# Multi-feed plant design for production of mono nitro aromatic product using CSTR in series

by

**Riya G. Deshpande**

**30EE21J26001**

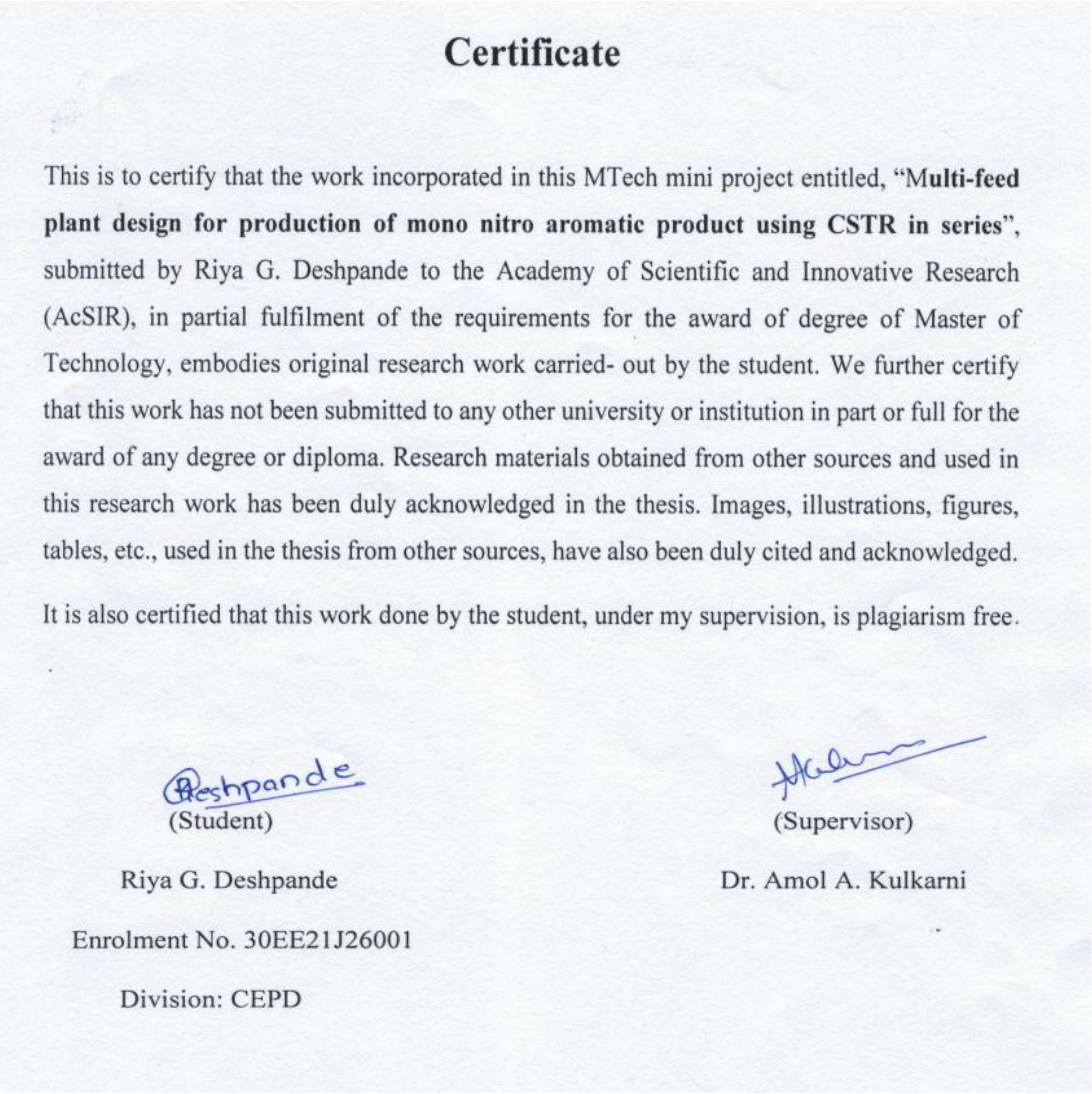
**Dissertation Project Report Submitted to the**
**Academy of Scientific & Innovative Research**
**for the award of the degree of**
**MASTER OF TECHNOLOGY**
**in**
**Engineering Sciences**

**Under the supervision of**
**Dr. Amol A. Kulkarni**

**CSIR-National Chemical Laboratory, Pune- 411038, India.**

**Academy of Scientific and Innovative Research**
**AcSIR Headquarters, CSIR-HRDC campus**
**Sector 19, Kamla Nehru Nagar,**
**Ghaziabad, U.P.- 201 002, India**

**October-2022**



# Certificate

This is to certify that the work incorporated in this MTech mini project entitled, "M**ulti-feed plant design for production of mono nitro aromatic product using CSTR in series**", submitted by Riya G. Deshpande to the Academy of Scientific and Innovative Research (AcSIR), in partial fulfilment of the requirements for the award of degree of Master of Technology, embodies original research work carried- out by the student. We further certify that this work has not been submitted to any other university or institution in part or full for the award of any degree or diploma. Research materials obtained from other sources and used in this research work has been duly acknowledged in the thesis. Images, illustrations, figures, tables, etc., used in the thesis from other sources, have also been duly cited and acknowledged.

It is also certified that this work done by the student, under my supervision, is plagiarism free.

Deshpande
(Student)

Riya G. Deshpande

Enrolment No. 30EE21J26001

Division: CEPD

(Supervisor)

Dr. Amol A. Kulkarni

# Index

## Contents

## List of figures

## List of tables

# Abstract

With the increase in demand for the nitroaromatic products which are used for synthesis of organic compounds, pharmaceutical intermediates, explosives, dyestuffs, agrochemical products, etc, there is a need to produce a large quantity of these products. In industries, batch mode of operation is utilized which provides certain disadvantages. Also, all the processes designed for aromatic nitration are based on the single feed-single product approach. In this study, a multi feed continuous optimized process flow was proposed. The process was designed for five aromatic substrates with complete mass and energy balance. Detailed design calculations and specifications were provided for the proposed process. The cost for annual production was estimated along with the cash flows and profitability analysis for each substrate. Based on all the factors, a typical plant layout for the aromatic nitration is suggested.

## 1. Introduction:

Aromatic nitration is an important class of reaction studied at laboratory and industrial scales due to its variety of applications. Nitro-aromatic products and their derivatives are used in the synthesis of organic compounds, as intermediates in the pharmaceutical and agrochemical sector, manufacture of synthetic dyestuffs and pigments, solvents, and explosives [1].

Nitro aromatic product, 5 nitro salicylic acid which is a major isomer upon further reduction yields 5-amino salicylic acid (mesalazine) which finds applications in the treatment of ulcerative colitis [2]. The sodium salt of 5-nitroguaiacol is used in large quantities as a plant growth regulator in the agricultural sector [3]. Nitro chlorobenzene and its derivatives are majorly used to produce industrially important chemical compounds, dyes, and antioxidants used in rubber processing [4]. It is a primary raw material used for manufacturing the most common generic medicine available globally, paracetamol. Hence the demand is ever increasing [5]. Nitro methyl benzoate and its derivatives are used to produce antibiotics, to produce dyes, foam, analgesics, anti-degradants for rubbers, and synthetic fibres [6]. Halo salts of nitro trifluoromethyl benzene are used for manufacturing fungicides [7]. Due to the increase in demand, there is a need to build a continuous plant to produce nitroaromatic products.

In industry, most of these reactions are performed in batch or semi-batch mode of operation which can lead to inadequate heat transfer area inducing difficulty in heat management, large reaction times due to slow additions of reactants, observed variations in each batch concerning conversion, selectivity and yield, use of excess nitric agents which occupies a significant volume of the reactor and the spent acid generated need to be neutralized thereby needing a large quantity of water, and generates a significant amount of inorganic salts, the reaction needs to be performed at low temperatures to ensure safety [8]. Due to the mentioned disadvantages, the production per day is low and the product quality is inconsistent.

In this work, a multi-feed continuous plant is designed to produce nitroaromatic products. Nitration of Salicylic acid, Acetyl guaiacol, Chlorobenzene, Methyl benzoate and trifluoromethoxy benzene is carried out continuously using 3 CSTRs in series to produce 1 TPD of the major desired isomer. Nitric acid/ Mixed acid is used as a nitrating agent. A process is proposed to achieve the desired results followed by complete mass balance and energy balance is performed for each substrate. Based on the data obtained CSTRs, vessels, decanter, pumps, and heat exchangers are sized and designed. The design of distillation columns is carried out using CHEMCAD. Reboiler and condenser duties are calculated. Costing analysis and profit analysis is provided with respect to each substrate. Finally, a plant layout is proposed.

## 2. Block Diagram:

The figure depicts the general process flow for manufacturing the nitro aromatic products via nitration of aromatic substrates.

