

MANUFACTURE OF OXALIC ACID FROM MOLASSES

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In

Chemical Engineering

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BONAFIDE CERTIFICATE

This is to certify that the project titled **MANUFACTURE OF OXALIC ACID FROM MOLASES** is a bonafide record of the work done by

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In partial fulfilment of the requirements for the award of the degree of **Bachelors of Technology** in **Chemical Engineering** of the **NATIONAL INSTITUTE OF TECHNOLOGY, TIRUCHARAPPALLI**, during the year 2017-2018.

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ABSTRACT

Oxalic acid is the simplest of dicarboxylic acid. Its name is derived from the Greek Oxyes meaning sharp, acidic suffering to the acidity common in the foliage of certain plants from which it was first isolated. Oxalic acid is commercially available as the di-hydrate containing 28.5% water. It is a monoclinic prism, particle sizes vary from fine powder to coarse granules that are also colorless, with a melting point of 187°C for the anhydrous form and 101.5°C for the di-hydrate form. Oxalic acid is one of the most widespread organic acid in plants.

In this project, a commercial method of preparation of Oxalic acid at 24 tons per day is presented using molasses as the starting material. This project highlights the material and energy balance as well as description of a few equipment used in this process.

Other important features includes cost estimation, a possible plant layout based on scientific principles and commercial requirement. It also involves details regarding Plant Safety.

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CHAPTER 1: INTRODUCTION

Oxalic acid is an organic compound with the formula $\text{H}_2\text{C}_2\text{O}_4$. It is a colorless crystalline solid that dissolves in water to give colorless solutions. It is classified as a dicarboxylic acid. In terms of acid strength, it is much stronger than acetic acid. Oxalic acid is a reducing agent and its conjugate base, known as oxalate ($\text{C}_2\text{O}_4^{2-}$), is a chelating agent for metal. Oxalic acid is a toxic organic acid, which is also called ethanoic acid.

1.1 PHYSICAL & CHEMICAL PROPERTIES

Appearance	Transparent, colorless, odorless crystals
Solubility	50 to 100 mg/mL at 75° F
Specific Gravity	1.65
Boiling Point	149 - 160°C (sublimes)
Melting Point	101.5°C(sublimes), 188.89°C(decomposes)
Vapor Density	4.4 (air=1)
Vapor Pressure	< 0.001 @ 20°C (mm Hg)
Molecular Weight	90.03
Chemical Formula	$\text{HOCCOOH} \cdot 2\text{H}_2\text{O}$
Hydrogen Bond Donor Count	2
Hydrogen Bond Acceptor Count	4
Rotatable Bond Count	1
XLogP3-AA	-0.3
Formal Charge	0
Auto-Ignition	Not Flammable
Heat of Combustion	-245kJ/mol

Table 1: Properties of Oxalic Acid

Oxalic acid is purchased usually in the form of oxalic acid dihydrate, which is a crystalline form with two water molecules attached to each molecule of oxalic acid. Oxalic acid is the simplest of dicarboxylic acid. Its name is derived from the Greek Oxeyes meaning sharp,

acidic suffering to the acidity common in the foliage of certain plants from which it was first isolated. Oxalic acid is commercially available as the dihydrate containing 28.5% water. Oxalic acid is odourless and white in colour. It is a monoclinic prism, with particles size varying from fine powder to coarse granules, which are also colourless, melting point 188.89°C of anhydrous form .Oxalic acid is a normal metabolite of carbohydrates in normal quantities, e.g., Fructose, Glycine, and Vitamin C.

Oxalic acid and its oxalate derivatives are found in several natural sources such as plants, vegetables, leaves, wood and coal, among others. It is also produced in the body by metabolism of glyoxylic acid or ascorbic acid

1.2 MOLASSES & ITS PROPERTIES

Molasses is a viscous by-product of the refining of sugarcane, grapes, or sugar beets into sugar. The word comes from the Portuguese melaço. Cognates include Ancient Greek μέλι (méli) (honey), Latin mel, Spanish melaza (molasses), and French miel (honey) The quality of molasses depends on the maturity of the source plant, the amount of sugar extracted, and the method employed. Molasses are of various type based on plant material from which it is produced. So different type of molasses produced base upon on various raw material are cane molasses from sugarcane, beet molasses from sugar beet and grape molasses from grapes. Molasses is a defining component of fine commercial brown sugar. Sweet sorghum syrup is colloquially called "sorghum molasses" in the southern United States. Molasses varies by amount of sugar, method of extraction, and age of plant. Its major constituents' are- .

- 1) Glucose – 35.9%
- 2) Fructose- 5.6%
- 3) Sucrose – 2.6%
- 4) Water - 23.5%

1.3 USES OF MOLASSES

Molasses is commonly used with yeast and water in the fermentation process of making rum. It is also used in creating some other alcoholic drinks such as stout and dark ales.

Some tobacco companies add it to their product for smoking through a certain type of pipe popular in the Middle Eastern countries.

Some anglers use molasses as a ground bait (also called chum or barley). This is designed to attract fish to the area that an angler is fishing.

It can also be mixed with water and used to remove rust.

It is used in some horticultural settings to promote microbe activity in the soil.

Molasses contains trace amounts of important vitamins and minerals. A certain type of molasses called blackstrap molasses has been sold as a health supplement for many years. People have made many claims about its health benefits, including the ability to prevent gray hair.

Molasses is also added to some cattle feed to add essential vitamins and minerals. Glucose, a major component of molasses also known as D-glucose, dextrose, or grape sugar is a simple monosaccharide found in plants. It is one of the three dietary monosaccharides, along with Fructose and Galactose that are absorbed directly into the bloodstream during digestion. An important carbohydrate in biology, cells use it as the primary source of energy and a metabolic intermediate. Glucose is one of the main products of photosynthesis and fuels for cellular respiration. Glucose exists in several different molecular structures, but all of these structures can be divided into two families of mirror-images (stereoisomers). Only one set of these isomers exists in nature, those derived from the "right-handed form" of glucose, denoted D-glucose. D-glucose is sometimes referred to as dextrose, although the use of this name is strongly discouraged. The term dextrose is derived from *dextrorotatory glucose*. This name is therefore confusing when applied to the enantiomer, which rotates light in the opposite direction. Starch and cellulose are polymers derived from the dehydration of D-glucose. The other stereoisomer, called L-glucose, is

hardly ever found in nature. The name "glucose" comes from the Greek word *glukus* meaning "sweet".

