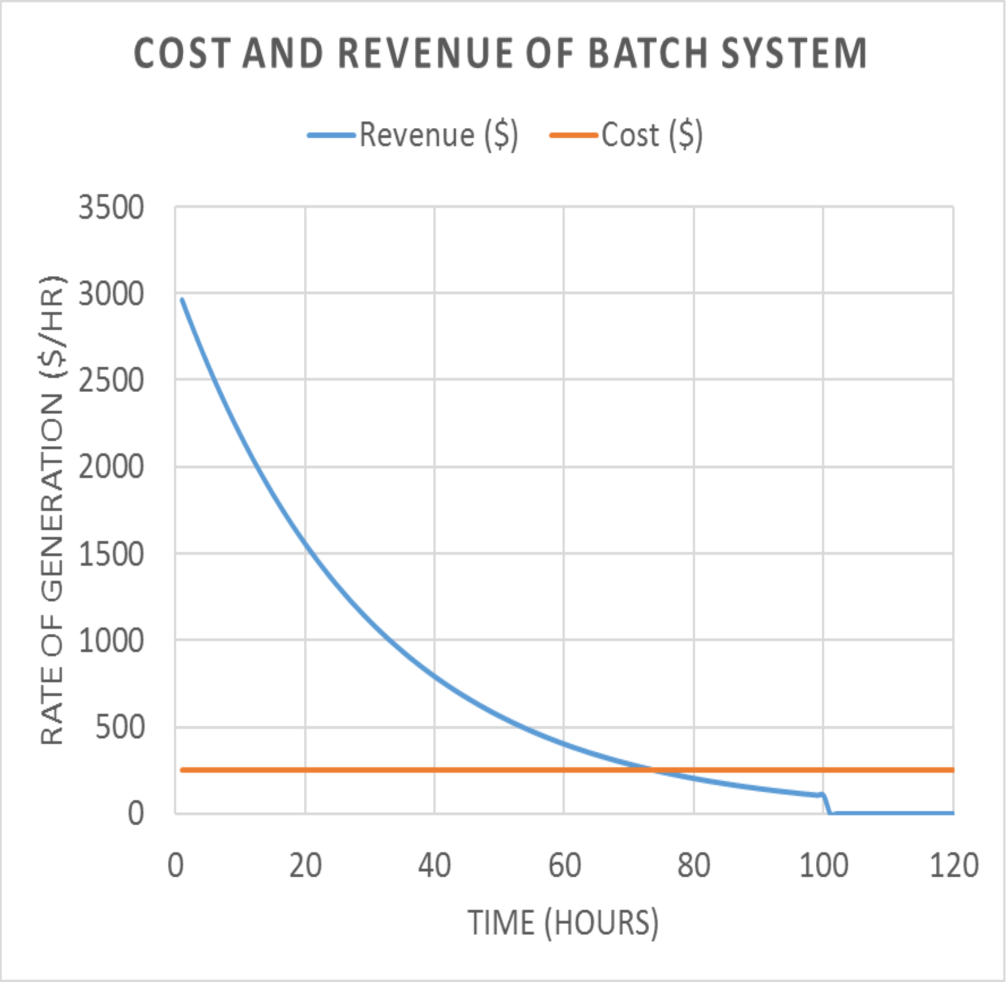


Figure 5-1 Graphically Determining the Decrease in Profitability

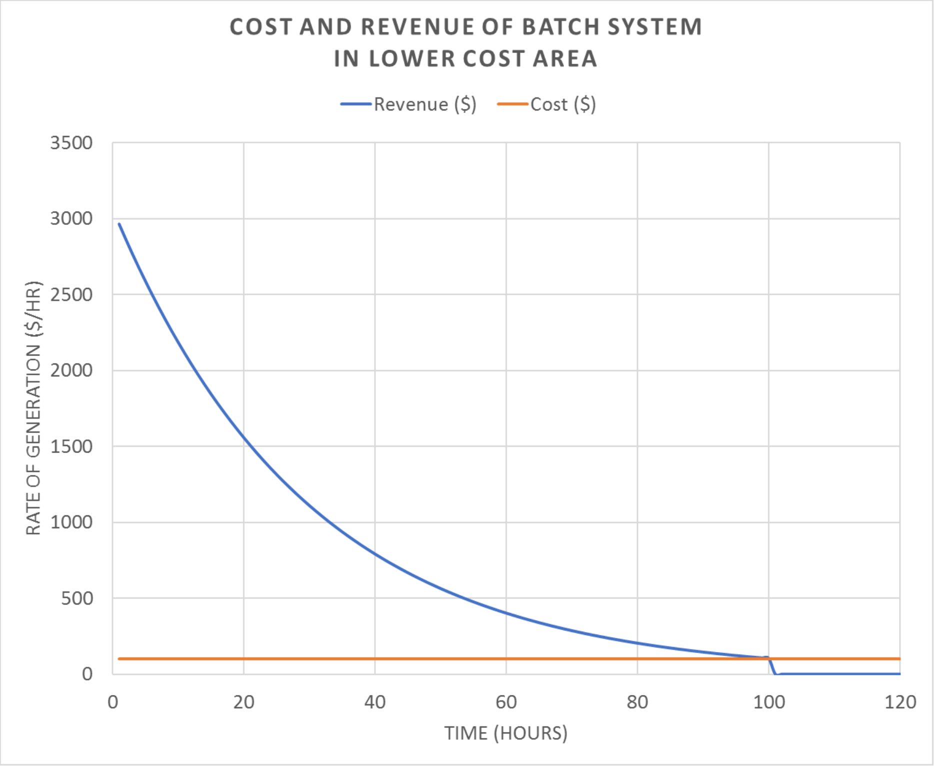


Figure 5-2 Graphical Determination of Profitability in a Low-Cost Area

As the batch system needs to periodically be emptied and reloaded, the operating cost continues past the point at which the steam supply is cut to the process, Figure 5-1 and Figure 5-2 show the cost continuing for 120 hours. It is only with the start of the next batch that the system generates revenue to balance the operating cost. A general flow chart of a batch system is shown in Figure 5-3 [1].

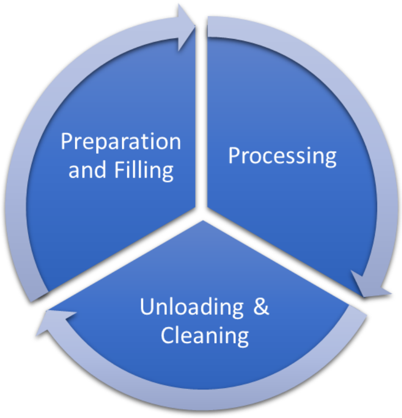


Figure 5-3 General Flow Chart of Batch Processes

The ability to run the process to 100 hours without entering a period of loss generation provides significant benefits. The higher resource recovery rate, assuming the batch is switched off at the point of profit being equal to zero, allows for more oil to be extracted from the same amount of material. A representation of which is included in Figure 5-4. Another benefit is in the relative time that the process is in the processing stage.