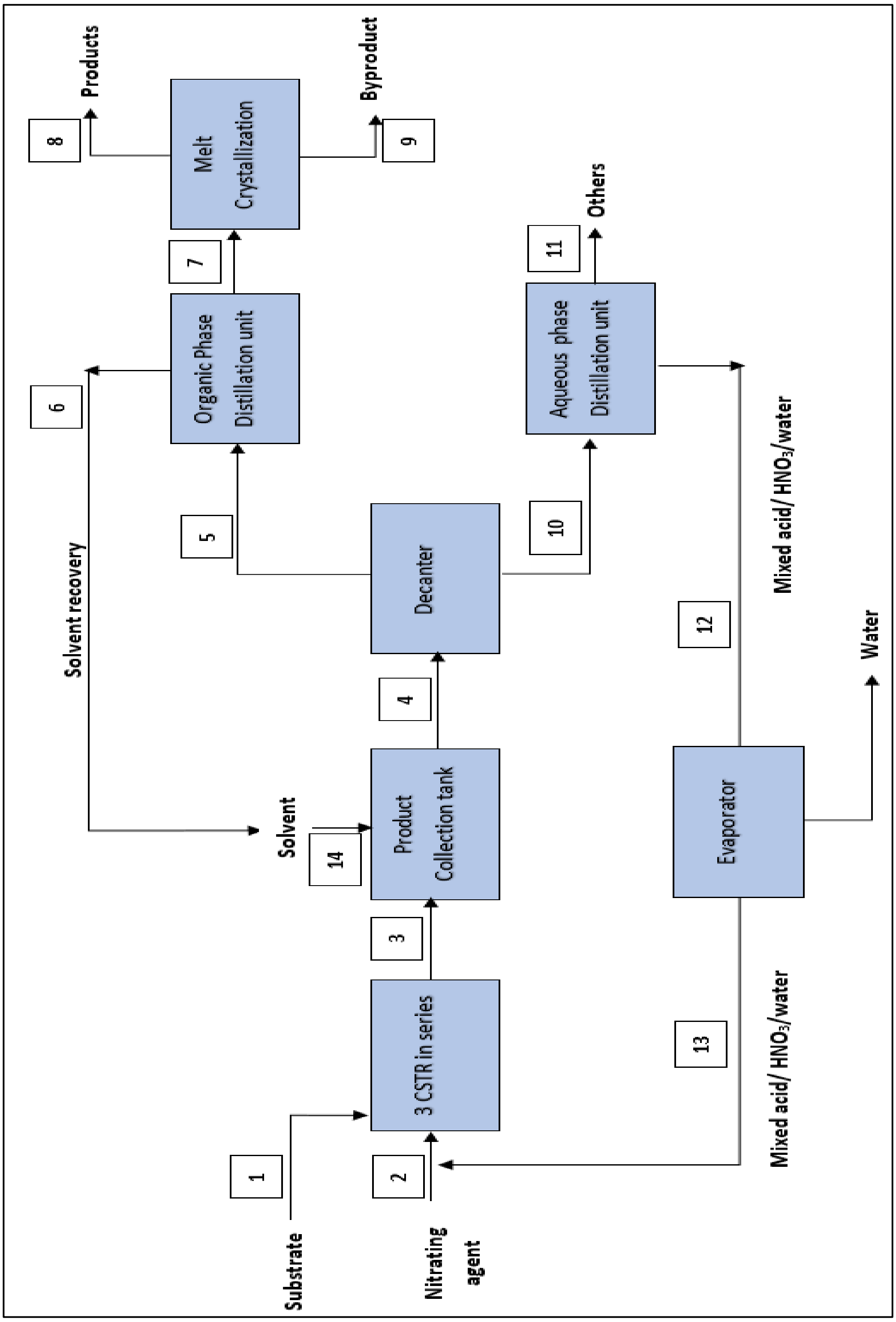


**Fig 1:** Block diagram of the proposed aromatic nitration process

## 3. Process Flow Diagram:

- **Proposed PFD for reaction section:**

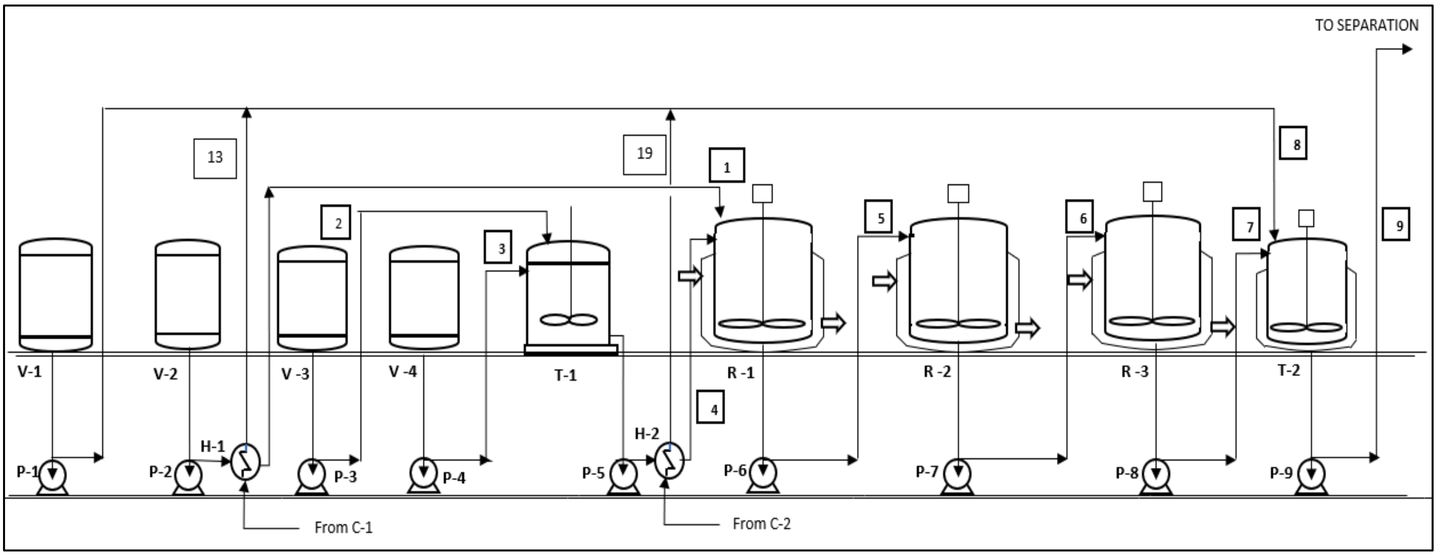


**Fig 2:** Proposed flow diagram for the reaction section

- **Proposed PFD for Separation section:**

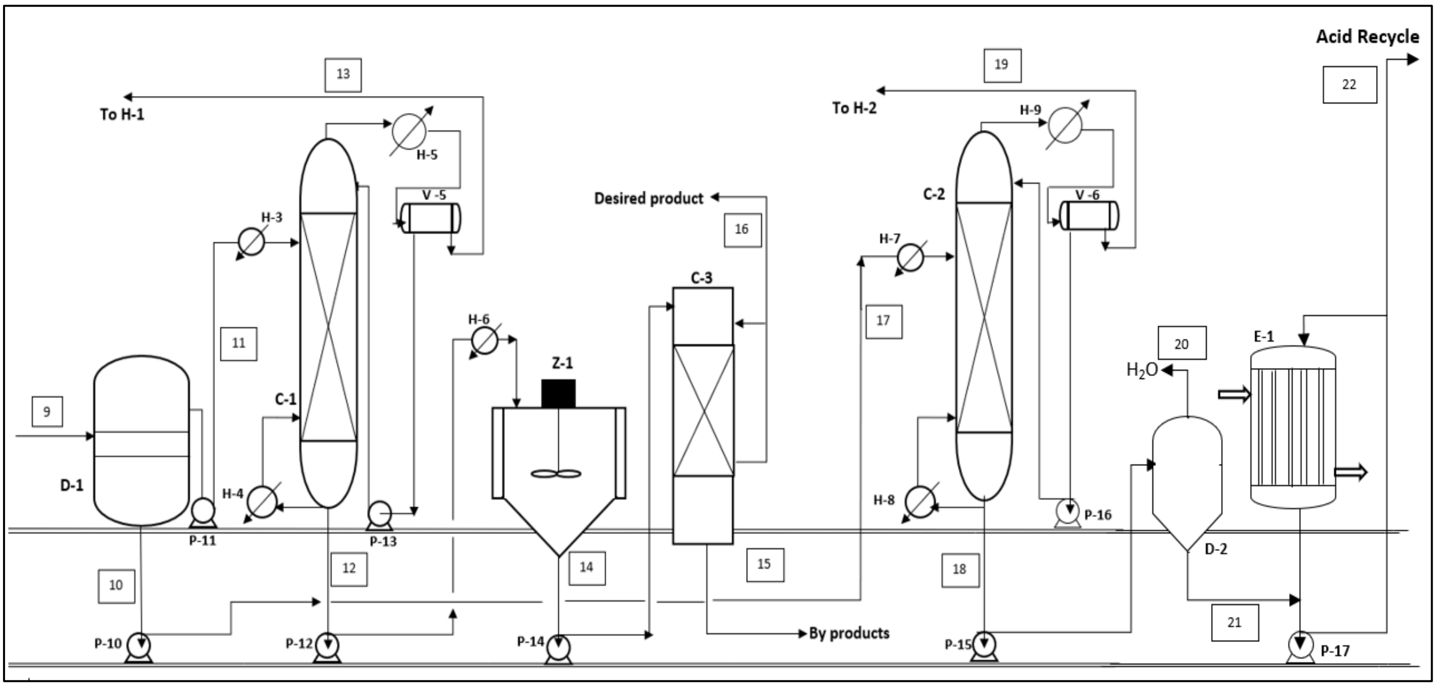


**Fig 3:** Proposed process flow diagram for the separation section

## Process description:

- **Reaction Section:**

The substrate is stored in the storage vessel V-2 which is preheated before feeding to the reactor (Stream 1) through P-2. Similarly, 69% $HNO_3$ and 98% $H_2SO_4$ are stored in vessels V-3 and V-4 respectively. In the mixer T-1, the acids are mixed in the required quantity, preheated, and fed to reactor R-1 (Stream 4). Three CSTRs are arranged in series with increasing volume. The substrate and nitrating agents react to produce mono-nitro products and byproducts. Being an exothermic reaction, a large amount of heat is generated in the reactor. Chilled water is passed through the jacket of each reactor to maintain the temperature inside each reactor. The outlet of the final CSTR is pumped to the tank where the product stream is quenched to avoid further nitration. The extraction solvent (Ethyl acetate) is pumped from a vessel (V-1) and mixed with the product stream collected in the quenching tank (T-1). After proper mixing, the mixture is passed to the separation section through stream 9 to the decanter.

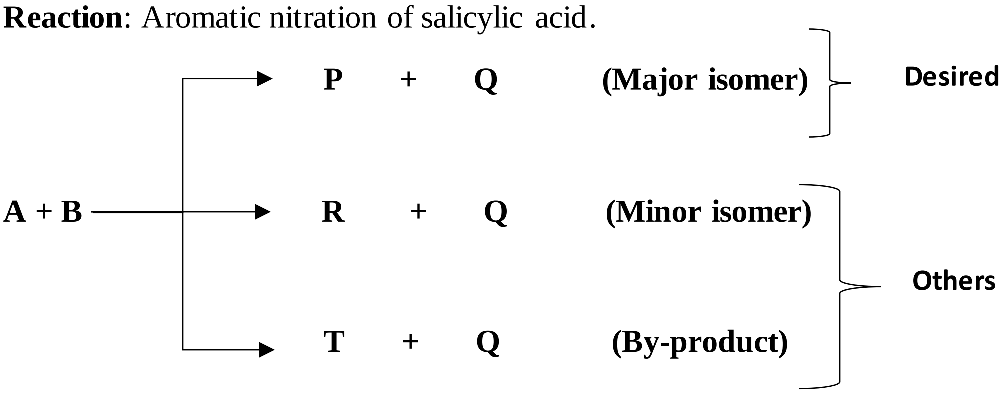
- **Separation section:**

Stream 9 is fed to the cylindrical decanter (D-1) where the separation of organic and aqueous phases takes place. The aqueous phase contains water, acids, and some amount of solvent is fed to the distillation column C-2, where the solvent is recycled back to the quenching tank (stream 19). The residue of C-2 mostly contains a mixture of acetic acid having a boiling point of 118°C which is used as a solvent (with substrate) in some cases and $H_2O/HNO_3$ azeotrope has a boiling point of 122°C. due to close boiling points, further separation becomes difficult. The bottom product of C-2 can be recycled back to the reactor. The excess water which will dilute the reaction mixture in the reactor is evaporated and the concentrated acid are recycled back.

The organic phase having low density is the top layer which is heated and fed to the fractionator (stream 11). EA recovered in the reflux drum (V-5) is recycled back to the reaction section, the quenching tank. Streams 13 and 19 are used to preheat the reactants. Since the residue from the fractionator (stream 12) has a close boiling point but different melting points, it is fed to the melt crystallizer where the crystallization takes place. The mother liquor containing the crystals of high melting isomer (stream 14) is passed through the column where the crystals of the desired product (in this case high melting isomer) form a bed and the mother liquor is collected from the bottom of the crystallizer. Over 99.99% separation can be achieved by refluxing the melt of desired product. The crystals are scrapped to get the pure desired product.

# 4. Material Balance:

**Reaction**: Aromatic nitration of salicylic acid.



**Basis: 1TPD of desired product is produced**

**Stoichiometry:**

| | | **Salicylic acid** | **Nitric acid** | $\longrightarrow$ | **5NSA** | **3NSA** | **water** |
|---|---|---|---|---|---|---|---|
| | | **A** | **B** | | **P** | **R, T** | **Q** |
| **Initial** | **kmol** | 3.000 | 3.000 | | 0.923 | 0.586 | 25.848 |
| | **kg** | 414.363 | 189.000 | | 84.483 | 53.623 | 465.258 |
| **1TPD** | **kg** | 5004.850 | 2282.821 | | 1020.417 | 647.676 | 5619.578 |
| | **lit** | 3468 | 1691 | | | | |

- **Results based on the MATLAB program [9]:**

All the parameters (physical, kinetic) are collected from literature [3,4,10-12]

Conversion= 100%

Yield= 61.16%

Mole ratio (5NSA/3NSA) = 1.58

- **Mole ratios:**

**HNO3/SA =** 5
**Acetic acid/SA =** 10
**Solvent/SA=** 5

- **Balance over reactor:**

| Component | kg/d |
|---|---|
| SA | 5004.85 |
| CH3COOH | 21759.27 |
| HNO3 (69%) | 11414.11 |
| EA | 0.00 |
| H2O | 5128.08 |
| 5NSA | 0.00 |
| 3NSA | 1.37 |
| **Total** | **43307.7** |

| Component | kg/d |
|---|---|
| SA | 0.00 |
| Acetic acid | 21759.27 |
| HNO3 | 9131.28 |
| EA | 0.00 |
| H2O | 10747.65 |
| 5NSA | 1020.42 |
| 3NSA | 649.05 |
| **Total** | **43307.67** |

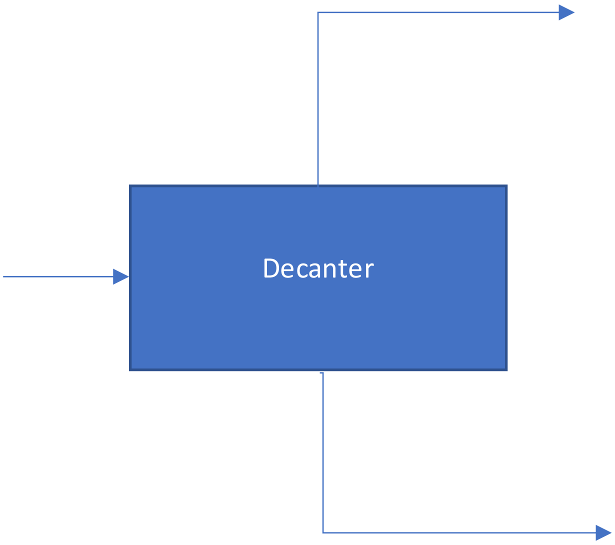
- **Balance over product collection tank:**

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 21759.27 |
| HNO3 | 9131.28 |
| EA | 15962.717 |
| H2O | 10747.65 |
| 5NSA | 1020.42 |
| 3NSA | 649.05 |
| **Total** | **59279.39** |

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 21759.27 |
| HNO3 | 9131.28 |
| EA | 15962.72 |
| H2O | 10747.65 |
| 5NSA | 1020.42 |
| 3NSA | 649.05 |
| **total** | **59270.39** |

- **Balance over decanter**

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 21759.27 |
| HNO3 | 9131.28 |
| EA | 15962.72 |
| H2O | 10747.65 |
| 5NSA | 1020.42 |
| 3NSA | 649.05 |
| **total** | **59270.39** |

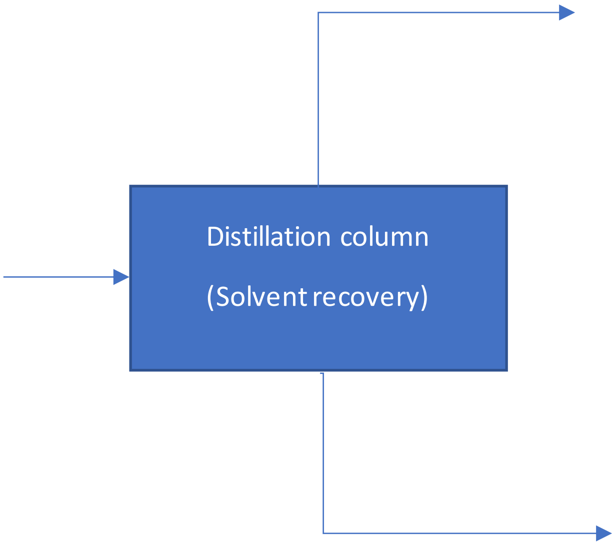


| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 15070.66 |
| H2O | 0.00 |
| 5NSA | 1020.41 |
| 3NSA | 647.65 |
| **total** | **16738.72** |

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 21759.27 |
| HNO3 | 9131.28 |
| EA | 892.06 |
| H2O | 10747.65 |
| 5NSA | 0.01 |
| 3NSA | 1.40 |
| **total** | **42531.67** |

- **Balance over distillation column for organic phase (solvent recycle):**

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 15070.66 |
| H2O | 0.00 |
| 5NSA | 1020.41 |
| 3NSA | 647.65 |
| **total** | **16738.72** |

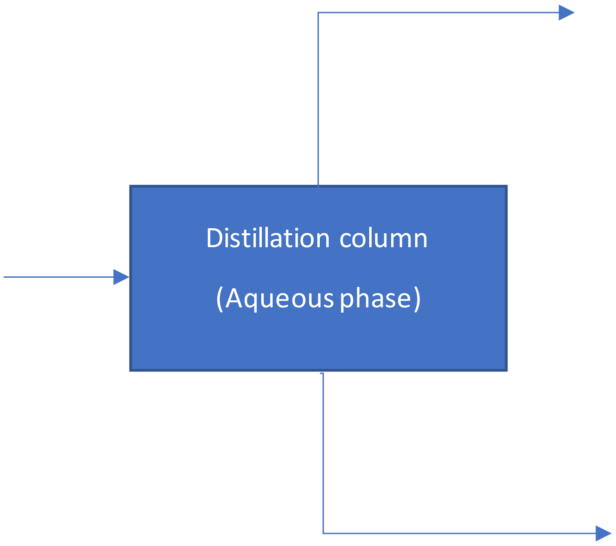


| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 15040.52 |
| H2O | 0.00 |
| 5NSA | 0.00 |
| 3NSA | 0.00 |
| **total** | **15040.52** |