1.4 OTHER FACTS ABOUT MOLASSES

The quality of Molasses depends on the maturity of the sugarcane or sugar beet, the amount of sugar extracted, and the method of extraction. Sweet sorghum is known in some parts of the United States as molasses. So molasses are of different kinds, based on their raw material. Sulfured molasses is made from young sugarcane. Sulfur dioxide, which acts as a preservative, is added during the sugar extraction process. Unsulphured molasses is made from mature sugarcane, which does not require such treatment. The three grades of molasses are: mild or Barbados, also known as first molasses; dark, or second molasses; and blackstrap. These grades may be sulphured or unsulphured. To make molasses, the cane of a sugar plant is harvested and stripped of its leaves. Its juice is extracted usually by crushing or mashing. The juice is boiled to concentrate it, which promotes the crystallization of the sugar. The result of this first boiling and of the sugar crystals is first molasses, which has the highest sugar content because comparatively little sugar has been extracted from the source. Second molasses is created from a second boiling and sugar extraction, and has a slight bitter tinge to its taste. The third boiling of the sugar syrup yields blackstrap molasses, known for its robust flavor. The majority of sucrose from the original juice has been crystallized and removed. The food energy content of blackstrap molasses is still mostly from the small remaining sugar content. However, unlike refined sugars, it contains trace amounts of vitamins and significant amounts of several minerals. Blackstrap molasses is a source of calcium, magnesium, potassium and iron, one tablespoon provides up to 20% of the daily value of each of those nutrients. Blackstrap has long been sold as a health supplement. It is also used in the manufacture of ethyl alcohol for industry and as an ingredient in cattle feed. Molasses made from sugar beet is different from sugarcane molasses. Only the syrup left from the final crystallization stage is called molasses; intermediate syrups are referred to as high green and low green, and these are recycled within the crystallization plant to maximize extraction. Beet molasses is about 50% sugar by dry weight, predominantly sucrose, but also contains significant amounts of glucose and fructose. Beet molasses is limited in biotin (vitamin H or B₇) for cell growth;

hence, it may need to be supplemented with a biotin source. The non-sugar content includes many salts, such as calcium, potassium, oxalate, and chloride. It also contains the compounds betaine and the trisaccharide raffinose. These are either as a result of concentration from the original plant material or as a result of chemicals used in the processing, and make it unpalatable to humans. Hence it is mainly used as an additive to animal feed (called "molassed sugar beet feed") or as a fermentation feedstock. It is possible to extract additional sugar from beet molasses through a process known as molasses desugarisation. This technique exploits industrial scale chromatography to separate sucrose from non-sugar components. The technique is economically viable in trade-protected areas, where the price of sugar is supported above the world market price. Molasses is also used for production of Oxalic acid.

Processes involved for production of oxalic acid from molasses are-

1. Using Nitric Acid
2. Fermentation Process
3. Using *Aspergillus Niger*

CHAPTER 2: LITERATURE REVIEW

2.1 METHODS OF OXALIC ACID PRODUCTION

2.1.1 PREPARATION OF OXALIC ACID BY MOLASSES USING NITRIC ACID

2.1.1.1 Laboratory Preparation

The sugar beet molasses used in the experiment contained 48.1% sucrose, 3.2% invert sugar and 19.4% water. The chemicals employed were 65% HNO_3 and 93% H_2SO_4 . As catalyst, 7.5% V_2O_5 on porous silica gel support was used.

2.1.1.2 Process in Detail

Three reactor are used in the experiments. While the reactor 1 was used to produce oxalic acid from molasses with mixed nitric acid and sulphuric acid in the optimum conditions of it is simultaneously utilized as a source of nitrogen oxides needed as reactant in the second and third reactors. The reaction gases released from the final reactor are absorbed in the columns at 0°C . All of the reactants are prepared per 50 g molasses for each reactor. A predetermined amount of Vanadium Pentoxide catalyst was added to each reactor. The magnetic stirrers, heaters, cooling water and a cooler are turned on and air started to bubble through. Mixed acid is added to the reactor at a rate of 6 mL/min through a funnel to the neck of the flask. The $\text{HNO}_3 + \text{H}_2\text{SO}_4$ mixture is added from the dropping funnel as a thin stream. Initially the reaction in the reactor is started. The reaction temperature is controlled by adjusting the heating unit or cooling it with water using a water bath. The oxides of Nitrogen evolving from the reaction in the reactor were carried, by air to the second and third reactors. These Oxides were meant to act as oxidizers in the second and third reactors that contained enough H_2SO_4 , but no HNO_3 . In order to see how the conditions in the second reactor ejected the Oxalic acid yield, temperature, quantity of sulphuric acid, catalyst, additional water and total air flow rate for the two reactors were chosen as parameters. A similar study was carried out for the third reactor as well. After the addition of acid-mixture to the reactor, evolution of brown-colored Nitrogen Oxides was visible immediately that indicates a rapid reaction. . However, it took the reaction in the second

reactor 16 min to give visible gaseous products, and another 45 min period elapsed before any colored gas evolution started in the third reactor.

