

# Design of a system that can harvest essential oils from plants for pharmaceutical uses

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## Abstract

There are several methods available used to extract essential oils. There are conventional and non-conventional methods with the latter being more efficient than the former. Currently at the Pharmacy department of the University of Zimbabwe there is no functional extraction machine. The ones that were used have worn out and thus have since been decommissioned. With this in mind, the present project sets out to solve this problem by coming up with a new system design that uses the subcritical water extraction method to extract essential oils faster and more efficiently as compared to the previously available steam distillation machine. The machine has a pressure tank which acts as the extraction vessel and the subcritical water is passed through the plant material inside the vessel. The temperature of the water inside is maintained at a specific temperature. The tank is pressurized using a pump and valves to maintain the pressure inside the extraction vessel. The products of the extraction process cooled and separated. The extraction tank and the pipes are analyzed using Autodesk Inventor to determine the safety of the design.

## Keywords

Subcritical, Water, Extraction, Essential, Oils

## 1. Introduction

According to Abdelouaheb and Amadou (2012) essential oils' broad medicinal values have been realized since ancient times and continue to be of prime importance until now. There has been a growing demand for natural medicine such as essential oils due to an increase in demand to reduce the use of synthetic chemicals to fight infections. This is due to the fact that microorganisms are resistant to synthetic antimicrobials (Rassem, et al., 2016; Nazzaro, et al., 2017). The increasing knowledge of the composition of essential oils and their pharmaceutical uses have led to development of their cultivation and markets where they represent an important industrial raw material for the pharmaceutical industry of commercial importance (Marques, et al., 2012; Abdelouaheb & Amadou, 2012).

Presently there are several methods of extraction of essential oils, such as hydrodistillation, Solvent extraction, steam distillation, cohobation and maceration (Rassem, et al., 2016) and among all these methods the most used is steam distillation, especially for commercial scale production (Tongnuanchan & Benjakul, 2014). The present project intends to introduce large scale local production of essential oils for pharmaceutical uses by designing a system that can harvest the oils from selected plants.

## 1.1 Problem statement

Essential oils extraction machines in Zimbabwe are outdated.

## 1.2 Aim

To design a system that can extract essential oils from plants for pharmaceutical uses.

## 1.3 Objectives

- To design a machine that is more efficient than the currently available machine at the University of Zimbabwe Pharmacy Department.
- To design a machine that can be used by a person that does not have knowledge of essential oils extraction.
- To design an electric powered machine.

## 1.4 Justification

Improved health. Essential oils components have many uses in pharmacology and can be used as an appropriate therapy for many pathologies (Abdelouaheb & Amadou, 2012). Economic benefits. Essential oils are a major group of agro-based industrial products with applications in not only pharmaceuticals industries but also in the food products, drinks, perfumes and cosmetics industries as they play different roles. According to Abdelouaheb & Amadou (2012) essential oils' economic importance is indisputable as the source of these molecules are inexhaustible.

## 2. Literature review

### 2.1 What are essential oils?

Essential oils are defined as hydrophobic volatile liquids that contain aromatic compounds, obtained from plants. They are plant-based oils that contain a complex mixture of over 300 different compounds including aromatic compounds that belong to various chemical classes, such as alcohols, oxides, amines, amides, heterocycles, hydrocarbons, phenols, aldehydes, esters, ketones and mainly the terpenes (Rassem, et al., 2016; Dhifi, et al., 2016). Essential oils can be extracted from almost any part of the plant for example, the leaves, flowers, roots, rhizomes, seeds, fruits, wood and bark. They are located in the cytoplasm of certain plant cell secretions in multiple plant organs such as, secretory hairs, epidermal cells, internal secretory cells and secretory pockets (Dhifi, et al., 2016).