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 30.14 |
| H2O | 0.00 |
| 5NSA | 1020.41 |
| 3NSA | 647.65 |
| **total** | **1698.20** |

- **Balance over distillation column for aqueous phase:**

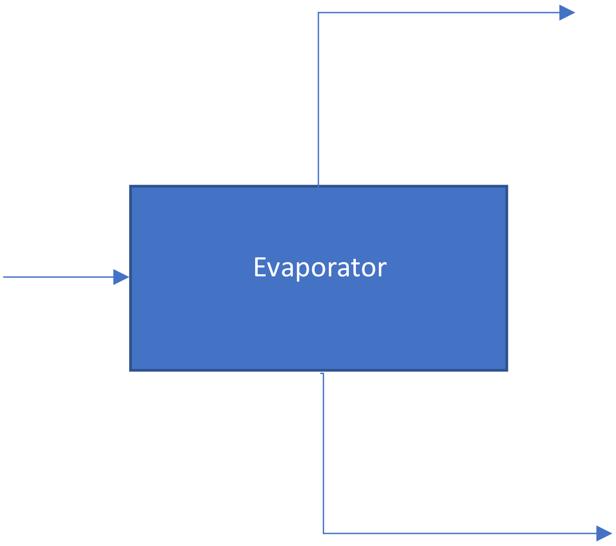
| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 21759.27 |
| HNO3 | 9131.28 |
| EA | 892.06 |
| H2O | 10747.65 |
| 5NSA | 0.01 |
| 3NSA | 1.40 |
| **total** | **42531.67** |



| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 892.06 |
| H2O | 0.00 |
| 5NSA | 0.00 |
| 3NSA | 0.00 |
| **total** | **892.06** |

| | |
|---|---|
| SA | 0.0 |
| Acetic acid | 21759.3 |
| HNO3 | 9131.3 |
| EA | 0.0 |
| H2O | 10747.7 |
| 5NSA | 0.0 |
| 3NSA | 1.4 |
| **total** | **41639.6** |

- **Balance over evaporator:**

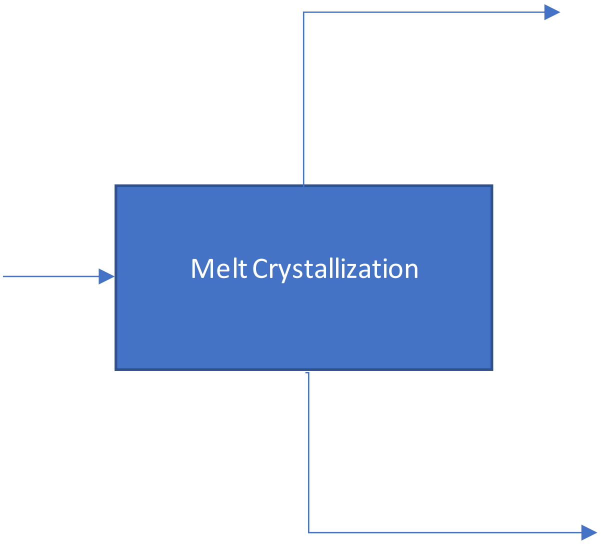


| | |
|---|---|
| SA | 0.0 |
| Acetic acid | 21759.3 |
| HNO3 | 9131.3 |
| EA | 0.0 |
| H2O | 10747.7 |
| 5NSA | 0.0 |
| 3NSA | 1.4 |
| **total** | **41639.6** |

| | |
|---|---|
| SA | 0.0 |
| Acetic acid | 435.2 |
| HNO3 | 182.6 |
| EA | 0.0 |
| H2O | 5619.6 |
| 5NSA | 0.0 |
| 3NSA | 0.0 |
| **total** | 6237.4 |

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 21324.09 |
| HNO3 | 8948.66 |
| EA | 0.00 |
| H2O | 5128.08 |
| 5NSA | 0.00 |
| 3NSA | 1.37 |
| **total** | **35402.19** |

- **Balance over melt crystallization**



| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 30.14 |
| H2O | 0.00 |
| 5NSA | 1020.41 |
| 3NSA | 647.65 |
| **total** | **1698.20** |

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 0.00 |
| H2O | 0.00 |
| 5NSA | 1000.00 |
| 3NSA | 0.00 |
| **total** | **1000.00** |

| | |
|---|---|
| SA | 0.00 |
| Acetic acid | 0.00 |
| HNO3 | 0.00 |
| EA | 30.14 |
| H2O | 0.00 |
| 5NSA | 20.00 |
| 3NSA | 647.65 |
| **total** | **697.79** |

**(All quantities are in kg/day)**

**For all balances mass in= mass out, hence verified.**

Similarly, for all five substrates calculations were performed. The quantities required for nitration of aromatic substrate after recycling of solvents and acids are mentioned below:

**Table 1:** Mass balance of all the substrates (**SA=**Salicylic acid**, AG=** Acetyl Guaiacol**, CB=** Chlorobenzene**, MB=** Methyl Benzoate**, TFMB=** Trifluoromethoxy Benzene)

| **Nitration of →** | **SA** | **AG** | **CB** | **MB** | **TFMB** |
|---|---|---|---|---|---|
| **Substrate** | 5005 | 3438 | 3683 | 4955 | 5785 |
| **$CH_3COOH$** | 435 | 249 | 0 | 0 | 0 |
| **$HNO_3$** | 2466 | 1329 | 2102 | 3164 | 2294 |
| **$H_2SO_4$** | 0 | 0 | 770 | 2853 | 700 |
| **Ethyl Acetate** | 30 | 17 | 28 | 28 | 30 |
| **Desired product** | 1000 | 1000 | 1000 | 1000 | 1000 |
| **By-products** | 698 | 117 | 251 | 93 | 908 |

**(All quantities are in kg/day)**

- **Volumes of CSTR in series for each substrate are mentioned below [13]:**

Sample calculation: to calculate volumes required for salicylic acid nitration

From stoichiometry:

Volumetric flowrates of salicylic acid = 3468 lit/day

Volumetric flowrates of nitric acid = 1691 lit/day

Total volumetric flowrate ($v_0$) = 5159 lit/day

**From MATLAB code:**

$\tau$ (1) = 2.66 min

$\tau$ (2) = 5.33 min

$\tau$ (3) = 8 min

Since, $V(i) = \tau(i).v_0$ … (1)

**Table 2:** Volume of CSTR in series for all substrates

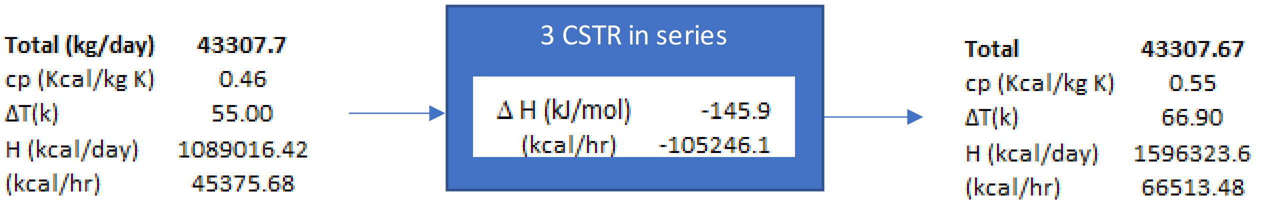
| **Substrate** | | Unit | SA | AG | CB | MB | TFMB |
|---|---|---|---|---|---|---|---|
| **Volume** | V1 | Lit | 14 | 16 | 18 | 18 | 15 |
| | V2 | | 28 | 32 | 36 | 35 | 30 |
| | V2 | | 41 | 47 | 54 | 53 | 46 |
| **Total Volume** | V | | 83 | 95 | 107 | 105 | 91 |
| **Production capacity** | | kg/day | 1000 | 1000 | 1000 | 250 | 1000 |
| **Volumetric flowrate** | v0 | lit/min | 5.16 | 3.39 | 4.46 | 1.32 | 5.70 |

**Since, volume of chlorobenzene is maximum it is used to size the CSTRs in series.**

## 5. Energy Balance:

Sample calculation for salicylic acid nitration:

- **Balance across reactor:**



| | |
|---|---|
| **Total (kg/day)** | **43307.7** |
| cp (Kcal/kg K) | 0.46 |
| ΔT(k) | 55.00 |
| H (kcal/day) | 1089016.42 |
| (kcal/hr) | 45375.68 |

| | |
|---|---|
| Δ H (kJ/mol) | -145.9 |
| (kcal/hr) | -105246.1 |

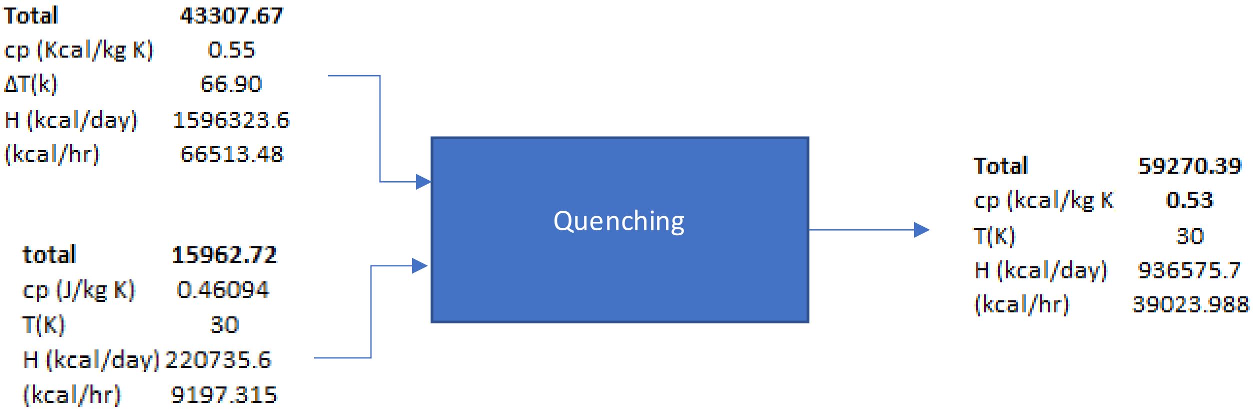
| | |
|---|---|
| **Total** | **43307.67** |
| cp (Kcal/kg K) | 0.55 |
| ΔT(k) | 66.90 |
| H (kcal/day) | 1596323.6 |
| (kcal/hr) | 66513.48 |

Therefore, to be heat removed **Q= 84108.3 kcal/hr**

| | |
|---|---|
| Q (kcal/hr) | **84108.3** |
| Cp(kcal/kg k) | 1 |
| ΔT(k) | 5 |
| m (kg/hr) | 16821.67 |

**Quantity of cooling water required = 16.83m$^3$/hr**

- **Balance across product collection tank:**



| | |
|---|---|
| **Total** | **43307.67** |
| cp (Kcal/kg K) | 0.55 |
| ΔT(k) | 66.90 |
| H (kcal/day) | 1596323.6 |
| (kcal/hr) | 66513.48 |

| | |
|---|---|
| **total** | **15962.72** |
| cp (J/kg K) | 0.46094 |
| T(K) | 30 |
| H (kcal/day) | 220735.6 |
| (kcal/hr) | 9197.315 |

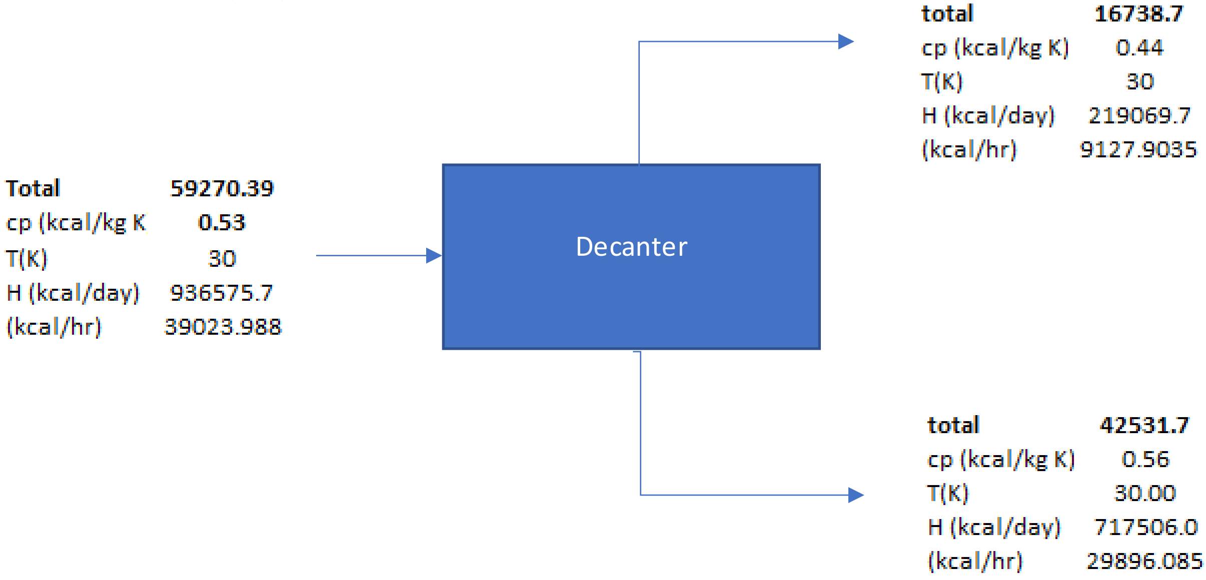
| | |
|---|---|
| **Total** | **59270.39** |
| cp (kcal/kg K | **0.53** |
| T(K) | 30 |
| H (kcal/day) | 936575.7 |
| (kcal/hr) | 39023.988 |

Therefore, to be heat removed **Q= 36686.81 kcal/hr**

| | |
|---|---|
| Q1 (kcal/hr) | 36686.81 |
| Cp (kcal/kg k | 1 |
| ΔT(k) | 5 |
| m (kg/hr) | 7337.362 |