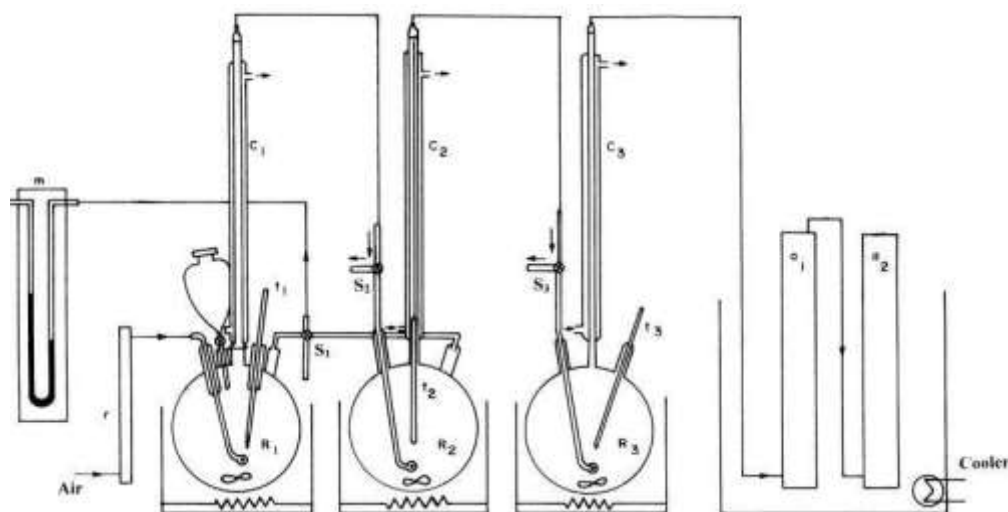


Figure 1. Flow diagram of experimental setup for production of Oxalic acid using nitric acid; a₁; a₂: absorption column; C₁; C₂; C₃: condenser; m: manometer; R₁;R₂;R₃: reactor; r: rot meter; S₁; S₂; S₃: sampling valve; t₁; t₂; t₃: thermometer.

Based on these observations the delay for the start of the reaction in the third reactor was assumed to be 61 min and this was used in the kinetic studies. The reaction was assumed to be completed when the color of the exhaust gases turned pale yellow. The reaction mixture was cooled to about 0°C and Oxalic acid crystals were separated by vacuum. Approximately eighty percent of the filtrate was evaporated gradually in a dish at about 45°C and then it was cooled to 5°C. The needle-shaped crystals of oxalic acid were separated again by vacuum filtration, and crystals were combined with the previous lot. For recrystallization, the crude product was dissolved in hot water, filtered and cooled to 0°C. Then, pure crystals were separated, and the residual filtrate was again evaporated in a dish at about 45°C by the same manner. The saturated solution was cooled to 0°C and filtered. The crystals were dried at about 60°C and weighed. The product was checked by determining the melting point, elemental composition and percent purity.

2.1.2 FERMENTATION BY USING POTTASIAM FERROCYANIDE

Optimum conditions for treatment of molasses sample, which in many respects is similar to the procedure of Eisenman, were found to be as follows: To 340 g of molasses partially diluted is added to 0.60 gm of Potassium Ferrocyanide (in solution), and the whole is made to 1 liter with distilled water. 10g of diatomaceous earth is added and mixed thoroughly. The medium thus prepared is allowed to stand overnight in a graduated cylinder 6°C. The medium is placed in the fermentation containers and autoclaved at 120° C for 15 minutes. It is then kept still for 4 days. The final medium thus prepared contains approximately 15 per cent sugar. A total of four samples of molasses are treated with Ferrocyanide. All of the samples give satisfactory Oxalic acid & citric acid yields, although optimum conditions are established for one sample (No. 1). The Ferrocyanide level, pH, and sterilization conditions required for maximum yields with the different samples is varied considerably. Thus yields obtained with the four samples of molasses are sterilized with the pH unadjusted. Under these conditions, molasses no. 1 will give better results when sterilized, the other three apparently giving better results in unsterile conditions. No trouble is encountered from contamination of the unsterile media in the course of fermentation, presumably because of the high concentration of the acid. Adjusting the pH of another sample (No. 2) before treatment with Ferrocyanide to an optimum (pH 6.0) and sterilizing the medium, helps increase total activity as total yield of citric acid and oxalic acid increases.

2.1.3 PREPARATION OF OXALIC ACID BY MOLASSES USING ASPERGILLUS NIGER

Materials – Microorganism *Aspergillus Niger* NCIM 548 strain used in this study. Medium composition- The medium contained the following components in (g/l): glucose (105.5) or sucrose (100) or lactose (102.6) plus glucose (2.51) - all with the same carbon content; NaNO₃ (1.5); KH₂PO₄ (0.5); MgSO₄*7H₂O (0.025); KCl (0.025); and yeast extract (1.6) Medium pH was adjusted to 6 with 4 M NaOH before sterilization. 20 ml of universal pH indicator solution was added per liter medium for observing its pH which was maintained within 6 to 7 by adding alkali during fermentation.

Spore suspensions -Spores from 7-day stationary culture at 30 °C in potato dextrose broth were suspended in 0.001% (v/v) Triton X-100 solution, and were counted under a light microscope in a Neubauer chamber.

2.1.3.1 Method:

Fifty milliliter culture medium taken in a 250-ml Erlenmeyer flask was inoculated with spores of *Aspergillus Niger* (range: 5 to 6 per ml medium), and was incubated at 30 °C on an orbital shaker at 215 rpm , & for 7 days it was kept in a dry condition . Culture filtrate from each triplicate set was analyzed for oxalic acid content. During fermentation, the pH of the medium was maintained at 6.5 with 4M NaOH. Growth and oxalic acid yield were monitored during the course of fermentation. Broths after fermentation were filtered through a 0.2-mm membrane. Oxalate in the filtrate was estimated by titration with KMnO_4 after precipitating with CaCl_2 . Sugars were estimated calorimetrically using orcinol. Briefly, 1.5 ml of cold orcinol reagent (200 mg orcinol dissolved in 100 ml of 70% (v/v) H_2SO_4 in water) was added to 0.5 ml of diluted sample in a test tube, heated in a boiling water bath for 20 min, and then cooled. Biomass of *Aspergillus Niger* grown under different conditions was measured gravimetrically after separating the mycelia from fermented broth by filtration, washing with water, and drying at 80 °C to constant weight.