### 2.2 Pharmacological activities of essential oils

Antibacterial activity. Due their hydrophobicity which allows them to partition into lipid of bacterial cell membranes, some essential oils are capable of disrupting the structure of the cell membranes of bacteria making them more permeable, and causing the loss of ions and cellular molecules through leakage leading to the cells' death (Abdelouaheb & Amadou, 2012; Dhifi, et al., 2016). Antioxidant activity. The antioxidant properties of essential oils depend on their composition. Essential oils with phenolic compounds, such as thymol (a monoterpene phenol,  $C_{10}H_{13}OH$ , found in oil extracted from thyme) and carvacrol (a monoterpanoid phenol), and secondary metabolites with conjugate double bonds show antioxidative properties. The phenolic compounds possess redox properties which are very important in neutralizing free radicals and peroxide decomposition (Dhifi, et al., 2016; Baser & Buchbauer, 2010). In line with Aruoma (1998) and Kamatou and Viljoen (2010) essential oils being able to scavenge for free radicals play an important part in prevent diseases such as brain dysfunction, cancer, heart disease, and immune system decline which result from cellular damage brought about by free radicals (cited in Dhifi, et al., 2016). Anti-inflammatory activity. According to Maruyama, et al., (2005) essential oils, for example *Melaleuca alternifolia* essential oil as pointed out by Koh, et al., (2002) have been used in clinical settings to treat inflammatory diseases, such as rheumatism, allergies or arthritis, which, according to Hart, et al., (2000) has its anti-inflammatory activity

correlated with its major compound  $\alpha$ -terpineol, which in line with Maruyama, et al., (2005) acts by inhibiting the release of histamine or reducing the production of inflammatory mediators (cited in Dhifi, et al., 2016). Anti-cancer activity. According to Pyun and Shin (2006) it is known that certain foods contain anti-cancer agents, for example, as stated by Milner (2001) garlic essential oils contain sulphur compounds known for their preventive effects against cancer (cited in Dhifi, et al., 2016). Some of the prominent compounds in anti-cancer activity are d-limonene (main constituent of essential oils of citrus fruit peel oils) and perillyl alcohol (Baser & Buchbauer, 2010).

### 2.3 Essential oils extraction

Essential oils can be extracted from different plant parts using various methods and machines. The manufacture of essential oils and extraction is dependent on the botanical material used, its state and form. The extraction method used also affects the quality of the essential oil. Inapt extraction of the oil can lead to the changing of the chemical signature of the oils, thus altering its bioactivity and natural properties (Tongnuanchan & Benjakul, 2014). Here are some extraction methods and the machines currently available that use these methods in table 1:

Table 1. Essential oils extraction methods

Traditional Methods	Non-conventional Methods
Hydro distillation	Supercritical fluid extraction
Steam distillation	Microwave assisted hydro distillation
Hydro diffusion	Ultrasound assisted extraction
Solvent extraction	Subcritical water extraction
Expression	

### 2.4 Subcritical water extraction

#### 2.4.1 Subcritical water extraction

The subcritical water extraction (SWE) is a modern technique that uses green technology to extract biochemical compounds such as essential oils from plant material (Patrycja, et al., 2017). The use of large quantities of organic solvents in the extraction of these compounds via conventional methods of extraction raises issues of safety, environmental concern and cost-effectiveness. By taking advantage of the special properties of water under subcritical conditions SWE represents a safe, cost effective environmentally-friendly alternative (Sefater, et al., 2016). SWE eliminates the use of organic solvents in the extraction of essential oils which contributes to quality and applicability of the plant extracts by replacing organic solvents with water which is cheap, safe and efficient (Patrycja, et al., 2017). According to Krishan, et al., (2017) SWE is the most promising engineering approach that offers an eco-friendly method for extraction of compounds from plants. According to A. Haghighi & Khajonoori (2013) researches published in recent years show that SWE is cleaner, faster and cheaper than conventional methods.

The technique uses water under subcritical conditions where high temperature and pressure reduce its viscosity, surface tension and dielectric constant strongly making low polarity organic compounds have solubilities about 20 000-fold in water at 200°C than at 25°C (PABLO, et al., 2016). In addition, SWE is an easy-to-adopt method with wide range of compounds of varying chemical and physical characteristics to be extracted and water does not exhibit toxicity associated problems like organic solvents. This selectivity of SWE allows for the manipulation of the composition of the extracted compounds by changing the operating parameters (Sefater, et al., 2016).