**Quantity of cooling water required to remove Q1 heat= 7.34 m$^3$/hr**

- **Balance across decanter**



- **Balance across distillation column (solvent recovery):**

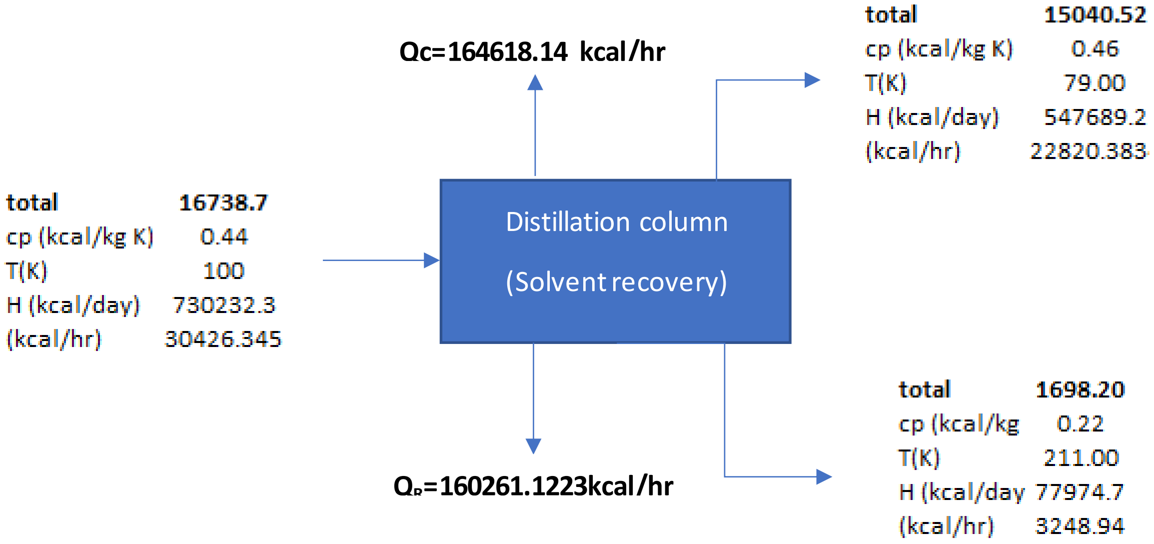


**Table 3:** Column C-1 specifications (Condenser and reboiler duty)

| | | |
|---|---|---|
| Feed (F) | kg/day | 42531.67 |
| Reflux ratio (R) | | 2 |
| Distillate (D) | kg/day | 15040.52 |
| Reflux stream (L) | kg/day | 30081.04 |
| vapor flowrate (V) | kg/day | 45121.56 |
| **Component** | **Latent heat** | **Quantity** |
| | **(kJ/kg)** | **(kg)** |
| **EA** | 366 | 15040.52 |
| **Condenser duty** | **16514491.81** | **(kJ/day)** |
| | **164618.1401** | **(kcal/hr)** |
| Bottom product | **W (kg/hr)** | 27491.15 |
| **Reboiler duty** | **160261.1223** | **(kcal/hr)** |

- **Balance across distillation column (aqueous phase):**

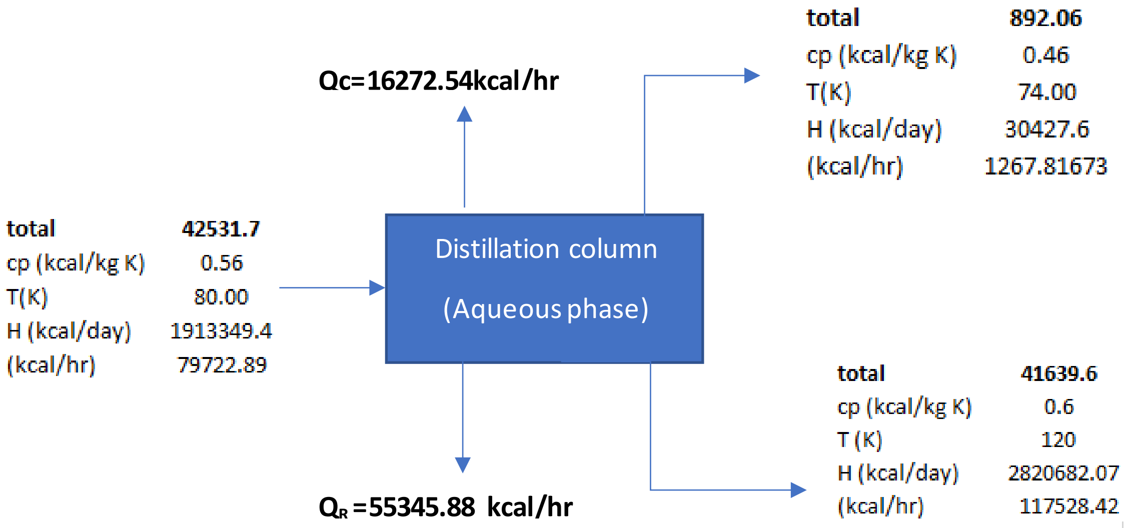
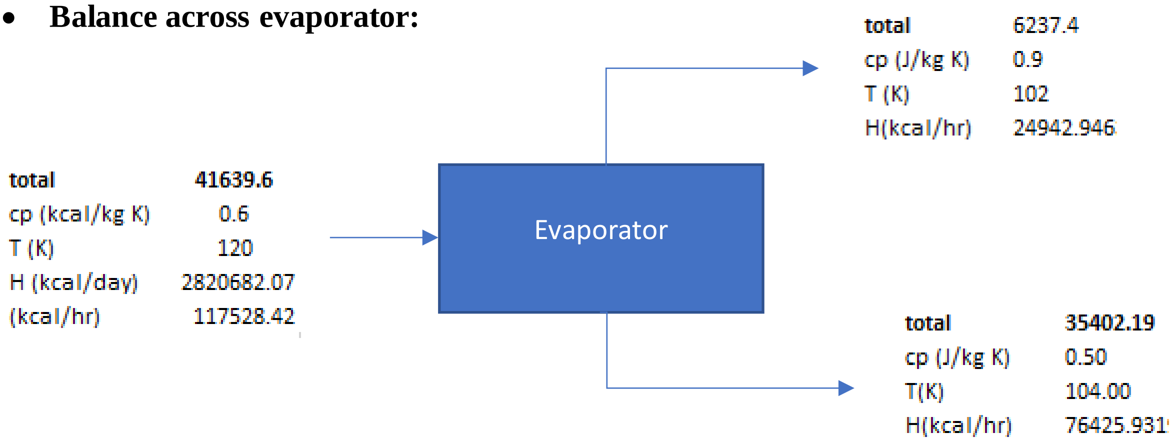


**Table 4:** Column C-2 specifications (Condenser and reboiler duty)

| | | |
|---|---|---|
| Feed (F) | kg/day | 42531.67 |
| Reflux ratio (R) | | 4 |
| Distillate (D) | kg/day | 892.06 |
| Reflux stream (L) | kg/day | 3568.22 |
| vapor flowrate (V) | kg/day | 4460.28 |
| **Component** | **Latent heat** | **Quantity** |
| | **(kJ/kg)** | **(kg)** |
| **EA** | 366 | 15040.52 |
| **Condenser duty** | **1632461.239** | **(kJ/day)** |
| | **16272.54026** | **(kcal/hr)** |
| **Bottom product** | **W (kg/hr)** | 41639.62 |
| **Reboiler duty** | **55345.88423** | **(kcal/hr)** |

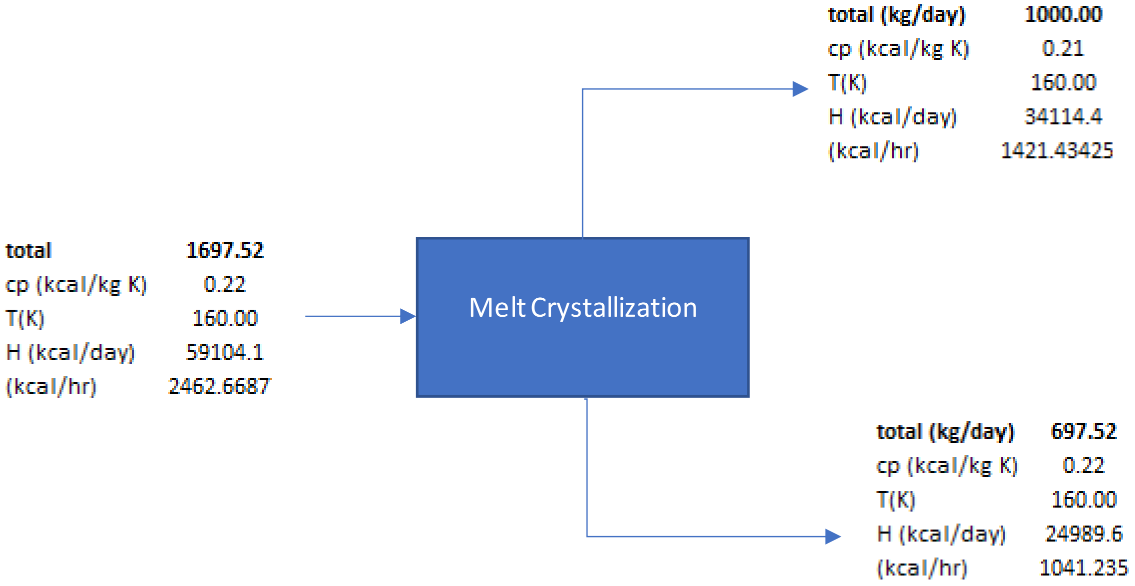
- **Balance across evaporator:**



Q= 16159.5 kcal/hr

Flowrate of steam = $m_s$ = 25.25kg/hr

- **Balance across Melt crystallization:**



- **Quantity of utility required**

**Table 5**: Quantity of utility required to run salicylic acid nitration plant

| | **Energy required** | **Utility required** | |
|---|---|---|---|
| | **Q (kcal/hr)** | **m (kg/hr)** | |
| Reboiler 1 (H-4) | 160261.1223 | 296.78 | Steam |
| Reboiler 2 (H-8) | 55345.88423 | 102.5 | |
| Heater 3 (H-3) * | 21960.78 | 40.67 | |
| Heater 6 (H-6) * | 1414 | 2.62 | |
| Heater 7 (H-7) * | 69729.63 | 129.13 | |
| Condenser 1 (H-5) | 164618.1401 | 12662.9 | Cooling water |
| Condenser 2 (H-9) | 16272.54026 | 1251.73 | |
| Reactor (R- 1,2,3) | 84108.3 | 16821.67 | |

*Calculations are shown later

**Total quantity of steam required =544.7 kg/hr**

**Total quantity of cooling water supplied = 30735.3**

## 6. Equipment sizing:

### 6.1 Reactor design:

**Calculations:**

Since volume of chlorobenzene is highest it is used for designing the CSTRs installed in the plant.

Volume of CSTR 1 =0.18m$^3$

from calculation

Considering volume of reacting fluid is 80% of total volume.

**Therefore, volume of CSTR 1= 0.22 m$^3$**

- **Mechanical Design [14]:**

  Assuming L/D ratio= 2.5

  Thus, L= 2.5 D

  D= 0.2248 m =225 mm

  L= 0.5622 m = 562 mm

**Vessel thickness based on internal pressure:**

Material: SS316L

Ultimate Tensile stress= 515 MPa

Minimum yield stress =205 MPa

Allowable stress= 137.89 MPa

Internal pressure = 0.101 N/mm$^2$

Internal design pressure (10% greater) = 0.1111N/mm$^2$

$$t = \frac{(P*Di)}{(2*f*J-P)} \qquad \text{…….(2)}$$

where, t= thickness of reactor (mm)

P= internal design pressure (N/mm$^2$)

Di= Internal diameter of reactor (mm)

f= Allowable stress (N/mm$^2$)

J = Joint efficiency

**Therefore, t= 1.5 mm (including corrosion allowance)**

**Vessel thickness based on external pressure:**

External pressure = 0.101 N/mm$^2$

Design external pressure (10% greater) = 0.1111 N/mm$^2$

Critical buckling pressure:

$$P_c = \frac{2.42\ E\ \left(\frac{t}{D_O}\right)^{5/2}}{(1-\mu^2)^{3/4}\left[\left(\frac{L}{D_O}\right)-0.45\left(\frac{t}{D_O}\right)^{1/2}\right]} \qquad \text{……..(3)}$$

Thickness (t) = 1.5 mm

Modulus of elasticity (E) = 193000 N/mm$^2$

Poisson's Ratio (μ) =0.27

Outer Diameter ($D_o$) = ($D_i$+ 2*t) = 228 mm

Length of reactor (L)= 562 mm

Thus, $P_c$ = 0.715 N/mm$^2$

Allowable Pressure= ($P_c$/4) = 0.18 N/mm$^2$

The value of allowable stress is greater than design pressure (0.1111 N/mm$^2$). Hence, proposed thickness (t=1.5mm) is acceptable.

- Jacket Thickness:

$$t_j = \frac{(P_{ext} * D_i)}{(2*f*J - P_{ext})} \qquad \text{…..(4)}$$

$t_j$= thickness of jacket (mm)

$P_{ext}$ = External design pressure (N/mm$^2$)

Therefore, $\underline{t_j = 2\ \text{mm}}$ (Including corrosion allowance)

- Check for loadings:

**1) Tangential or hoop stress**

$$f_t = \frac{P_i\ (D_i+t)}{2t} \qquad \text{…...(5)}$$

**= 7.62 N/mm$^2$**

**2) Axial Stress due to internal pressure**

$$f_1 = \frac{(P_i * D_i)}{4t} \quad \ldots\ldots(6)$$

**$= 3.79\ N/mm^2$**

**3) Stresses due to weight of the vessel**

**a) Volume of cylindrical section = 0.22 $m^3$**

**b) Volume of dished ends**

$V = 0.081\ D_i^3$

$= 0.000921\ m^3$

Straight portion of head = 40mm

Thus, volume = (3.142/4) *(Di)*0.04 =0.00159 $m^3$

Volume of 2 heads = 2* (0.00092+ 0.00159)

= 0.00501 $m^3$

Total volume of vessel= 0.027$m^3$= 0.03 $m^3$

Weight of vessel with water= 0.03*1000= 30kg

Weight of empty vessel = (3.142) *Di*H* t*ϱ

= 4.754kg

**Weight of dished ends**

Assuming

Crown radius = Internal diameter = 225 mm

Knuckle radius ($R_k$)= 6% of internal diameter = 13.5 mm

Blank Diameter (B)= $1.024D_o + 0.67(R_k) + 2\ S_f$

= 0.3224 m

Weight of dish head= (3.142/4) * $(0.3224)^2$*(1.5/1000) * 7980

= 1kg

Weight of heads= 2*1 =2kg

**Total weight of empty vessel = 4.75 +2 = 6.75 kg**

= 10 kg (10-20% more for nozzles, manholes)

Total weight= 10 + 30= 40kg

$f_2 = W/ (3.142) *(t\ (t+ D_i))$

$= 0.37\ N/mm^2$

$f_a = f_1 + f_2 + f_3$

$= 3.414\ N/mm^2$

(here, $f_3$= wind load)

4) Stress due to offset piping

Assuming torque= 500000 N/mm)

fs = 2T/ (3.142 t Di (Di+t))

$= 4.17\ N/mm^2$

5) Combining all preceding stresses based on strain energy theory

$f_R = [f_t^2 - f_t*f_a + f_a^2 + 3f_s^2]^{0.5}$

$= 9.79\ N/mm^2$

Thus, all above stresses are below allowable stress limit. Hence, thickness of 1.5mm is safe.