2.2 COMPARATIVE STUDY

All of them uses raw material molasses for both the process (lab & industrial). For optimization, so we have to select the best process in term of cost & efficiency of products.

PROCESS	TIME REQUIRED	YIELD (%)
Using Nitric acid	2-3 hours	76
Fermentation Process	3-4 days	52
<i>Aspergillus Niger</i>	7-8 days	62

Table 2- Comparative analysis of all three process producing oxalic acid.

Except the first process all the process involved are fermentation process provide less yield and are time consuming, hence preparation of oxalic acid from molasses using nitric acid is the best method available.

CHAPTER 3: PROCESS DESCRIPTION

Initially molasses was preheated & temperature of it was increased from 37°C to 65.5°C. After that it was fed into a CSTR. Simultaneously Nitric acid was also fed into the CSTR along with Vanadium Penta-oxide which act as a catalyst. This mixture was mixed thoroughly and was allowed to react for 2-3 hours. After 2-3 hours, Oxalic acid, un-reacted molasses, un-reacted Nitric acid, Nitrogen Oxide was formed. Oxalic acid along with un-reacted molasses, un-reacted Nitric acid and Vanadium Penta-oxide comes out from the bottom section of CSTR and undergoes further separation process. During this process Vanadium Penta-oxide gets first get separated out with help of a filter. Oxalic acid & mother liquor (un-reacted Nitric acid & molasses) is separated in a 2 stage process. In the first stage, solution that has been filtered is fed into a crystallizer in which oxalic acid crystals along with mother liquor comes out and further these are separated with help of a centrifuge. After Oxalic acid gets separated, to remove inclusion (process by which a solvent particles get trapped inside a crystal), it is re-crystallized by adding hot water inside a crystallizer containing these Oxalic acid crystals. After separating out the mother liquor again, Oxalic acid crystals are sent into drier to remove the moisture present on the surface of the crystals. Nitrogen Oxide gas which comes out from the top surface of CSTR cannot be discharged directly to the atmosphere, as it can cause air pollution, so this gas was sent into a compressor to increase the pressure & to a steam heater to increase the temperature. After that this gas was sent to fluidized-bed reactor & in presence of Al_2O_3 catalyst, Nitrogen dioxide gas is formed. Al_2O_3 after this get separated by cyclone separator. After this process, in an absorber, Nitrogen dioxide gas was allowed to react with water sprayed inside the absorber to produce Nitric acid & Nitrogen oxide gas. Nitric acid (20%) obtained which is less concentrated than the Nitric acid (95%) which was used as a feed can further be used in other industrial process and Nitrogen oxide gas can be recycled back to stream leading to fluidized bed reactor.