#### 2.4.2 Scientific background

Subcritical water refers to hot water between the boiling and critical point (100°C – 374°C) under sufficient pressure to keep it in the liquid phase (M.S. Yogendra, et al., 2011; Sefater, et al., 2016). Water has many unusual properties due to its very strong hydrogen bonds between its molecules. When water is superheated the hydrogen bonds break, reducing the dielectric constant and polarity of the water. This increases the concentration of hydrogen ions at elevated temperatures and also, the water starts to act as a solvent for hydrophobic matter. By modifying the temperature of the water the dielectric constant value and polarity can be affected which gives rise to the ability of subcritical water to selectively extract compounds of varying polarity (Shitu, et al., 2015; AWALUDDIN, et al., 2016; Yajie, et al., 2017; Sefater, et al., 2016).

At room temperature and pressure water has one of the highest dielectric constants ( $\epsilon = 80$ ) which makes it unsuitable as a solvent for the extraction of low polarity compounds. When the temperature is raised the dielectric constant decreases to  $\epsilon = 27$ , which is around the range of organic solvents such as ethanol, methanol and acetone. The extraction is based on the enhanced solubility of the compounds and mass transfer which is brought about by the processes of diffusion and convection. Subcritical water has reduced viscosity and increased diffusivity allowing for better penetration into the matrix of the plant material (Sefater, et al., 2016).

### 2.4.3 System set up

A Typical SWE unit would consist of three subunits: the pumping section, the extraction subunit and extract recovery subunit. A schematic diagram of a SWE unit shown in figure 1 as proposed by Ghoreishi & R. Gholami, (2009).

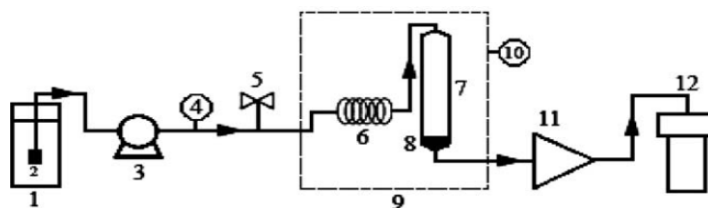


Figure 1. System setup of subcritical water extraction

- A. Pumping section
  - (1) Solvent reservoir or tank
  - (2) Filter
  - (3) Pump
  - (4) Pressure gauge
  - (5) Valve
- B. Extraction subunit
  - (6) Coil preheater
  - (7) Extraction cell
  - (8) Cotton wool filter
  - (9) Oven
  - (10) Temperature controller
- C. Extract recovery subunit
  - (11) Backpressure regulator
  - (12) Collection container

All the joints, fittings, tubings, valves and vessels of the extraction unit are made out of stainless steel in order to resist corrosion.

### 2.5 Extraction process

The plant material is ground to the required sample particle size. The material sample to be extracted is then loaded into the extraction cell which is located inside the oven at a predetermined sample to liquid ratio. The system is flushed with nitrogen gas to remove the air inside and avoid possible oxidation during extraction process. The oven is set to the desired temperature using the temperature controller with a thermocouple to maintain the selected temperature value and the backpressure regulator is locked. Distilled water in the solvent tank is purged by nitrogen to remove dissolved oxygen. The distilled water is then pumped at a certain flow rate through the coil preheater inside the oven so that the water acquires enough thermal energy to be brought to the required temperature under which the extraction is taking place. The filter makes sure that no solid particles enter the pump. The pressurized hot water flows into the extraction cell where the extraction process takes place. During the extraction process a working range of pressure in

the system is kept by setting the pressure regulator and is checked by a manometer. The backpressure regulator is opened depending on the desired working pressure and the hot extract flows into the collection container through a cooling coil or a heat exchanger where it is cooled down and collected through the outlet (PABLO, et al., 2016; Sefater, et al., 2016; Nataša, et al., 2018; Patrycja, et al., 2017). A process flow diagram of the SWE method is shown in figure 2.