**Head Thickness**

Assuming straight portion of dished heads to be 40mm

Top dished end (Internal pressure only)

Stress intensification factor

$W = (1/4)\ [3 + (R_c/R_k)^{0.5}]$ …… (7)

Assuming,

Crown Radius= Internal Diameter= 225mm

Knuckle Radius= 6% Internal Diameter = 13.5mm

Therefore, W= 1.771

$t_h = (P_i\ R_c\ W)/\ (2\ f\ J)$

= 0.17mm

Say 1mm

**Bottom dished end**

Following thumb rule Design pressure for bottom head = 1.67 $P_i$

$= 0.169\ N/mm^2$

$$t_h = \left(\frac{P_d\ R_c\ W}{2\ f\ J}\right) \quad \text{……..(8)}$$

= 0.189 mm

Say $t_h$ = 1 mm

Similarly, calculations were performed for CSTR 2 and CSTR 3 which are mentioned in the table:

**Table 6:** Specifications of CSTR in series

| **Parameter** | **Unit** | **CSTR 1** | **CSTR 2** | **CSTR 3** |
|---|---|---|---|---|
| **MOC** | | **SS316L** | **SS316L** | **SS316L** |
| **Volume** | lit | 18 | 36 | 54 |
| **Internal diameter of reactor** | mm | 225 | 284 | 324 |
| **Height of reactor** | mm | 563 | 708 | 811 |
| **Thickness of reactor** | mm | 1.5 | 1.5 | 1.5 |
| **Jacket of reactor** | mm | 2 | 3 | 3 |
| **Head thickness** | mm | 1 | 1.5 | 1.5 |

## 6.2 Decanter Design [15]:

Since, mass flowrate entering the decanter is highest for chlorobenzene nitration, it is sized based on the flowrates of chlorobenzene nitration.

**Properties of light and heavy phase [16]**:

| | | | | density | solution density | viscosity | solution viscosity |
|---|---|---|---|---|---|---|---|
| | | kg/day | wt/wt | g/m3 | kg/m3 | Pa s | kg/m s |
| light phase (organic) | CB | 0.000 | 0.0000 | 1.1100 | 950.27 | 0.0077 | 0.0029 |
| | EA | 13807.32 | 0.9186 | 0.9020 | | 0.0030 | |
| | 4NCB | 1020.40 | 0.0679 | 1.5200 | | 0.0011 | |
| | 2NCB | 203.7 | 0.0136 | 1.3680 | | 0.0021 | |
| | Total | 15031.43 | | | | | |
| heavy phase (aqueous) | H2SO4 | 76951.61 | 0.86 | 1.83 | 1733.37 | 0.0267 | 0.0232 |
| | HNO3 (69%) | 4122.41 | 0.05 | 1.35 | | 0.0012 | |
| | EA | 605.73 | 0.01 | 0.90 | | 0.003 | |
| | H2O | 7297.93 | 0.08 | 1 | | 0.001 | |
| | Total | 88977.68 | | | | | |

**Table 7:** Properties of the components in the decanter

**Assumption:**

Droplet Diameter= 150 µm

- Settling velocity:

$$u_d = \frac{d_d^2\, g\, (\rho_d - \rho_c)}{18\, \mu_c} \qquad \text{…..(9)}$$

Where, $d_d$ = Droplet diameter (m)

$u_d$= settling velocity of the dispersed phase droplet with diameter (m/s)

$\varrho_c$ = density of the continuous phase (kg/$m^3$)

$\varrho_d$ = density of the dispersed phase (kg/$m^3$)

$\mu_c$=viscosity of the continuous phase (N s/ $m^2$)

g = gravitational acceleration (m/$s^2$)

Therefore, $u_d$ = -0.000413 m/s

Negative sign indicates that drops are rising.

As the flowrate is small, use a vertical cylindrical decanter.

Volumetric flowrates of continuous phase

$L_c$ = 88977.68/ (1733.37* 24* 60*60)

= 0.000594 $m^3$/s

$u_c$= $L_c/A_i$

Hence, $A_i$ = 0.00059/0.000413

= 1.438$m^2$

$$r = \sqrt{\frac{1.438}{\pi}} = 0.68$$

Diameter = 1.35 m

Assumption, H/D= 2

Therefore, height of decanter = 2.71 m

Take dispersion band as 10 percent of the height =0.24 m

Residence time of the droplet in the dispersion band = 0.24/ $u_d$

= 655.5 sec = 11 min (approx.)

Velocity of light phase = (15031.43/950.27) *(1/3600) *(1/1.44) *(1/24)

= 0.000127 m/s

Using equation (9),

Diameter ($d_d$) = 83.27 µm

Since diameter of light phase is less than heavy phase, the results obtained are satisfactory.

**Piping arrangement**

To minimise entrainment by the jet of liquid entering the vessel, the inlet velocity should be less than 1m/s.

$$\text{Flowrate} = \left[\frac{88977.68}{1733.37} + \frac{15031.43}{950.2679}\right]\frac{1}{3600}$$

$= 0\ .00078\ m^3/s$

Area of pipe = 0.00078 $m^2$

Pipe diameter = 0.99mm

Say d= 1mm

The position of the interface is assumed to be half way in the decanter and liquid-liquid offtake is at 90% of the total height of decanter.

Z1 =0.9 * 2.71 = 2.44 m

Z2 = 0.5 * 2.71 = 1.351m

Z3 = (2.44-1.351) * (900/1000) +1.35 = 1.95m

Say 2m

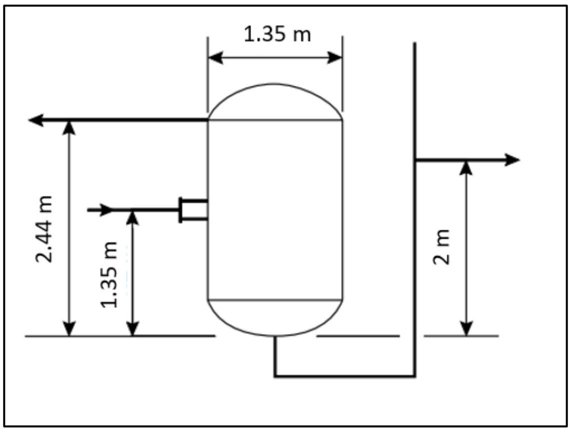


**Fig 4:** Proposed design of decanter

### 6.3 Sizing of storage vessels and tanks:

The largest flowrate was selected for each vessel and based on those values calculations are performed.

For all calculations,

Optimum H/D ratio= 3

Internal pressure = 0.101 MPa

Design pressure (10% greater) =0.1111 MPa

- Sample calculations for substrate storage vessel:

Substrate flowrate= 5785.4 kg/day

Density of substrate =1300 kg/m$^3$

Holding time = 1day

MOC= SS316

Permissible stress= 137.89 MPa

Volume of vessel = (5785.4 *1)/1300

= 4.45m$^3$

Hence, diameter of vessel = 1.24 m

= 1236.32mm

Height= 1.24*3= 3.71m

=3708.98 mm

Using, t = (P* D)/ (2 f J)

Therefore, t= 0.586mm

Thickness with corrosion allowance =2.1mm

Similarly, all calculations are performed accordingly and are mentioned below:

**Table 8:** Specification of the storage vessels

| Sr no. | Description | code | MOC | Flowrate kg/d | density kg/m$^3$ | Holding time day | Volume m3 | diameter mm | Height mm | Thickness mm |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Solvent storage vessel | V-1 | SS316 | 15722.7 | 900 | 1 | 17 | 1950 | 5341 | 3 |
| 2 | Substrate storage vessel | V-2 | SS316 | 5785.4 | 1300 | 1 | 4 | 1236 | 3709 | 2 |
| 3 | HNO3 Storage vessel | V-3 | SS316L | 34976.5 | 1353 | 1 | 26 | 2222 | 4636 | 3 |
| 4 | H2SO4 Storage vessel | V-4 | SS316L | 76951.6 | 1830 | 1 | 42 | 2614 | 6674 | 2 |
| 5 | Acid mixing tank | T-1 | SS316L | 3464.0 | 1806.03 | 0.02 | 1 | 741 | 1797 | 2 |
| 6 | Product collection tank | T-2 | SS316L | 4333.7 | 1500 | 0.04 | 3 | 1071 | 1982 | 2 |

## 6.4 Pumps

Pumps are used to transfer the material from one vessel. Centrifugal pumps are most used for industrial application [15]. Selection of pumps is based on the nature of material, the head, and the flowrates. Metering pumps can be used to achieve accurate flowrates.

In this study, preliminary design is shown. Overall losses are assumed as 10%, pump efficiency as 60% and motor efficiency as 80 %.

Sample calculations:

$$\text{Power (BKW)} = \frac{Q\ h}{36.71\ \epsilon} \qquad \text{……(10)}$$

Q= Flowrate in m3/hr

h= head in MWC

ϵ = pump efficiency

$$\text{Motor rating} = \frac{BKW}{e\ \alpha} \qquad \text{…....(11)}$$

e= losses due to friction

α= Motor efficiency

**Table 9:** Specifications of centrifugal pumps

| Sr No. | Description | code | Capacity | | Power | Motor rating |
|---|---|---|---|---|---|---|
| | | | Q(m3/hr) | Head (MWC) | BKW | kW |
| 1 | Ethyl Acetate Transfer | P-1 | 0.737 | 8 | 0.27 | 0.37 |
| 2 | Substrate transfer | P-2 | 0.892 | 8 | 0.32 | 0.45 |
| 3 | Nitric acid Transfer | P-3 | 0.352 | 8 | 0.13 | 0.18 |
| 4 | H2SO4 Transfer | P-4 | 1.752 | 10 | 0.80 | 1.10 |
| 5 | Mixed acid Transfer | P-5 | 1.924 | 10 | 0.87 | 1.21 |
| 6 | Product stream from CSTR 1 | P-6 | 2.196 | 10 | 1.00 | 1.38 |
| 7 | Product stream from CSTR 2 | P-7 | 2.196 | 10 | 1.00 | 1.38 |
| 8 | Product stream from CSTR 3 | P-8 | 2.196 | 10 | 1.00 | 1.38 |
| 9 | Transfer from quenching unit | P-9 | 2.549 | 8 | 0.93 | 1.29 |
| 10 | Aqueous phase transfer | P-10 | 2.557 | 12 | 1.39 | 1.93 |
| 11 | Organic phase transfer | P-11 | 0.472 | 10 | 0.21 | 0.30 |
| 12 | Residue transfer from C-1 | P-12 | 0.054 | 8 | 0.02 | 0.03 |
| 13 | Reflux from C-1 | P-13 | 0.016 | 12 | 0.01 | 0.40 |
| 14 | Transfer from crystallizer | P-14 | 0.054 | 10 | 0.02 | 0.03 |
| 15 | Residue transfer from C-3 | P-15 | 2.046 | 8 | 0.74 | 1.03 |
| 16 | Reflux from C-2 | P-16 | 0.106 | 12 | 0.06 | 0.08 |
| 17 | Transfer from evaporator (recycle) | P-17 | 1.901 | 10 | 0.86 | 1.20 |
| | | | | | Total | 13.74 |

## 6.5 Distillation column [15]:

In distillation column, separation of two or more components is based on differences in their relative volatilities in the mixture. Both the organic and aqueous phases separated from the decanter are fed to the distillation columns C-1 and C-2. As the design of the column is based on the vapor volumetric flowrate, the distillation column is designed based on the substrate which has maximum vapor volumetric flowrate.