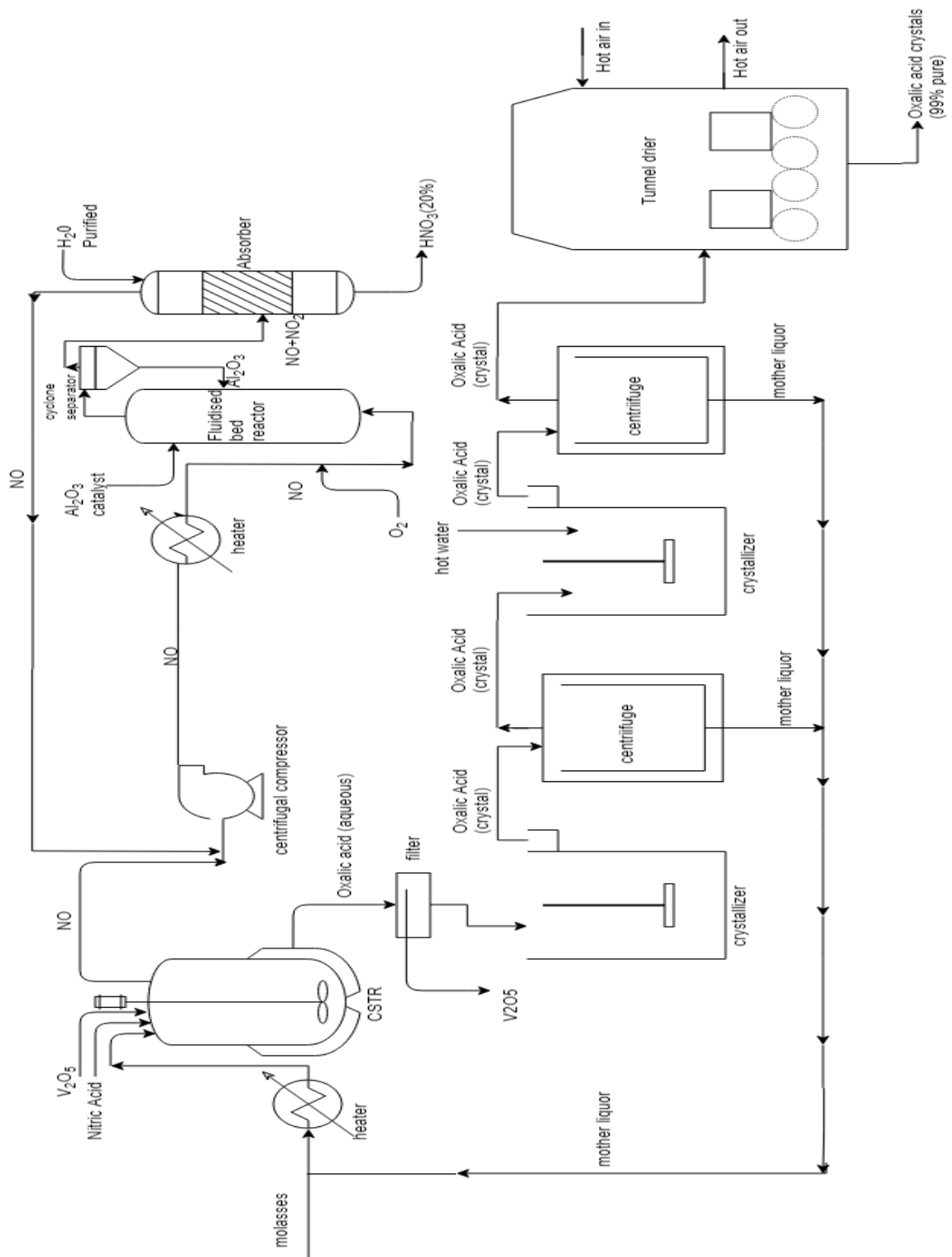


Figure 2: Process Flow Sheet Diagram of Manufacture of Oxalic Acid from Molasses

CHAPTER 4: MASS BALANCE

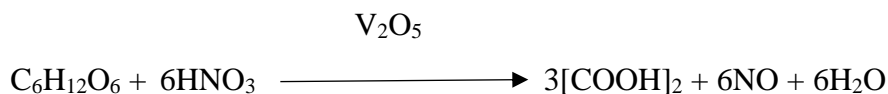
It is desired to produce 1000 kg per hour of Oxalic acid using molasses as the starting material.

4.1 MATERIAL BALANCE FOR THE CSTR

Oxalic acid to be produced = 1000 kg

$$= 11.111 \text{ kmol}$$

Reaction-



1 kmol of Glucose gives 3 kmol of oxalic acid.

Therefore, to produce 11.111 kmol of Oxalic acid we need $= 11.111/3 = 3.7036$ kmol of glucose

Yield is assumed as 76%.

Therefore, glucose taken $= 3.7036/0.76 = 4.8732$ kmol

Therefore, glucose un-reacted $= (4.8732 - 3.7036) = 1.1696$ kmol.

1 kmol of Glucose reacts with 6 kmol of Nitric acid

Therefore, 4.8732 kmol of Glucose reacts with $(4.8732 * 6) = 29.2392$ kmol of Nitric acid

Yield is assumed 76%.

So Nitric acid reacted $= (29.2392 * 0.76) = 22.2219$ kmol

Un-reacted Nitric acid $= (29.2392 - 22.2219) = 7.4074$ kmol

6 kmol water is formed per 1 kmol of glucose reacted.

Hence, the total water produced in the reaction = $(3.7036 \times 6) = 22.2216$

Similarly, nitrogen oxide produce = 22.2216 kmol

Vanadium pentoxide used = 2% of feed (glucose) = 0.0987

kmol

INPUT			OUTPUT		
COMPONEN T	FEED (in kmol)	FEED (in kg)	COMPONEN T	PRODUC T (in kmol)	PRODUC T (in kg)
Glucose	4.8684	877.072	Glucose (un- reacted)	1.1696	210.7058
Nitric Acid	29.239 2	1841.782 3	Nitric acid (un- reacted)	7.4074	466.7466
Vanadium pentoxide	0.0987	17.9634	Water	22.2216	399.9888
			Oxalic Acid	11.111	1000
			Nitric Oxide	22.2216	666.648
			Vanadium pentoxide	0.0987	17.9634
TOTAL		2736.817 7	TOTAL		2762.0526

Table 3: Mass Balance for CSTR

4.2 MATERIAL BALANCE FOR THE FLUIDIZED BED REACTOR

Reaction –



22.2216 kmol of NO gas enters the reactor.

Conversion of NO gas to NO₂ is assumed to be 90%.

So amount of NO reacted = $(22.2216 \times 0.9) = 19.9997$ kmol

Un-reacted NO = 2.2221 kmol

So from the reaction,

1 kmol of NO reacts with 0.5 kmol of Oxygen.

Hence, 19.9997 kmol of NO reacts with = $(19.9997 \times 0.5) = 9.9998 = 10$ kmol of Oxygen.

Considering total consumption of Oxygen gas.

Amount of NO₂ produced from the reaction = 19.9997 kmol

Al₂O₃ used = 0.5% of feed (NO) = 0.111 kmol

INPUT			OUTPUT		
COMPONENT	FEED (in kmol)	FEED (in kg)	COMPONENT	PRODUCT (in kmol)	PRODUCT (in kg)
NO	22.2216	666.65	NO ₂	19.9997	919.985
Oxygen	10	320	Unreacted NO	2.2221	66.663
Al ₂ O ₃	0.111	11.32	Al ₂ O ₃	0.111	11.322
TOTAL		997.97	TOTAL		997.97

Table 4: Mass Balance for Fluidized Bed Reactor

4.3 MATERIAL BALANCE FOR THE ABSORBER

Reaction –



From the reaction from 1 kmol of Nitrogen di-oxide gas gives 0.66 kmol of Nitric acid is produced.

Let the conversion be assumed to be 90%.

Amount of NO₂ entering in to the absorber = 19.9997 kmol.

So amount of NO₂ gas reacted = $(0.9 \times 19.9997) = 17.9997$ kmol

Nitric Acid Produced = $(0.66 \times 17.9997) = 11.8798$ kmol

Un-reacted NO₂ gas = 2 kmol

For 1 kmol NO₂, 0.33 kmol of Water is required for the reaction.

Hence water required = $(17.9997/3) = 5.9999$ kmol

From 3 kmol of NO₂ = 1 kmol of NO gas is produced.

NO gas produced = $(17.9997/3) = 5.99$ kmol.

Un-reacted NO gas in input = 2.2221 kmol

Total NO gas output = 8.2221 kmol

INPUT			OUTPUT		
COMPONENT	FEED (in kmol)	FEED (in kg)	COMPONENT	PRODUCT (in kmol)	PRODUCT (in kg)
NO ₂	19.9997	919.987	Nitric Acid	11.8798	748.427
H ₂ O	5.99	108	NO	8.2221	246.63
Unreacted NO	2.2221	66.663	Unreacted NO ₂	2	92
TOTAL		1087.09	TOTAL		1087.09

Table 5: Mass Balance for Absorber

4.4 MATERIAL BALANCE FOR THE CRYSTALLIZER 1

Initial temperature of Oxalic acid and unreacted mixture = 65.5°C.

Final temperature of Oxalic acid and unreacted mixture =
0°C.