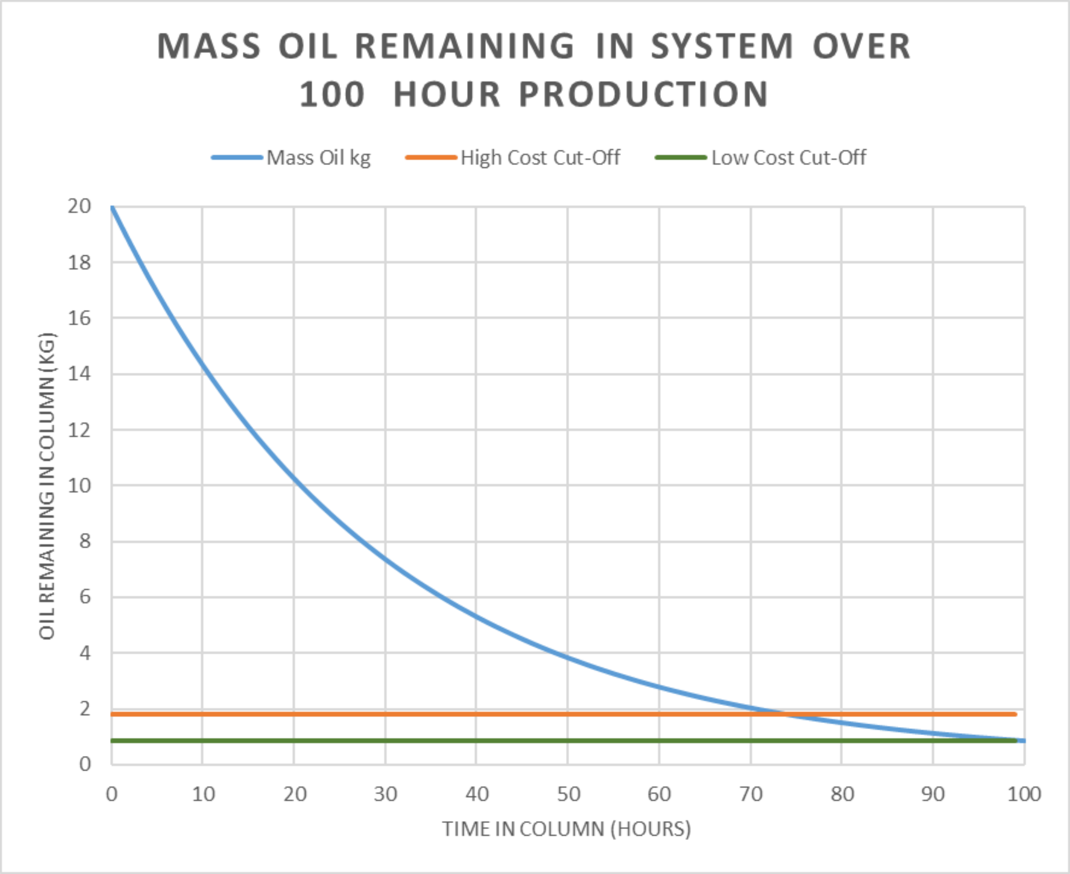


Figure 5-4 Mass of Oil Remaining in System

If the batch is discontinued at the point the profit transitions from positive to negative, and the turnaround time for the batch is 20 hours. The proportion of time in the processing stage is altered between the two example states, this is shown in Table 5-5. The low-cost area will be in the production phase of the batch system for approximately 5% of the total time longer than the high cost area.

Table 5-5 Time to Process Batch with Proportions

Area	Low Cost	High Cost
Processing Hours (Proportion)	100 (0.83)	72 (0.78)
Turnaround Hours (Proportion)	20 (0.17)	20 (0.22)
Total Time for Batch	120	92

The distillation time of the example provided is on the order of 3 – 4 days of actual running time, a relatively slow process in which the turnaround time does not account for a large portion. The production of tea tree oil has a distillation time of 2 -3 hours [18]. To remain profitable, and competitive, the turnaround time for a batch of tea tree oil must have a maximum turnaround proportionate to 20% of the total batch time. The turnaround time would be required to be in the range of 30 – 45 minutes. Essential oils with fast processing times require short turnaround periods to produce oils at meaningful usage of plant capacity.

5.4 Comparison between Systems

The goal of this section is to provide a comparison between the batch and counter-current processes in terms of their production capabilities. The batch system was run to the point at which the rate of profit became zero. And the continuous system operated to the conditions that were determined within Excel to produce the greatest rate of profit per ton processed.

The use of a counter-current system allows for the processing of material to be performed with minimal interruption. The objective of developing the system is to process the material without the requirement of replenishing the material at regular intervals. When operating at a steady state, the counter-current model will have the same rate of production over a long period of operation. Contrasting this is the batch system. Initially the rate of production is high; however, this gradually, over the course of the process, diminishes to a rate that becomes no longer viable.

To compare the batch system and the counter-current system, the average revenue and profit rate was taken over the length of operating. This resulted in the profit rate of the counter-current system being the steady state profit rate. Whereas the batch system required the profit and revenue rates to be averaged over the length of the distillation process.