**Function:** To remove Extraction solvent (EA) from both aqueous and organic phase.

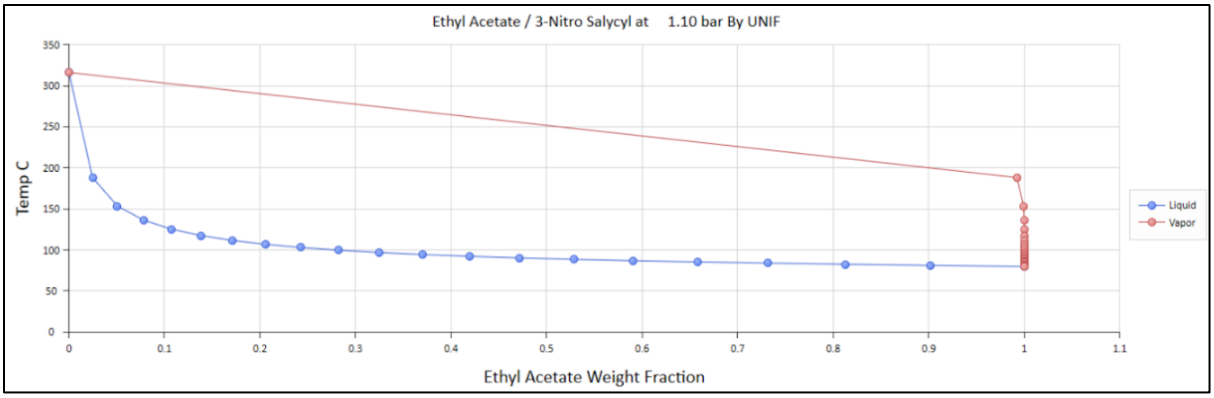


**Fig 5:** T-x-y data for ethyl acetate / 3- Nitro salicylic acid system

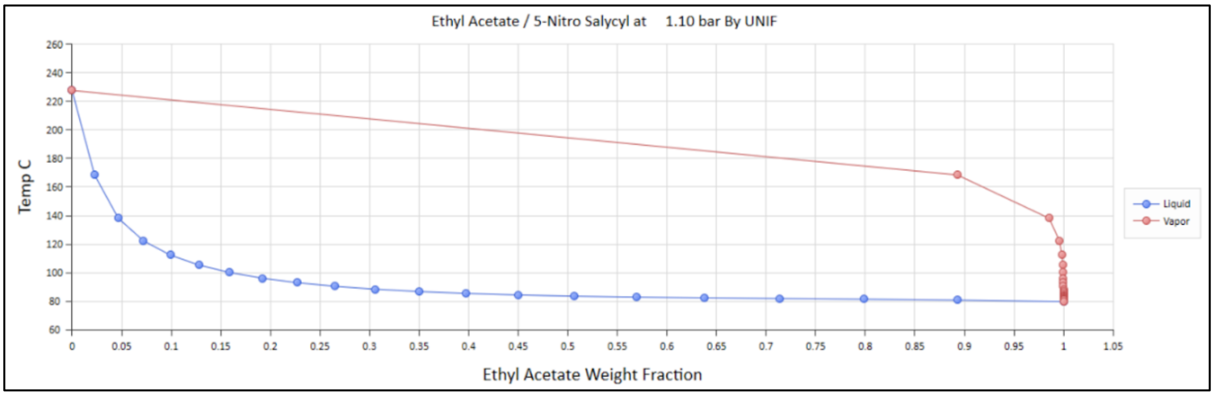


**Fig 6:** T-x-y diagram for ethyl acetate / 5 nitro salicylic acid system

Design calculation of distillation column C-1

**Top section:**

Liquid mass flowrate = 1256 kg/hr

Vapor mass flowrate = 1884 kg/hr

Density of liquid $\varrho_L$ = 825 kg/m$^3$

Density of vapor $\varrho_V$ = 3.05kg/m$^3$

Therefore, $F_{LV\ top} = (1256/1884) * (3.05/825)^{0.5}$

$= 0.041$

**Bottom section:**

Liquid mass flowrate = 1827 kg/hr

Vapor mass flowrate = 938 kg/hr

Density of liquid $\varrho_L$= 1277 kg/m$^3$

Density of vapor $\varrho_V$ = 4.54 kg/m$^3$

Therefore, $F_{LV\ bottom}$ = (1827/938) * (4.54/1277)$^{0.5}$

= 0.1161

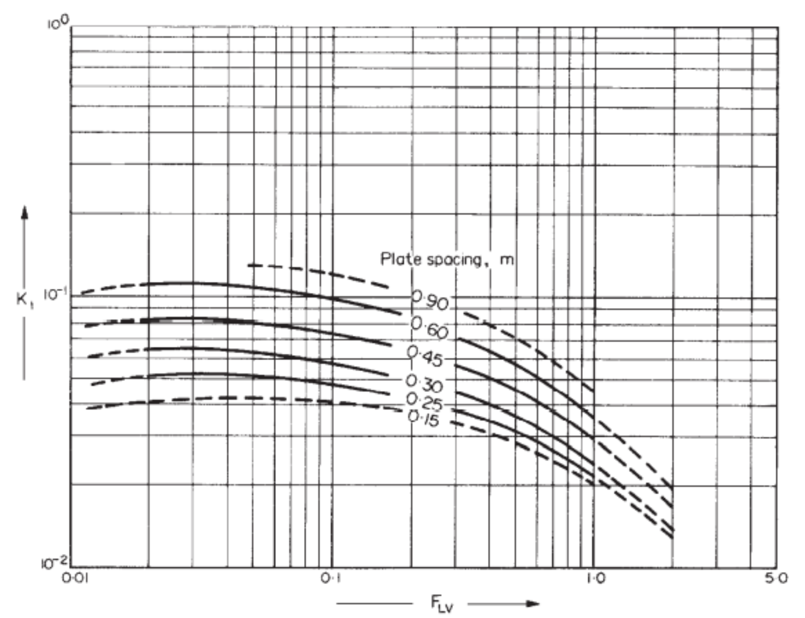


**Fig 7**: $F_{LV}$ vs K1 based on plate spacing

Take plate spacing as 0.5m

Base $K_1$= 7.5 *10$^{-2}$

top $K_1$= 9.0*10$^{-2}$

To calculate flooding velocity:

$$u_f = K1\sqrt{\frac{\rho_L - \rho_V}{\rho_V}}$$

where, uf= flooding velocity (m/s)

$\varrho_L$= density of liquid (kg/m$^3$)

$\varrho_V$= density of vapor (kg/m$^3$)

$u_{f\ Bottom}$= 1.231m/s

$u_{f\ Top}$= 1.507 m/s

Design for 85% flooding at maximum flowrate

Base $u_v$ = 1.047 m/s

Top $u_v$ = 1.28 m/s

**Maximum volumetric flow-rate**

Base = (1827)/ (3600*4.54) = 0.111 $m^3/s$

Top = (1884)/ (3600*3.05) = 0.172 $m^3/s$

**Net area required**

Bottom = 0.111/ 1.047 = 0.106 $m^2$

Top = 0.172/ 1.28 = 0.134 $m^2$

Assuming, downcomer area as 12% of total

Therefore, column cross sectional area

Base =0.106//0.88 = 0.12 $m^2$

Top = 0.134/0.88 = 0.152 $m^2$

**Column diameter**

Base = $\sqrt{\frac{0.12*4}{\pi}} = 0.39$m

Top = $\sqrt{\frac{0.152*4}{\pi}} = 0.44$m

From CHEMCAD,

Reflux ratio required =2

No. of plates = 8

Feed on 4th plate

Since plate spacing = 0.5m

Height of column = (0.5 * 8) +0.5 = 4.5 m

Condenser duty ($Q_c$) = 163880 kcal/hr

Reboiler duty ($Q_R$) = 160261.12 kcal/hr

Similar calculations were performed for distillation column C-2

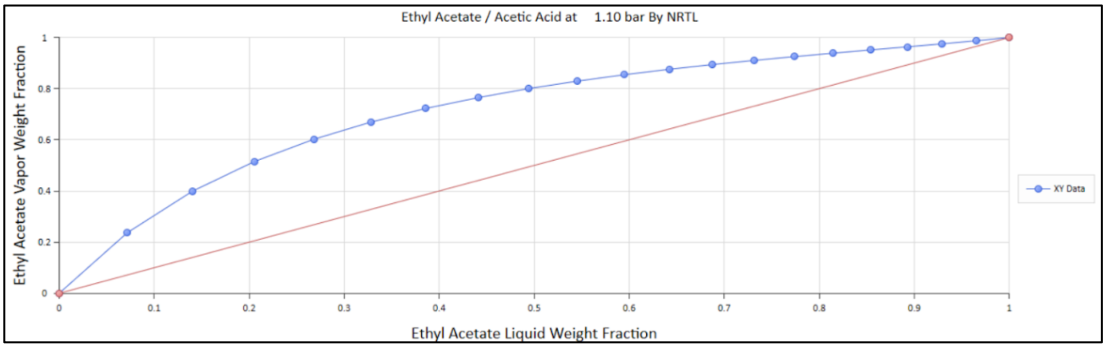


**Fig 8:** x-y data for ethyl acetate - acetic acid system

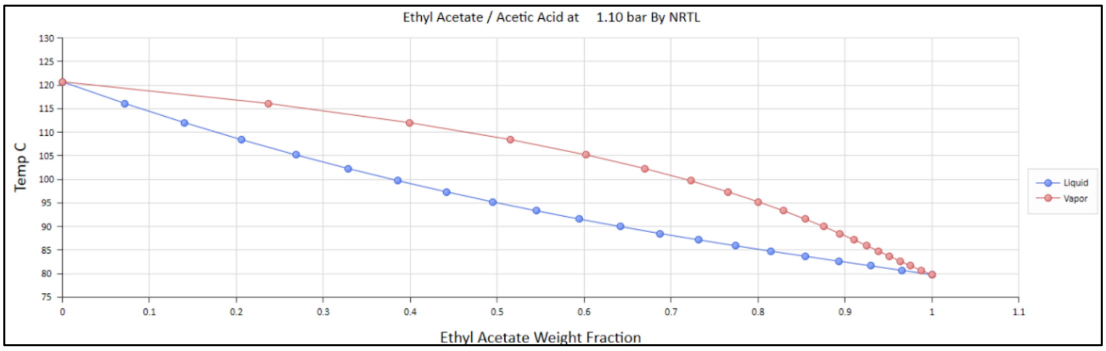


**Fig 9:** T-x-y diagram for ethyl acetate – acetic acid system

**Table 10:** Calculations for calculating column diameter (C-2)

| | **unit** | **Top** | **Bottom** |
|---|---|---|---|
| Liquid mass flowrate | kg/hr | 162 | 1869 |
| Vapor mass flowrate | kg/hr | 202 | 135 |
| Density of liquid | kg/m$^3$ | 843 | 1087 |
| Density of vapor | kg/m$^3$ | 2.59 | 1.24 |
| Flooding velocity | m/s | 1.15 | 2.26 |
| Maximum volumetric flowrate | m3/s | 0.017 | 0.41 |
| Cross sectional area | m$^2$ | 0.017 | 0.206 |
| Diameter of column | m | 0.147 | 0.512 |

From CHEMCAD,

Reflux ratio required =4

No. of plates = 30 (Feed on 10th plate)

Since plate spacing = 0.5m

Height of column = (0.5 * 30) +0.5 = 15.5 m

Condenser duty ($Q_c$) = 16272.54 kcal/hr

Reboiler duty ($Q_R$) = 55345.88 kcal/hr

## 6.6 Falling film evaporator:

Assuming $\Delta T = 10K$ for which proper wetting is achieved

From mass balance:

Total mass to be evaporated = 260kg/hr

Hence, heat load = 260*540 = 140400kcal/hr

**To calculate circulation rate:**

$140400 = m\ C_p\ \Delta T$

Therefore, m=280800 kg/hr = 2808 $m^3$/hr

Steam is at 120°C

$$\Delta Tlm = \frac{(120-112)-(120-102)}{\ln\left[\frac{120-112}{120-102}\right]}$$

$\Delta T_{lm} = 12.33\ K$

**To calculate heat transfer area:**

$Q = U\ A\ \Delta T_{lm}$

Where, Q= heat load (kcal/hr)

U= Overall heat transfer coefficient (kcal/$m^2$ K)

Therefore, A= 140400/ (1500 *12.33)

**A =7.59 $m^2$**

$A = n * 3.14 * D_o * L$

Assuming, Length of tubes =6m

Diameter of tube = 1”

Therefore, **n= 15.86 ≈ 16** (No. of tubes)

### 6.7 Heat exchanger

- **Heater-1**

$Q= m' \times c_p' \times \triangle T = m \times c_p \times \triangle T$ = 12532.19 kcal/hr

Therefore,

$626.44 * 0.49 * (74-T) = 12532.19$

$T = 33.17\ ^oC$

$$\triangle T_{lm} = \frac{(74 - 55) - (33.17 - 30)}{\ln [(74 - 55)/ (33.17 - 30)]} = 8.84^oC$$

$Q = A \times U \times \triangle T_{lm}$

Overall heat transfer = 1200 kcal/m$^{2o}$C

$A = 12532.19/ (1200 \times 8.84) = 1.18\ m^2$

$$\text{No. of tubes required} = \frac{1.18}{(3.14 \times 0.0254 \times 1.5)}$$

No. of tubes required = 10

**Table 11:** Specifications of heat exchangers

| Sr no. | HE | Heat transfer area | no. of tubes | length of HE |
|---|---|---|---|---|
| | | m$^2$ | | m |
| 1 | HE-1 | 2.04 | 17 | 6 |
| **2** | HE-2 | 0.24 | 2 | 6 |
| 3 | HE-3 | 0.39 | 4 | 6 |
| 4 | HE-4 | 4.33 | 9 | 6 |
| 5 | HE-5 | 5.21 | 11 | 6 |
| 6 | HE-6 | 0.03 | 1 | 6 |
| 7 | HE-7 | 1.24 | 11 | 6 |
| 8 | HE-8 | 1.49 | 4 | 6 |
| 9 | HE-9 | 0.515 | 2 | 6 |

## 7. Equipment List Summary

A summary of all the equipments used and their specifications are provided in this section.