Mass of Oxalic acid and unreacted mixture = 207744.12
kg

Solubility of Oxalic acid at 65.5°C = 80.5mol/100mol of
water.

Solubility of Oxalic acid at 25°C = 0.2 mol/100mol of
water.

$$X_f = 1000/2077.4412 = 0.4814$$

$$X_m = 0$$

Assumption: - Oxalic acid is 100% crystallized and contains impurity of 10% (wt.) water only. Glucose and Nitric acid is completely removed in the mother liquor.

$$X_c = 0.9$$

$$F = C + M$$

$$207744.12 = C + M \dots\dots\dots (1)$$

$$F \cdot X_f = C \cdot X_c + M \cdot X_m$$

$$\text{Equation: } (2077.4412 \cdot 0.4814) = C \cdot 0.9 + (0 \cdot M) \dots\dots\dots (2)$$

Upon solving (1) and (2) we get,

$$C = 1111.2002 \text{ kg and } M = 966.241 \text{ kg}$$

4.5 MATERIAL BALANCE FOR CRYSTALLIZER 2

Initial temperature of Oxalic acid and Water mixture = 90°C.

Final temperature of Oxalic acid = 0°C.

Mass of Oxalic acid crystals = 1111.2002 kg.

Mass of Water added = x kg

Solubility of Oxalic acid at 90°C = 112.6mol/100mol of water.

Solubility of Oxalic acid at 0°C = 0.2mol/100mol of water.

$$X_c = 0.99 \text{ (assumption)}$$

$$X_m = 0$$

$$\text{We know, } F \cdot X_f = C \cdot X_c + M \cdot X_m$$

$$F = C + M$$

Where,

F = Feed

C = Weight of crystal

M = Weight of mother liquor

$$\text{Equation: } 1000.0802 = C \cdot 0.99 + (0 \cdot M)$$

Upon solving, we get,

$$C = 1010.182 \text{ kg and } M = (1010.182 + s \times) \text{ kg}$$

4.6 MATERIAL BALANCE FOR THE CENTRIFUGE 1

Amount of feed entering the centrifuge = 2077.4412 kg

Amount of oxalic acid crystals obtained = 1111.2002 kg

Amount of mother liquor obtained = 966.241 kg

4.7 MATERIAL BALANCE FOR THE CENTRIFUGE 2

Amount of feed entering the centrifuge = $(1111.2002 + x) \text{ kg}$

Amount of oxalic acid crystals obtained = 1010.182 kg

Amount of mother liquor obtained = $(101.0182 + x) \text{ kg}$

4.8 MATERIAL BALANCE FOR THE DRIER

For evaporation of 1 kg of water at 1 atm of pressure, hot air requirement is 10-20 m³

$$= 15 \times 29 = 435 \text{ kg}$$

Therefore, for 111.2002 kg, amount of air needed $435 \times 111.2002 = 48372.087 \text{ kg}$