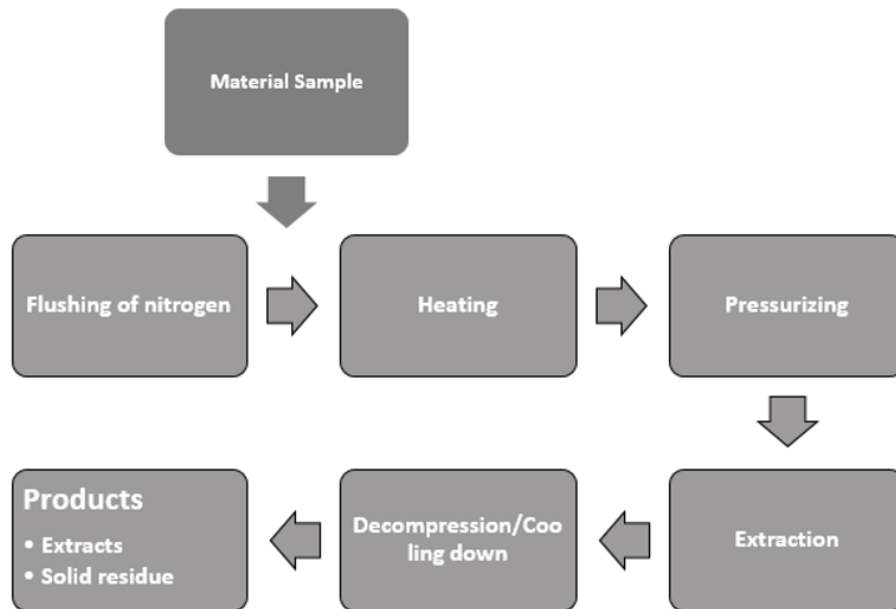


Figure 2: Process for subcritical water extraction

The SWE process is typically done using one of the three modes viz: the static, dynamic or the combined mode which combines both the static and dynamic modes. The static mode involves the retaining of the solvent in the extraction cell that is no outflow of water is allowed until extraction equilibrium is reached between the solvent and the compounds. The system is then purged with fresh solvent and the extract is collected. The extraction is done in batches (Sefater, et al., 2016). In this process the efficiency of recovery is controlled by the solubility of the compounds and the partition equilibrium constant. Thus, the disadvantage of the static mode is that the solvent might reach equilibrium soon and no further extraction will happen regardless of the temperature, retention time or pressure (Sefater, et al., 2016). In the dynamic mode a continuous flow of solvent is allowed through the extraction cell throughout the extraction process. This gives room for fresh solvent to be introduced into the extraction cell which avoids or minimizes solute-fluid equilibrium if correctly optimized. A greater volume of solvent is used as compared to the static mode which means energy efficiency is reduced (Sefater, et al., 2016). For the combined static-dynamic mode the static mode is first executed for a period and then the extraction is followed by a dynamic mode. This mode was found to be advantageous with good precision (Sefater, et al., 2016).

### 3. Materials and methods

The technical specifications for the design are shown in table 2.

Table 2. Technical specifications

Component	Quantity
Volume of extraction vessel	10l
Extraction temperature range	100°C – 374°C
Extraction pressure range	0.1 MPa – 22 MPa
Automation	Yes
Extraction solvent	Water
Material	Stainless steel
Pump	Yes
Pressure gauge	Yes
Valves	Yes
Backpressure regulator	Yes

## 4. Results and discussion

### 4.1 Extraction vessel design

A length/diameter ratio of 3 is chosen for the vessel. Thus the internal diameter is:

$$\text{Height} \quad h = 3d_i$$

$$d_i = \sqrt[3]{\frac{4v}{3\pi}}$$

$$d_i = \sqrt[3]{\frac{4 \times 0.0075}{\pi \times 3}}$$

$$d_i = 0.147 \text{ m}$$

$$\text{Say} \quad d_i = 150 \text{ mm}$$

The design of the extraction vessel will be done under the assumption that the vessel is a thick cylinder subjected to an internal pressure of 22.064 MPa only.