**Table 12.1:** Equipment list

| Sr. No | Description | code | Specification | | | MOC |
|---|---|---|---|---|---|---|
| 1 | Reactor | R-1 | Cylindrical vessel | | | SS316L |
| | | | Diameter | 225 | mm | |
| | | | Height | 562 | mm | |
| | | | thickness | 1.5 | mm | |
| | Reactor | R-2 | Cylindrical vessel | | | SS316L |
| | | | Diameter | 263 | mm | |
| | | | Height | 658 | mm | |
| | | | thickness | 1.5 | mm | |
| | Reactor | R-3 | Cylindrical vessel | | | SS316L |
| | | | Diameter | 301 | mm | |
| | | | Height | 753 | mm | |
| | | | thickness | 1.5 | mm | |
| 2 | Ethyl acetate storage vessel | V-1 | Diameter | 1950 | mm | SS316 |
| | | | Height | 5341 | mm | |
| | | | thickness | 2.4 | mm | |
| | Substrate storage vessel | V-2 | Diameter | 1236 | mm | SS316 |
| | | | Height | 3709 | mm | |
| | | | thickness | 2.1 | mm | |
| | $HNO_3$ Storage tank | V-3 | Diameter | 2222 | mm | SS316L |
| | | | Height | 4636 | mm | |
| | | | thickness | 2.6 | mm | |
| | $H_2SO_4$ Storage vessel | V-4 | Diameter | 2614 | mm | SS316L |
| | | | Height | 6674 | mm | |
| | | | thickness | 1.2 | mm | |
| | Acid mixing vessel | T-1 | Diameter | 741 | mm | SS316L |
| | | | Height | 1797 | mm | |
| | | | thickness | 2 | mm | |
| | Product collection vessel | T-2 | Diameter | 1071 | mm | SS316L |
| | | | Height | 1982 | mm | |
| | | | thickness | 2 | mm | |
| 3 | Decanter | D-1 | Diameter | 1350 | mm | SS316L |
| | | | Height | 2710 | mm | |
| 4 | Distillation column | C-1 | Diameter | 440 | mm | SS316 |
| | | | Qc | 163880 | kcal/hr | |
| | | | QR | 160261 | kcal/hr | |
| | | C-2 | Diameter | 512 | mm | SS316 |
| | | | Qc | 16273 | kcal/hr | |
| | | | QR | 55346 | kcal/hr | |

| | | | | | | |
|---|---|---|---|---|---|---|
| 5 | Evaporator | E-1 | Area | 7.59 | m$^2$ | SS316 |
| | | | no. of tubes | 16 | | |
| 6 | Crystallizer | Z-1 | Capacity | 0.042 | m$^3$ | SS307 |

**Table 12.2:** Equipment list for pumps

| Sr. No | Description | code | Specification | | | MOC |
|---|---|---|---|---|---|---|
| 7 | EA pump | P-1 | Q | 0.737 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 8 | MWC | |
| | substrate pump | P-2 | Q | 0.892 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 8 | MWC | |
| | Acid pump | P-3 | Q | 0.352 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 8 | MWC | |
| | | P-4 | Q | 1.752 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | Mix acid pump | P-5 | Q | 1.924 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | CSTR Outlet | P-6 | Q | 2.196 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | | P-7 | Q | 2.196 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | | P-8 | Q | 2.196 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | Quenching tank | P-9 | Q | 2.549 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 8 | MWC | |
| | Aqueous phase transfer | P-10 | Q | 2.557 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 12 | MWC | |
| | Organic phase transfer | P-11 | Q | 0.472 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | Residue transfer from C-1 | P-12 | Q | 0.054 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 8 | MWC | |
| | Reflux from C-1 | P-13 | Q | 0.016 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 12 | MWC | |
| | Transfer from crystallizer | P-14 | Q | 0.054 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |
| | Residue transfer fromC-2 | P-15 | Q | 2.046 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 8 | MWC | |
| | Reflux from C-2 | P-16 | Q | 0.106 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 12 | MWC | |
| | Transfer from evaporator | P-17 | Q | 1.901 | m$^3$/hr | CF-8 |
| | | | $\Delta h$ | 10 | MWC | |

**Table 12.3:** Equipment list for Heat exchangers

| Sr. No | Description | code | Specification | | | MOC |
|---|---|---|---|---|---|---|
| 8 | Heat exchangers | HE-1 | Area | 2.04 | $m^2$ | SS316 |
| | | | no. of tubes | 17 | | |
| | | HE-2 | Area | 0.24 | $m^2$ | SS316 |
| | | | no. of tubes | 2 | | |
| | | HE-3 | Area | 0.39 | $m^2$ | SS316 |
| | | | no. of tubes | 9 | | |
| | | HE-4 | Area | 4.33 | $m^2$ | SS316 |
| | | | no. of tubes | 9 | | |
| | | HE-5 | Area | 5.21 | $m^2$ | SS316 |
| | | | no. of tubes | 11 | | |
| | | HE-6 | Area | 0.03 | $m^2$ | SS316 |
| | | | no. of tubes | 1 | | |
| | | HE-7 | Area | 1.24 | $m^2$ | SS316 |
| | | | no. of tubes | 11 | | |
| | | HE-8 | Area | 1.49 | $m^2$ | SS316 |
| | | | no. of tubes | 4 | | |
| | | HE-9 | Area | 0.515 | $m^2$ | SS316 |
| | | | no. of tubes | 2 | | |

## 8. Cost estimation [18]:

### 8.1 Purchased equipment cost (Based on cost index):

**Table 13:** Purchased equipment cost

| Sr no. | Equipment | Quantity | Cost/ unit Lakh Rs. | Total cost Lakh Rs. |
|---|---|---|---|---|
| 1 | Reactor (R-1) | 1 | 1.02 | 1.02 |
| 2 | Reactor (R-2) | 1 | 1.32 | 1.32 |
| 3 | Reactor (R-3) | 1 | 1.7 | 1.7 |
| 4 | Storage vessels (V-1 to 6) | 1 | 210.16 | 210.16 |
| 5 | Acid mixing tank (T-1) | 1 | 8.47 | 8.47 |
| 6 | collection tank (T-2) | 1 | 16.37 | 16.37 |
| 7 | Distillation column 1 (C-1) | 1 | 12.483 | 12.483 |
| 8 | Distillation column 1 (C-2)) | 1 | 14.19 | 14.19 |
| 9 | Crystallizer unit | 1 | 152.43 | 152.43 |
| 10 | Decanter | 1 | 4.631 | 4.631 |
| 11 | Evaporator | 1 | 129.13 | 129.13 |
| 12 | Heat exchanger | 9 | 1.76 | 15.84 |
| 13 | Centrifugal pumps | 17 | 0.365 | 6.205 |
|  | **Purchased Equipment cost (E)** | **Total (Lakh Rs.)** |  | **573.949** |

### 8.2 Capital Cost Estimation:

**Table 14:** Capital Cost estimation

| **Direct costs** | **Range of FCI%** | **Normalized Percentage** | **Cost (Lakh Rs)** |
|---|---|---|---|
| Purchased Equipment | 25 | 0.2262 | 573.949 |
| Purchased Equipment Installation | 8 | 0.0724 | 183.664 |
| Instrumentation and controls | 4 | 0.0362 | 91.832 |
| Piping | 7 | 0.0633 | 160.706 |
| Electrical systems | 5 | 0.0452 | 114.790 |
| Building | 9 | 0.0814 | 206.622 |
| Yard Improvement | 2 | 0.0181 | 45.916 |
| Service Facilities | 15 | 0.1357 | 344.369 |
| Land | 1.5 | 0.0136 | 34.437 |
|  |  |  |  |
| **Indirect cost** | **Range of FCI%** | **Normalized Percentage** | **Cost (Lakh Rs)** |
| Engineering and supervision cost | 10 | 0.0905 | 229.580 |
| Construction Expenses | 10 | 0.0905 | 229.580 |
| Legal Expenses | 2 | 0.0181 | 45.916 |
| Contractor Fee | 2 | 0.0181 | 45.916 |
| Contingency | 10 | 0.0905 | 229.580 |
| Total | 110.5 | 1 | 2536.85 |

Fixed Capital Investment (FCI) = Direct Cost + Indirect Cost

=1756.28 + 780.51

= 2536.85 lakh

Total Capital Investment (TCI)= Working capital Investment + Fixed Capital Investment

Fixed capital investment is 80% of Total Capital Investment.

Therefore, TCI = 3171.068 lakh Rs.

Working Capital Investment (WCI) = 634.21 lakh Rs

## 8.3 Total Production cost Estimation:

**Sample calculations:**

**Raw Material cost:**

**Table 15:** Total production cost estimation

| Reactants | Cost /kg (Rs) | kg of reactant/ 1000kg of product | Cost per 1000kg product (Lakh Rs) |
|---|---|---|---|
| Salicylic acid | ₹ 455.00 | 5002.84 | ₹ 22.76 |
| Acetic acid | ₹ 340.00 | 435.01 | ₹ 1.47 |
| Nitric acid | ₹ 300.00 | 2464.46 | ₹ 7.39 |
| Ethyl acetate | ₹ 100.00 | 30.13 | ₹ 0.03 |
| TOTAL | | | ₹ 31.66 |

Assuming working days per year as 330 days

Thus, annual raw material cost = 330*31.6655 = 10449.6 Lakh Rs

Assuming raw material cost is 50% of total production cost

TPC =20899.21 lakh Rs

**Income from sales:**

**Table 16:** Income from sales per day

| Products | Cost/ kg (Rs) | Kg of product produced/day | total sales/day (Lakh Rs) |
|---|---|---|---|
| 5Nitro salicylic acid | ₹ 2,500.00 | 1000 | ₹ 25 |
| 3 Nitro salicylic acid | ₹ 7,000.00 | 647.39 | ₹ 45.31 |
| Total | | | ₹ 70.31 |

Annual income from sales = 330*70.31 =23204.68 lakh Rs.

Similarly, all calculations are performed:

**Table 17:** Annual income from sales for each substrate

| Substrate | Unit | SA | AG | CB | MB | TFMB |
|---|---|---|---|---|---|---|
| **Total production cost** | Lakh Rs | ₹20,899.24 | ₹ 20,672.64 | ₹ 3,551.82 | ₹ 7,309.20 | ₹ 50,567.85 |
| **Annual income from sales** | Lakh Rs | ₹ 23,204.68 | ₹ 22,860.18 | ₹ 5,133.92 | ₹ 16,150.2 | ₹1,03,484.84 |

### 8.4 Depreciation (Double Declining Balance Method)

Taking fixed percentage factor f=0.2

Initial depreciable amount =FCI = 2536.85 lakh Rs

Assuming salvage value= 0

Depreciation for 1$^{st}$ year= f * FCI = 507.37 Lakh Rs

Thus, asset value at the end of 1$^{st}$ year = FCI – Depreciation

= 2029.48 lakh Rs

Thus, generating depreciation for 10 years:

**Table 18:** Depreciation values for 10 years

| Year | Depreciation (Lakh Rs) | Asset value (Lakh Rs) |
|---|---|---|
| 0 | | 2536.85 |
| 1 | 507.37 | 2029.48 |
| 2 | 405.90 | 1623.59 |
| 3 | 324.72 | 1298.87 |
| 4 | 259.77 | 1039.10 |
| 5 | 207.82 | 831.28 |
| 6 | 166.26 | 665.02 |
| 7 | 133.00 | 532.02 |
| 8 | 106.40 | 425.61 |
| 9 | 85.12 | 340.49 |
| 10 | 68.10 | 272.39 |

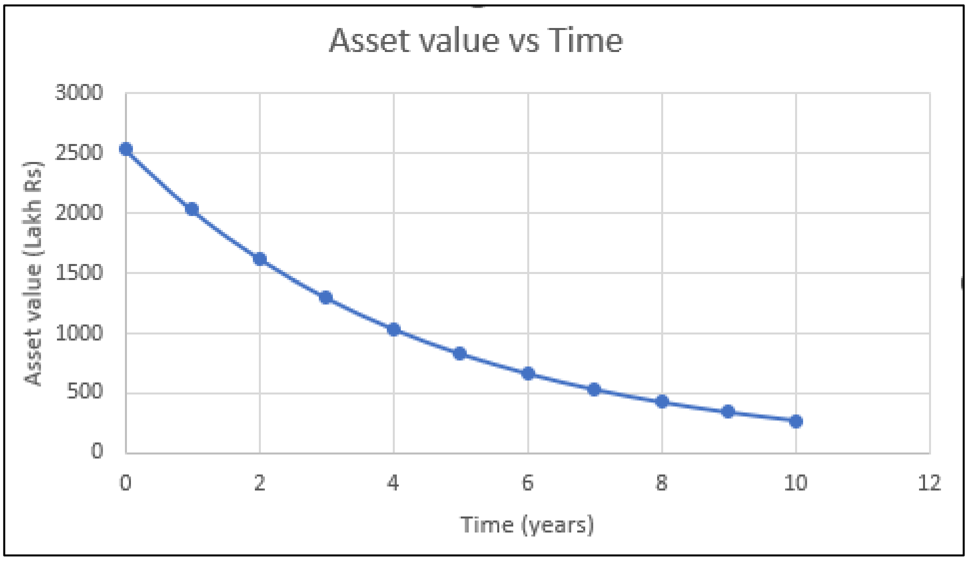


**Fig 10:** Asset value (Lakh Rs) vs time (years)

## 8.5 Cash Flow

Annual income from sales of salicylic acid =23204.68 Lakh Rs.

Total product cost = 20899.24 lakh Rs

Thus,

Gross profit = 2305.45 Lakh Rs.

Deducting depreciation

Net profit before taxes = 1798.08 Lakh Rs (Taxable income)

Assuming 35% Income tax

Net profit after taxes = 1168.75 lakh Rs

**Table 19:** Profit analysis for each substrate.

| **Parameter** | **Unit** | **SA** | **AG** | **CB** | **MB** | **TFMB** |
|---|---|---|---|---|---|---|
| Annual income from sales | Lakh Rs | ₹ 23,204.68 | ₹ 2,860.18 | ₹ 5,133.92 | ₹16,150.20 | ₹1,03,484.84 |
| Total production cost | Lakh Rs | ₹ 20,899.24 | ₹ 20,672.6 | ₹ 3,551.8 | ₹ 14,618.4 | ₹1,01,135.69 |
| Gross profit | Lakh Rs | ₹ 2,305.45 | ₹ 2,187.54 | ₹1,582.11 | ₹ 1,53.80 | ₹ 2,349.15 |
| Net profit before taxes | Lakh Rs | ₹ 1,798.08 | ₹ 1,680.17 | ₹ 1,074.74 | ₹ 1,024.43 | ₹ 1,841.78 |
| Net profit after taxes | Lakh Rs | ₹ 1,168.75 | ₹ 1,092.11 | ₹ 698.58 | ₹ 665.88 | ₹ 1,197.16 |

## 8.6 Cash flow for Salicylic acid Nitration (Lakh Rs)

**Table 20:** Cash flow for salicylic acid nitration plant

| year | Depreciation | Asset value | Gross profit | Net profit before taxes | Net profit after taxes | Cumulative Cash flow | Cash position |
|---|---|---|---|---|---|---|---|
| 0 | | 2536.85458 | | | | | -3171.07 |
| 1 | 507.37 | 2029.48 | 2305.45 | 1798.08 | 1168.75 | 1168.75 | -2002.32 |
| 2 | 405.90 | 1623.59 | 2305.45 | 1899.55 | 1234.71 | 2403.46 | 401.14 |
| 3 | 324.72 | 1298.87 | 2305.45 | 1980.73 | 1287.47 | 3690.93 | 4092.07 |
| 4 | 259.77 | 1039.10 | 2305.45 | 2045.67 | 1329.69 | 5020.62 | 9112.69 |
| 5 | 207.82 | 831.28 | 2305.45 | 2097.63 | 1363.46 | 6384.08 | 15496.76 |
| 6 | 166.26 | 665.02 | 2305.45 | 2139.19 | 1390.47 | 7774.55 | 23271.31 |
| 7 | 133.00 | 532.02 | 2305.45 | 2172.44 | 1412.09 | 9186.64 | 32457.95 |
| 8 | 106.40 | 425.61 | 2305.45 | 2199.04 | 1429.38 | 10616.02 | 43073.97 |
| 9 | 85.12 | 340.49 | 2305.45 | 2220.32 | 1443.21 | 12059.23 | 55133.19 |
| 10 | 68.10 | 272.39 | 2305.45 | 2237.35 | 1454.28 | 13513.50 | 68646.69 |