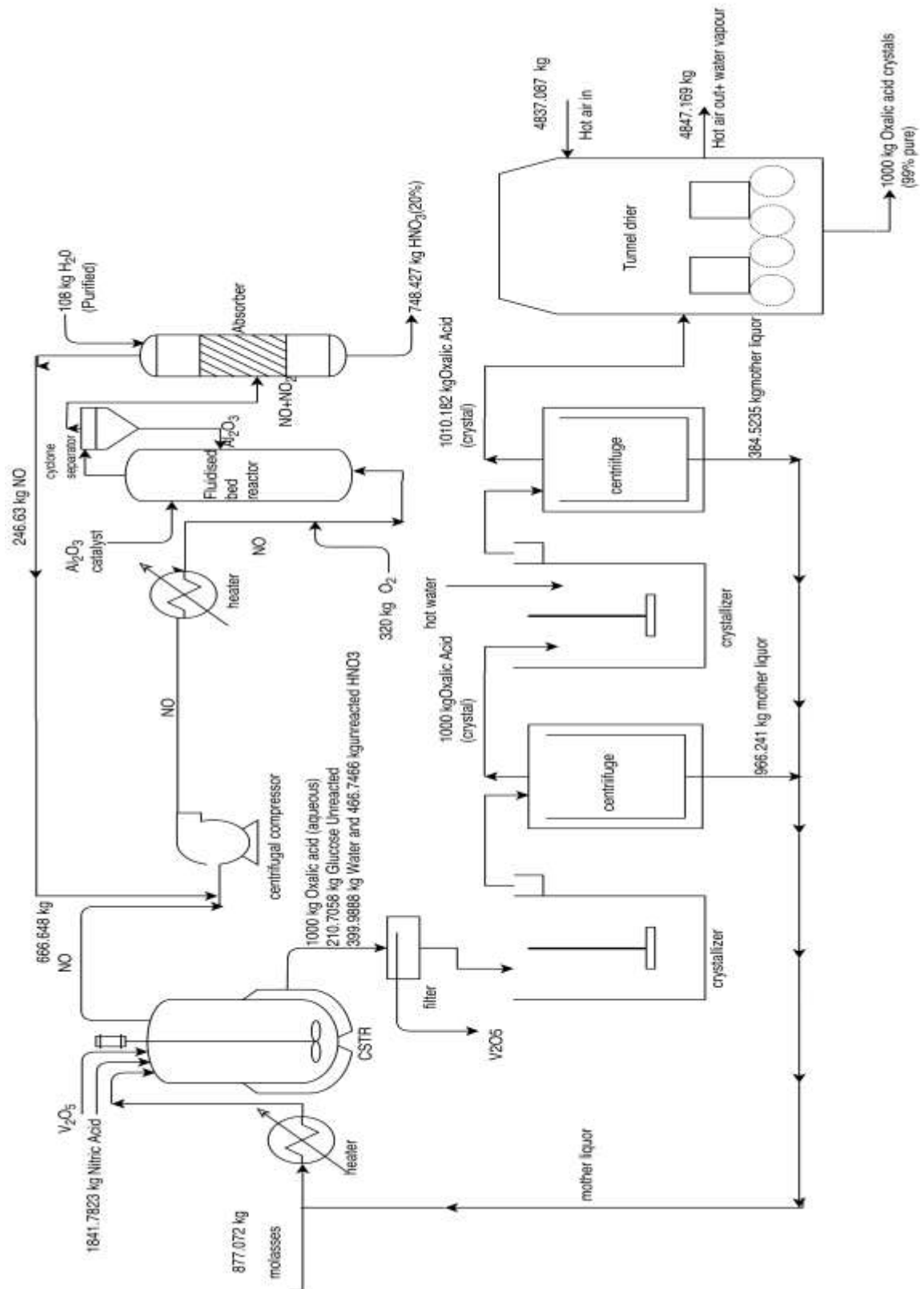


Figure 3: Process Flow Diagram with Mass Balance

CHAPTER 5: ENERGY BALANCE

Component	Glucose	HNO ₃	Oxalic Acid	NO	H ₂ O	O ₂	NO ₂
Specific Heat (C _p) (kJ/kg K)	1.21	1.74	1.61	0.99	4.187	0.92	0.815

Table 6: Specific Heats of Various Components

5.1 ENERGY BALANCE FOR THE STEAM HEATER 1

Initial temperature of molasses = 37°C

Final temperature of molasses = 65.5°C

$$Q = mC_p\Delta t = 877.072 \times 1.21 \times (338.5 - 310) = 30245.828 \text{ kJ}$$

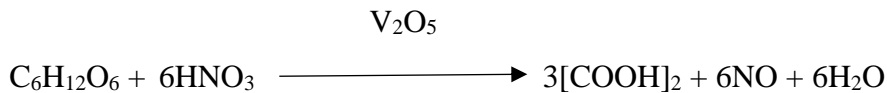
Latent heat of vaporization = $L_v = 2260 \text{ kJ/kg}$

Amount of Water required in steam heater = Q / L_v

$$= 30245.828 / 2260 = \mathbf{13.3831 \text{ kg}}$$

5.2 ENERGY BALANCE FOR THE CSTR

Reaction -



Total Energy of the Reactants

We know $Q = mC_p\Delta t$

$$Q_{\text{glucose}} = 666.3622 \times 1.21 \times 338.5 = 272931.9617 \text{ kJ}$$

$$Q_{\text{HNO}_3} = 1375.0357 \times 1.74 \times 338.5 = 814536.7728 \text{ kJ}$$

$$Q_{in} = 1087.4687 \times 10^6 \text{ J}$$

Total Energy of the Products

$$Q_{\text{Oxalic acid}} = 1000 \times 1.61 \times 338.5 = 544985 \text{ kJ}$$

$$Q_{\text{NO}} = 666.648 \times 0.99 \times 338.5 = 223403.7445 \text{ kJ}$$

$$Q_{\text{H}_2\text{O}} = 399.9888 \times 4.18 \times 338.5 = 565956.1528 \text{ kJ}$$

$$Q_{out} = 1334.34489 \times 10^6 \text{ J}$$

Energy of the CSTR

$$Q = Q_{out} - Q_{in} = 1334.34489 \times 10^6 - 1087.4687 \times 10^6 = 24687.62 \times 10^6 \text{ J}$$

5.3 ENERGY BALANCE FOR THE STEAM HEATER 2

For a fluidized bed reactor, temperature of Nitrogen oxide gas temperature is raised to 85.5°C

Initial temperature of NO Gas = 65.5°C

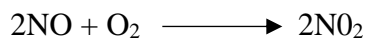
Final temperature NO gas = 85.5°C

$$Q = mC_p\Delta t = 666.648 \times 0.99 \times 20 = 13199.6304 \text{ KJ/hr} = 3.667 \text{ kW}$$

$$\text{Amount of Water required for steam heater} = 13199.6304 / 2260 = 5.8405 \text{ kg/hr}$$

5.4 ENERGY BALANCE FOR THE FLUIDIZED BED REACTOR

Reaction -



Total Enthalpy of the Reactants

$$Q = mC_p\Delta t$$

$$Q_{\text{NO}} = 666.648 \times 0.99 \times 358.5 = 236603.3749 \text{ kJ}$$

$$Q_{\text{O}_2} = 320 \times 0.92 \times 358.5 = 105542.4 \text{ kJ}$$

$$Q_{in} = 342.1458 \times 10^6 \text{ J}$$

Total Enthalpy of the Products

$$Q_{NO} \text{ (unreacted)} = 66.663 \times 0.99 \times 358.5 = 23659.6986 \text{ kJ}$$

$$Q_{NO_2} = 919.985 \times 0.815 \times 358.5 = 268798.9173 \text{ kJ}$$

$$Q_{out} = 292.4586 \times 10^6 \text{ J}$$

Energy for the Fluidized Bed Reactor

$$Q = Q_{out} - Q_{in} = 292.4586 \times 10^6 - 342.1458 \times 10^6 = -49.6872 \times 10^6 \text{ J}$$

5.5 ENERGY BALANCE FOR THE ABSORBER

Reaction –



Total Enthalpy of Reactants

$$Q_{NO_2} = 827.987 \times 0.815 \times 358.5 = 241919.1717 \text{ kJ}$$

$$Q_{H_2O} = 108 \times 4.187 \times 358.5 = 162112.266 \text{ kJ}$$

$$Q_{in} = 241919.1717 \text{ kJ} + 162112.266 \text{ kJ} = 404.03143 \times 10^6 \text{ J}$$

Total Enthalpy of Products

$$Q_{NO} = 180 \times 0.99 \times 358.5 = 63884.70 \text{ kJ}$$

$$Q_{HNO_3} = 748.427 \times 1.74 \times 358.5 = 466861.2783 \text{ kJ}$$

$$Q_{out} = 63884.70 \text{ kJ} + 466861.2783 \text{ kJ} = 530.74597 \times 10^6 \text{ J}$$

Energy for the Absorber

$$Q = 530.7459 \times 10^6 \text{ J} - 404.0314 \times 10^6 \text{ J} = 126.7145 \times 10^6 \text{ J}$$