The circumferential stress or maximum allowable stress,  $\sigma_H$  is:

$$\begin{aligned} \sigma_H &= 0.8 \times \sigma_y \\ &= 0.8 \times 230 \text{ MPa} \\ &= 184 \text{ MPa} \end{aligned}$$

#### 4.1.1 Determination of cylinder thickness $t$

From the Lamé's equations and the maximum shear stress theory:

$$t = R_i \left[ \sqrt{\frac{\tau}{(\tau - P)}} - 1 \right]$$

Since the value of shear stress is usually taken as one half the tensile stress, it follows that:

$$\tau_{max} = \frac{\sigma_{H(max)}}{2}$$

$$\text{Thus; } t = R_i \left[ \sqrt{\frac{\sigma_H}{(\sigma_H - 2P)}} - 1 \right]$$

$$\text{When } R_i = 75 \text{ mm}$$

$$\sigma_H = 184 \text{ N/mm}^2, \text{ and}$$

$$P = 22.064 \text{ N/mm}^2$$

$$t = 75 \left[ \sqrt{\frac{184}{(184 - (2 \times 22.064))}} - 1 \right]$$

$$t = 11.021 \text{ mm}$$

Say  $t = 15 \text{ mm}$

#### 4.1.2 Cylinder head design

The design of the cylinder head involves the determination of the head's thickness  $t_h$ . This is a rigidly fixed type of connection between the head and the cylinder. Since the pressure inside the vessel will be uniformly distributed the head thickness may be found as follows.

$$t_h = \frac{P(d_i^2 + 4C^2)}{16\sigma_H C}$$

Where  $C = \text{camber or radius of the dish plate}$

For hemispherical heads  $C = \frac{d_i}{2}$

$$t_h = \frac{P(d_i)}{2\sigma_H}$$

$$t_h = \frac{22.064(150)}{2 \times 184}$$

$$t_h = 9 \text{ mm}$$

Say  $10 \text{ mm}$

#### 4.1.3 Extraction vessel closure design

The extraction vessel closure is made out of the same material as the cylinder and has threads and a groove to place an O ring to seal the vessel. The closure is made up of two parts one with external threads which is screwed inside the cylinder and the other has internal threads and is screwed onto the outside of the vessel.

#### 4.2 Pipe selection

The appropriate pipe for this system is the schedule 80 (80S) pipe with the in accordance with the ANSI standards.

- Outside diameter  $D_o = 33.4 \text{ mm}$  (ANSI/ASME B36.19M)
- Wall thickness  $t = 4.55 \text{ mm}$  (ANSI/ASME B36.19M)
- Internal diameter  $D_i = 24.3 \text{ mm}$

#### 4.3 Heat exchanger design

##### 4.3.1 The effectiveness-NTU method

$$\text{Effectiveness } \epsilon = \frac{\text{actual heat transfer}}{\text{maximum possible heat transfer}}$$

##### 4.3.1.1 Determining the actual heat transfer

Now considering the counter flow heat exchanger the actual heat transfer is (Holman, 2010):

$$\text{Actual heat transfer } q = \dot{m}_h c_h (T_{h_1} - T_{h_2}) = \dot{m}_c c_c (T_{c_1} - T_{c_2})$$

Where the subscripts

$h$  = hot fluid  
 $c$  = cold fluid  
 $\dot{m}_h$  = mass flow rate of fluid  
 $c_h$  = specific heat capacity of hot fluid  
 $T_{h_1}$  = inlet temperature of hot fluid  
 $T_{h_2}$  = outlet temperature of hot fluid  
 $\dot{m}_c$  = mass flow rate of cold fluid  
 $c_c$  = specific heat capacity of cold fluid  
 $T_{c_1}$  = outlet temperature of cold fluid  
 $T_{c_2}$  = inlet temperature of cold fluid

The exchanger is being designed for the maximum temperature difference that means the inlet temperature of the hot fluid will be 374°C. The desired outlet temperature of the hot fluid is 95°C.