- **Cash flow for all substrates (Lakh Rs)**

**Table 21:** Cash flow for all substrates

| Year | SA | AG | CB | MB | TFMB |
|---|---|---|---|---|---|
| 0 | -3171 | -3171 | -3171 | -3171 | -3171 |
| 1 | -2002 | -2079 | -2802 | -2505 | -1974 |
| 2 | 401 | 171 | -1999 | -1107 | 486 |
| 3 | 4092 | 3632 | -708 | 1075 | 4263 |
| 4 | 9113 | 8346 | 1113 | 4084 | 9397 |
| 5 | 15497 | 14347 | 3497 | 7954 | 15923 |
| 6 | 23271 | 21662 | 6472 | 12711 | 23868 |
| 7 | 32458 | 30312 | 10059 | 18378 | 33253 |
| 8 | 43074 | 40315 | 14275 | 24971 | 44097 |
| 9 | 55133 | 51684 | 19135 | 32504 | 56411 |
| 10 | 68647 | 64432 | 24649 | 40989 | 70209 |

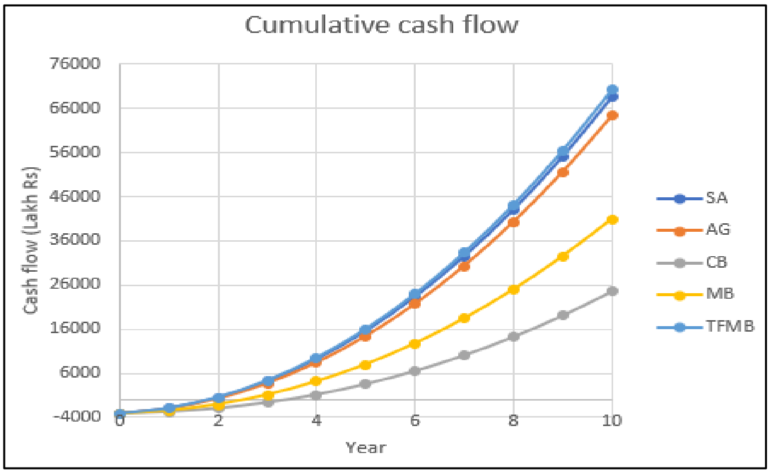


**Fig 11:** Cumulative cash flow

- **Payback period = Depreciable FCI/ Annual net profit**

**Table 22:** Payback period for all substrates

| | SA | AG | CB | MB | TFMB |
|---|---|---|---|---|---|
| Payback period (years) | 2.17 | 2.32 | 6.88 | 3.81 | 2.12 |

- **Rate of return on investment = Annual Net Profit/ Total capital investment**

**Table 23:** Rate of return on investment for all substrates

| | SA | AG | CB | MB | TFMB |
|---|---|---|---|---|---|
| ROI (%) | 36.86 | 34.44 | 11.63 | 21 | 37.75 |

***For all substrates production rate is 1000kg/day except for Methyl Benzoate (MB) it is 250kg/day

## 9. Plant Location and layout:

After developing a process flow diagram and completing the preliminary plant design, it is necessary to designate the location and design the layout for the proposed process before beginning with the detailed piping, structural and electrical design. The geographical location of the plant has a strong influence to determine the success of the industry. The final location of the plant should be selected by considering the pro and cons of the available real estate. A detailed survey is necessary and many different factors must be scrutinized. The preliminary requirement is that the cost of production and distribution should be minimum but other factors such as safe living conditions for the plant operation and space for plant expansion cannot be ignored. The principal factors which must be considered before choosing the final site are as follows [18]:

a) Availability of raw materials.
b) Market area and transportation facilities.
c) Utilities: Energy supply and water supply.
d) Climatic conditions.
e) Effluent disposal and environmental norms.
f) Local community considerations.
g) Availability of labor.
h) Legal restrictions.

The first four factors are very essential. But the final decision should be based on considering all the factors mentioned above.

The layout should be designed such that efficient coordination is possible between all the facilities present in the plant. An optimum layout is proportional to efficient operation, reduction in manufacturing and distribution cost, easy handling, supply, and storage of materials, and easy access to the required facilities without disturbing any operation on the plant. The design of the layout will vary as no two plants are alike in terms of operation and plant location. Hence no generalized plant layout is ideal for the designed process. The factors that should be included in the designing of the plant layout are as follows:

a) Future expansion of the present site or new developments.
b) Type and quantity of the product that should be produced and stored.
c) Accessibility to all facilities and convenience in operation.
d) Economical distribution of utilities and services.

e) Health and medical considerations.

f) Waste disposal and its treatment.

g) Type of building, space available, and space required.

h) Roadways and railroads.

By studying all the points, a preliminary layout for the aromatic nitration process can be designed as follows:

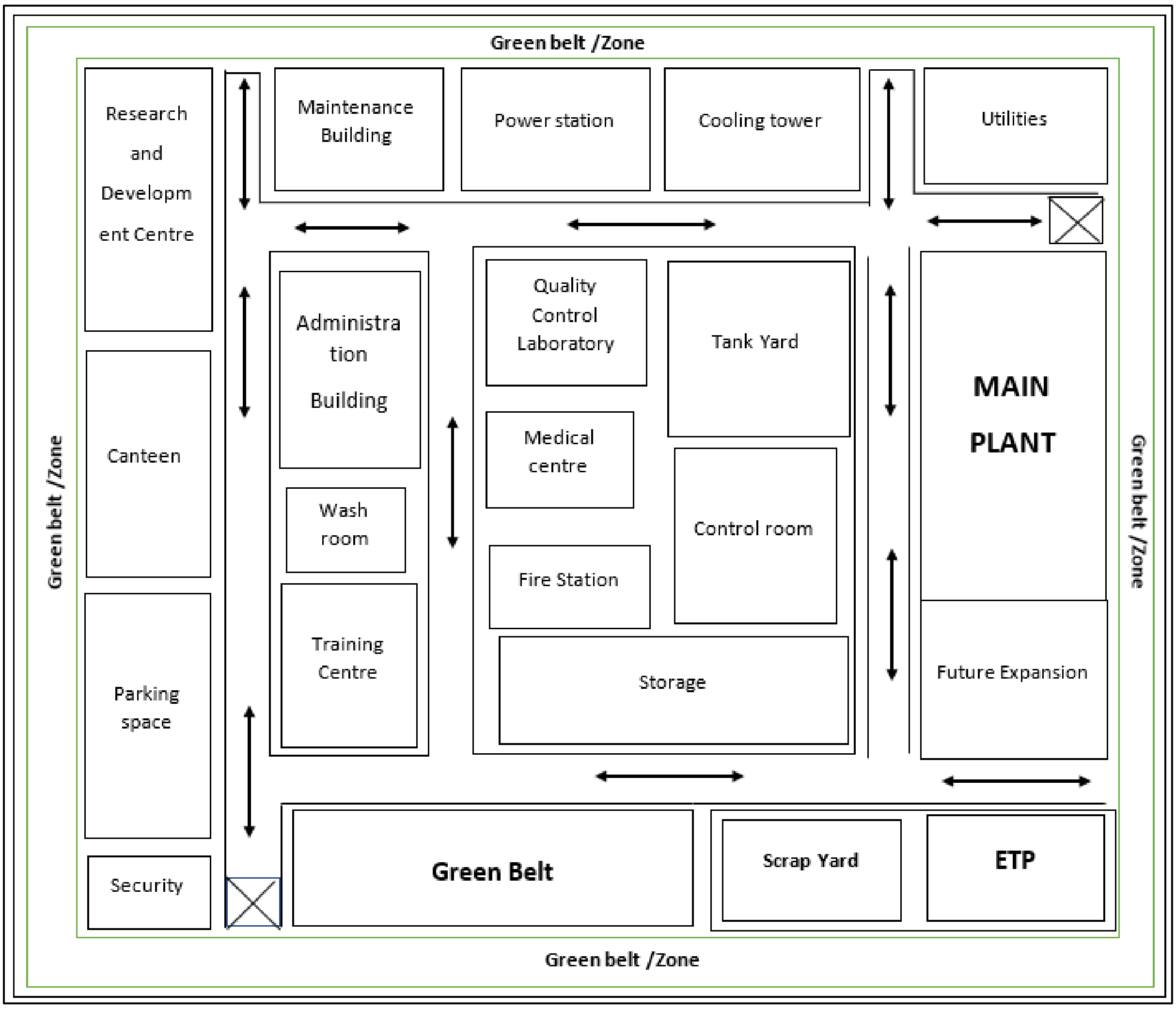


**Fig 12:** Suggested plant layout for the aromatic nitration process

## 10. Comparative study:

This section provides the comparative study between the process (batch) which is recently used in industry versus the process designed in this work in terms of footprint.

- **Footprint comparison:**

For salicylic acid nitration, 100% conversion was achieved in 16 min residence time using overall volume of 83 lit. For batch reaction, feeding, reaction and transfer rate needs to be considered which will increase the total reaction (cycle) time.

Assuming, feeding time to be 10 mins (slow addition of reactants because of the exothermicity). The transfer time is assumed to be 10 mins and the reaction time is 20 mins.

Feeding rate (F)= 10 min
Reaction time (R)=20 min
Transfer rate (T)= 10 min
Hence total time per cycle for batch = 20+10+20 = 50 mins
No. of reactors required = [(F+R)/T] + 1
= 4 reactors
No. of reactors discharged per day = (60*24)/10 =144

Volume to be discharged per day = (43307/ (1.7*144)) = 177 lit
Per reactor volume discharged= (177/4) = 44.3 lit
Therefore, for batch operation overall volume required is 177 lit (44.3 lit each)
The obtained volume is 2.2 time more than that required for this process.
Considering L/D ratio as 2.5
Therefore, diameter of each batch reactor= 304 mm
Height of each reactor = 760 mm
For continuous reactor, diameter of reactor 1,2 and 3 are 208 mm, 261mm and 297 mm

→Total floor area required for batch process is 1.62 times the total floor area required for continuous process.

- **Type of feed:**

The designed process can be used to produce multiple nitroaromatic products without any change in the process. Hence multiple feed can be processed just by changing the operating parameters which is not the case in the processes used till date.

## 11. Conclusion

Aromatic nitration plant with multi-feed approach to achieve 1TPD of capacity was successfully designed. The work presented in this report can serve as one of the preliminary steps for developing a continuous commercial process for manufacturing of multiple nitro aromatic products. Detailed design of equipments along with mass and energy balance over a proposed continuous process were carried out. The cost for annual production was estimated along with the cash flows and profitability analysis for each substrate. The annual profit achieved provided high return on investments with a low pay back period. From the comparative analysis it was found that the footprint for the proposed process was 1.62 times less than the conventionally used process. Multiple aromatic feeds can be processed to produce multiple nitro aromatic products using the single plant just by changing the operating condition. Further studies based on the environmental factors needs to be carried out followed by designing of an effective effluent treatment strategy.

## References

1. G. Olha, R. Malhotra, S. Narang, 1988. ‘Nitration methods and mechanisms: Organic nitro chemistry series’
2. A. Kulkarni, N. Nivangune, V. Kalyani, et al, ‘Continuous Flow Nitration of Salicylic Acid’ Organic Process Research & Development, 2008, 12, 995-1000.
3. C. Zhang, J. Zhang and G. Luo: ‘Kinetic study and intensification of Acetyl Guaiacol Nitration with Nitric acid- Acetic Acid System in a microreactor’ J. Flow Chem. 2016, 6 (4), 309-314.
4. Y. Cui, J. Song, C. Du, et al. ‘Determination of kinetics of chlorobenzene nitration using a homogenously continuous microflow’2021. doi:10.22541/au.162027391.120018968.
5. ‘Para nitro chlorobenzene Market size, share and & trend analysis report by application (Dyes, Pesticides, Rubber, chemicals), by end use (Pharmaceuticals, chemicals), and segment forecasts, 2020-2027’, www.grandviewreasearch.com.
6. I. Di Somma, R. Marotta, R. Androezzi, et al. ‘Kinetic and Safety Characterization of the Nitration Process of Methyl Benzoate in Mixed Acid’ organic process research and development, ACS Publication, 2012, 16, 2001−2007.
7. Z. Wen, M. Yang, et al. ‘Kinetics study of heterogeneously continuous-flow nitration of trifluoromethoxybenzene’ Royal society of chemistry, 2013, 1-3.
8. A. Kulkarni: Continuous flow nitration in miniaturized devices, Beilstein Journal of Organic Chemistry, 10(1), 405-424, 2014.
9. G. M. Mule, S. Kulkarni, A. A. Kulkarni, An assessment of a multipoint dosing approach for exothermic nitration in CSTRs in series. Royal Society of Chemistry, 2022.
10. Andreozzi R, Canterino M, Caprio V. et al: Salicylic acid nitration by means of Nitric acid/ Acetic acid System: Chemical and kinetic Characterization. Org Process Research & Development, 2006, 10, 1199-1204.

11. Debnath S, Kienle A, Kulkarni A: Evaluation of multipoint Dosing strategy in a miniaturized tubular reactor: Nitration of Salicylic Acid. Chem. Eng. Technol, 2014, 37, No.6, 927-937.
12. Veretennikov E., Lebedev B., and Tselinskii I., Nitration of chlorobenzene with nitric acid in a continuous installation, Russ J. Appl. Chem, 2001, 74(11), 1872-1876.
13. O. Levenspeil, ‘Chemical Reaction Engineering’, 3rd edition, John Wiley and Sons.
14. M. V. Joshi, V.V. Mahajani, ‘Process Equipment design’ MacMillan Publication, India.
15. R. K. Sinnot, ‘Coulson & Richardson’s Chemical Engineering- Volume 6, Chemical Engineering Design’, 4th Edition, Elsevier Butterworth- Heinemann.
16. K.G. Joback and R. C. Reid, estimation of pure-component properties from group contributions, Chem. Eng Commun, 1987, 57(1-6), 233-243.
17. R. H. Perry, D.W. Green, ‘Perry’s Chemical Engineers’ Handbook’ 7th edition, McGrawHill Companies.
18. M. S. Peters, K. D. Timmerhaus, ‘Plant Design and economics for chemical Engineers, 4th edition, McGraw-Hill Companies.