A comparison of a batch and counter-current system is shown in Table 5-6. The rate of profit for the counter-current system on an hourly basis, is nearly double that of the batch system. The batch system is at the optimal running condition at the beginning of the distillation process, shown in Figure 5-1 Conversely, the counter-current system is operating at the optimal condition for the entirety of the process. This is directly translated into the higher hourly rate of profit and the higher profit on a tonnage basis.

Table 5-6 Profit Comparison of Batch and Counter-current Steam Distillation

	Batch	Counter-current
Hours in Process	80	50
Revenue ($/Hour)	$1,146	$2,002
Cost ($/Hour)	$209	$205
Profit ($/Hour)	$937	$1,798
Profit ($/Ton)	$37,825	$44,938

Comparing the two methods of operations gives for an interesting display on the merits of converting to a counter-current system. The fundamental problem with this analysis is in the modelling of the oil as a single component. Essential oils are comprised of numerous compounds, all which carry over with the steam at different rates. The production of oil by the counter-current system would need to account for the variation in volatility of the constituent compounds. With respect to this, the optimal production rate for the pseudo-oil may produce an oil that is lacking in key distinctive components of the overall essential oil.

5.5 Cost of Counter-current

Calculating the cost of a counter-current system for essential oils cannot be completed on a generic basis. Analysis should be carried out on the essential oil of interest with the results determining the viability of transitioning from batch processing. It has been noted in the literature that introducing and removing large quantities of solid materials from a pressurised vessel involves considerable cost [19]. This considerable cost needs to be justified in the case of the essential oil that the application is intended for.

A consequence of transitioning to a counter-current system is in the loss of process flexibility. A batch process allows for the processing of several different materials using the same equipment [20].

Chapter 6 Conclusion

The investigation of conversion of a batch system of steam distillation to a counter-current model has given an insight into the associated cost benefits and production volumes associated with the transition. Theoretical conversion to the counter-current system, based on the reduction in processing costs, is a feasible solution to increase the competitiveness of the essential oil industry. There are however issues surrounding the technical aspects of the conversion.

The technical aspects, until overcome, bring uncertainty to any estimation of the cost of both converting an existing plant or the construction of a new plant. This uncertainty carries through the determining the plant running cost. For this reason, the fixed overhead running costs were not adjusted in this report as a worst-case scenario. The fixed overheads would be expected to fall, due to the reduction in restocking requirements of the vessel translating to a reduction in the required workforce.

The project has identified that the application of counter-current systems for essential oil extraction must be evaluated on a case by case basis. There is a large time gap between any reported introduction of non-batch processes, which given the processing cost savings does not favour the idea that the system is easily implemented.

Models generated within Simulink have provided a method of simulating the extraction of essential oils from the base plant materials. The accuracy of the model appears to be adequate based on the single material that data was available for. The Simulink model requires further testing of different essential oils with confirmed parameters to determine the robustness of the model.

6.1 Future Recommendations

The use of MATLAB/Simulink as simulation software presented numerous challenges, and would not be recommended in the future. Dedicated modelling software for chemical process engineering exist, for example ASPEN Plus, developing the system within these environments are likely to increase the accuracy of the modelling, with additional testing of a physical process. In addition to the accuracy, the addition of pressure and temperature drops across the column would further improve the model. These are major factors in the design of distillation columns and have been disregarded to the detriment of the project.

As there is also little information in the published literature for processing essential oils by counter-current steam distillation. A bench scale or small pilot scale test to provide experimental data would allow for improved model development and validation. With the implementation of a trial plant, there are numerous parameters to monitor. A system to measure the input and output concentration of the oil in material in near real time would increase the controllability of the plant.

The Rural Industries Research and Development Corporation of the Australia Government should be contacted in the future to explore collaboration with future researchers in Australia.

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Appendix A

Appendix Table A-1 Calculation of Costs for Differing Material Flows

Parameter	Material Flow = 20 kg/hr	Material Flow = 30 kg/hr
Residence Time Hours * Fixed overhead rate	100*200	66.67*200
Steam Consumed *unit cost of steam	200 * 100* 0.045	200* 66.67*0.045
Outlet Material Concentration (kg oil kg^{-1} material)	0.000106	0.000133
Mass of oil extracted	1000*(0.015 - 0.000106) *5000	1000*(0.015 - 0.000133) *5000