#### 4.3.1.2 Determining the maximum heat transfer

The maximum heat transfer is obtained from the fluid that undergoes the temperature change that is equal to the maximum temperature difference in the heat exchanger ( $T_{h_1} - T_{c_2}$ ) (Holman, 2010). The maximum temperature difference occurs to the fluid with the minimum value of  $\dot{m}c$  thus the maximum possible heat transfer is given by the equation:

$$\text{Maximum possible heat transfer } q_{max} = (\dot{m}c)_{min} (T_{h_1} - T_{c_2})$$

The flow rate of the cooling water (i.e. cold fluid) must be higher than that of the extraction solution (i.e. hot fluid). With this in mind, a flow rate of the cooling water has been chosen which is 5 l/min (0.0833 kg/s). This means that the maximum possible heat transfer will be given by:

$$\text{Maximum possible heat transfer } q_{max} = \dot{m}_h c_h (T_{h_1} - T_{c_2})$$

#### 4.3.1.3 Determining the effectiveness

The effectiveness  $\epsilon$  will be:

$$\begin{aligned} \text{Effectiveness } \epsilon &= \frac{\dot{m}_h c_h (T_{h_1} - T_{h_2})}{\dot{m}_h c_h (T_{h_1} - T_{c_2})} \\ &= \frac{(T_{h_1} - T_{h_2})}{(T_{h_1} - T_{c_2})} \\ &= \frac{(374 - 95)}{(374 - 25)} \\ &= 0.799426934 \\ &= 80\% \end{aligned}$$

#### 4.3.1.4 The number of transfer units (NTU)

The effectiveness of a heat exchanger can be expressed in a different way depending on the type of heat exchanger (Holman, 2010). Within these expression the terms  $\frac{UA}{c_{min}}$  is the number of transfer units (NTU) and it indicates the size of the heat exchanger (Holman, 2010). Where  $c_{min} = (\dot{m}c)_{min}$ . The relations between the effectiveness and the NTU is represented in the form of a chart for different heat exchanger arrangements as shown in figure 3.



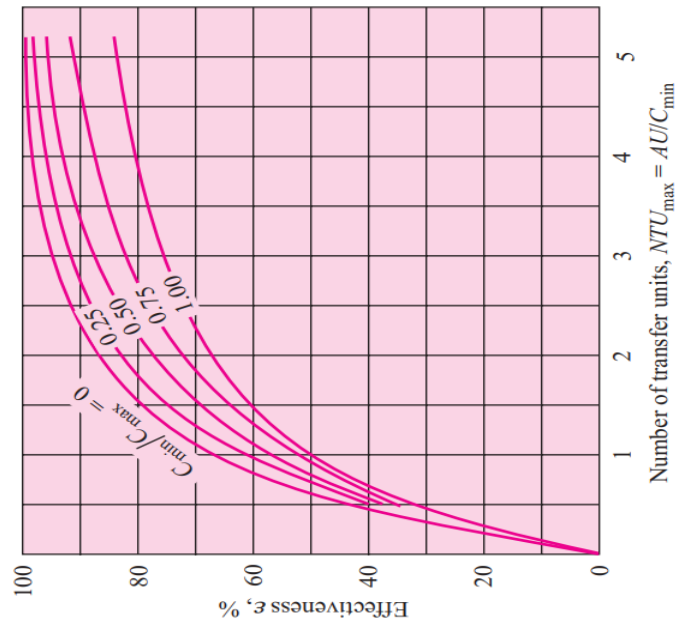


Figure 3. Effectiveness for counter flow exchanger performance (Holman, 2010)

Equations for effectiveness  $\epsilon$  in terms of NTU and capacity ratio ( $c_{min}/c_{max}$ ), and the NTU in terms of effectiveness and capacity ratio are available. The main goal is to determine the area required to transfer a certain amount of heat energy per unit time considering the temperature difference available (Sinnott, 2005). Heat transfer  $q$  (in watts, W) across an area  $A$  ( $m^2$ ) is given by (Sinnott, 2005):

$$q = UAF\Delta T_m$$

Where  $U$  = overall heat transfer coefficient,  $W/m^2 \cdot ^\circ C$

$\Delta T_m$  = mean temperature difference,  $^\circ C$

$F$  = the correction factor

Thus the area  $A$  required to transfer the energy will be given by:

$$N = NTU = \frac{UA}{c_{min}}$$

$$A = \frac{NC_{min}}{U}$$

$$c_{min} = \dot{m}_h c_h$$

Ignoring the effects of temperature on the value of  $c_h$  we have the value of  $c_h$  as  $4180 J/kgK$  assuming that the essential oils do not affect the heat capacity of the extraction water and assuming the density remains constant.

Thus

$$c_{min} = 0.0417 \times 4180$$

$$= 174.167 W/K$$

And

$$c_{max} = \dot{m}_c c_c$$

$$= 0.0833 \times 4180$$

$$= 348.194 W/K$$

Thus the capacity ratio  $C = \frac{C_{min}}{C_{max}} = \frac{174.167}{348.194} = 0.5$

Since the effectiveness is known and the capacity ratio has been found, the NTU can now be found using one of the equations in the tables above. For a one shell pass and 2, 4, 6 tube pass shell and tube heat exchanger, the following equation will be used.

$$N = -\sqrt{(1 - C^2)} \times \ln \left[ \frac{\frac{2}{\epsilon} - 1 - C - \sqrt{(1 + C^2)}}{\frac{2}{\epsilon} - 1 - C + \sqrt{(1 + C^2)}} \right]$$

Thus 
$$N = -\sqrt{(1 - 0.5^2)} \times \ln \left[ \frac{\frac{2}{0.8} - 1 - 0.5 - \sqrt{(1 + 0.5^2)}}{\frac{2}{0.8} - 1 - 0.5 + \sqrt{(1 + 0.5^2)}} \right]$$

$$= 2.500$$

The value of U is 800 as chosen from table shown below.  
Area required for the heat transfer will be:

$$A = \frac{NC_{min}}{U}$$

$$= \frac{2.5 \times 174.167}{800}$$

$$= 0.544 \text{ m}^2$$

Using the average velocity used for the design of pipes:

$$v = \frac{R_e \times \mu}{D_i \times \rho_v}$$

$$= \frac{4000 \times (0.181282 \times 10^{-3})}{24.3 \times 1000}$$

$$= 0.0298 \text{ m/s}$$

The flow area  $A_f$  is calculated as follows:

$$A_f = \frac{\dot{m}_h}{\rho v}$$

$$= \frac{0.0417}{1000 \times 0.0298}$$

$$= 0.001399328 \text{ m}^2$$

This area is the product of the number of tubes and the flow area per tube. Assuming that the same tubes designed earlier are also used in the heat exchanger then;

$$A_f = n \frac{\pi D_i^2}{4}$$

$$n = \frac{4A_f}{\pi D_i^2}$$

number of tubes  $n = 3.017$

The length of the tubes is found using the area A for a two tube pass exchanger as follows:

$$A = 2n\pi D_i L$$

$$L = \frac{A}{2n\pi D_i}$$

$$\begin{aligned} &= \frac{0.544}{2 \times 3 \times \pi \times 0.0243} \\ &= 1.188 \text{ m} \end{aligned}$$

#### 4.3.1.5 Pump selection

A pump reciprocating pump with a head of up to 22 MPa and with flow rate ranging from 1 l/min to 5 l/min is chosen for the design

#### 4.4 Von Mises analysis for assembled pressure tank

The maximum stress being 207.5 MPa. Since it is required that the von Mises stress be less than the yield stress of the material for the design to be considered safe, the designer can safely assume at this point that the design is indeed safe since,  $\sigma_v < \sigma_y$  (i.e. 207.5 MPa < 230 MPa)

#### 4.5 Von Mises stress analysis for pipes

The maximum von Mises stress shown in the analysis of the pipes is 106.6 MPa which is less than the yield stress of the material used, thus the design is indeed safe. In addition to the von Mises stress analysis, the strain analysis was also carried and the results were as follows:

*Maximum equivalent strain of the extraction tank* =  $9.921 \times 10^{-4}$  and  
*Maximum equivalent strain of the pipe* =  $4.932 \times 10^{-4}$

### 5. Recommendations and conclusion

Although the design has been shown to be safe, it has done so by a small margin. In light of this some improvements may be done to the design to improve its strength and reliability and these are;

- The design safety factor has to be increased in order to stronger components
- The heating method may need some improvements. The oven can be replaced by a more efficient heating element which heats the vessel directly.
- The design needs to be automated.

The present design has the potential of delivering a more efficient, faster and a greener method of essential oils extraction which will produce high quality products that satisfy the world's standard.

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