5.6 ENERGY BALANCE FOR CRYSTALLIZER 1

Heat Input

$$Q_{\text{Oxalic Acid}} = 1000 * 1.61 * 338.5 = 544985 \text{ kJ}$$

$$Q_{\text{H}_2\text{O}} = 399.9888 * 4.187 * 338.5 = 566903.9262 \text{ kJ}$$

$$Q_{\text{Glucose}} = 210.7058 * 1.21 * 338.5 = 86301.9351 \text{ kJ}$$

$$Q_{\text{HNO}_3} = 466.7466 * 1.74 * 338.5 = 274909.08 \text{ kJ}$$

$$Q_{\text{in}} = 1473.09994 \times 10^6 \text{ J}$$

Total Enthalpy of Products

$$Q_{\text{Oxalic Acid}} = 1000 * 1.61 * 273 = 439530 \text{ kJ}$$

$$Q_{\text{H}_2\text{O}} = 399.9888 * 4.187 * 273 = 457207.5978 \text{ kJ}$$

$$Q_{\text{Glucose}} = 210.7058 * 1.21 * 273 = 69602.44691 \text{ kJ}$$

$$Q_{\text{HNO}_3} = 466.7466 * 1.74 * 273 = 221713.9699 \text{ kJ}$$

$$Q_{\text{out}} = 1188.05401 \times 10^6 \text{ J}$$

Energy Change for Crystallizer

$$Q = 1473.09994 \times 10^6 \text{ J} - 1188.05401 \times 10^6 \text{ J} = 285.04593 \times 10^6 \text{ J}$$

5.7 ENERGY BALANCE FOR CRYSTALLIZER 2

Assumption – Heat input = Heat output

Heat change for Oxalic Acid

$$Q_{\text{Oxalic Acid}} = 1000 * 1.61 * (363 - 273) = 144900 \text{ kJ}$$

Heat change for Water

$$Q_{\text{H}_2\text{O}} = (111.2002 + x) * 4.187 * (363 - 273) = (111.2002 + x) * 376.83 \text{ kJ}$$

Total Water to be added:

$$144900 \text{ kJ} = (111.2002 + x) * 376.83 \text{ kJ}$$

$$X = 273.3233 \text{ kg}$$

5.8 ENERGY BALANCE FOR THE DRIER

Amount of Oxalic acid crystal required = 1000 kg

Amount of Oxalic acid fed into the drier = 1010.182 kg

Therefore amount of water evaporated = 1010.182 - 1000 = 10.182 kg

Inlet temperature of feed = 25°C

Let the required temperature of hot air be 't' °C

$$\Delta t = (t - 25) \text{ } ^\circ\text{C}$$

Latent heat of vaporization = 2260 kJ/kg

Therefore heat required to evaporate 10.1802 kg of water:

$$Q = 2260 * 10.182 = 23011.32 \text{ kJ}$$

Specific heat of water = 4.187 kJ/kg °C

But according to energy balance

$$mc_p \Delta t = m\lambda$$

$$\text{Hence, } 10.182 * 4.187 * (t - 25) = 23011.32 \text{ kJ}$$

$$t = 539.765 + 25 = 564.7659 \text{ } ^\circ\text{C}$$

There is no energy change for the centrifuges since no mass change or temperature changes are occurring.

CHAPTER 6: EQUIPMENT DESIGN

6.1 DESIGN OF THE CSTR

$V/F_{A0} = \tau/C_{A0} = (X_A/-r_A)$ - Formula to be used

Where C_{A0} = molar flow rate



Above reaction is 1st order reaction

Where rate of reaction is directly proportional to initial concentration of reactant

So $-r_A = k_1$ [Oxalic acid]

Feed entering: Glucose = 4.8664 kmol = 877.072 kg & Nitric

acid = 29.2392 kmol = 1841.7823 kg

Density of Glucose = 1540 kg/m³

Density of Nitric acid = 1510 kg/m³

Therefore v_1 = volumetric flow rate = $(877.072/1540 + 1841.7823/1510)$ m³/hr = 1.7892 m³/hr

Concentration = (molar flow rate) / (volumetric flow rate)

(Kmole/hr) / (m³/day) = 34.1056 / 1.7892 = 19.03 kmol/m³

$X_A = 0.75$

$C_A / C_{A0} = 1 - X_A = 1 - 0.75 = 0.25$

So $C_A = 0.25 * C_{A0} = 0.25 * 19.03 = 4.76$ Kmole/m³

6.1.1 Rate Constant Calculation

For first order reaction,

$$\ln [C_A] = -kt + \ln [C_{A0}]$$

$$\ln [C_A / C_{A0}] = -kt$$

$$k = \ln [C_{A0} / C_A] / t$$

$$= 1.386 \text{ hr}^{-1}$$

$$\text{Space time } \tau = (C_{A0} - C_A) / k * C_A$$

$$= 14.27 / 1.386 * 4.76$$

$$= 2.16 \text{ hr.}$$

We allow 30% of volume of fluid as the free space in the fermenter. Hence

Total Volume of reactor $V = 1.3 * v_1 = 1.3 * 1.7892 = 2.326$ m³/hr.

6.1.2 Diameter and Length Calculation

$$V = \pi/4 * d^2 * l$$

For CSTR l/d ratio = 3

$$l = 3 * d$$

$$\text{So } d^3 = 2.326 * 4 / \pi * 3$$

$$D = 0.996 \text{ m}$$

$$L = 2.988 \text{ m}$$

6.1.3 Mechanical Design of CSTR

Material used for making the shell is carbon steel.

Thickness of shell, t_s :

$$t_s = [(p D) / (2f J - p)] + c$$

Where,

Inner Diameter of vessel, $D_i = 0.996 \text{ m}$

Working Pressure = $1.013 \times 10^5 \text{ N/m}^2$

Design Pressure, $p = 1.05 \times 1.013 \times 10^5 = 1.063 \times 10^5 \text{ N/m}^2$

Permissible Stress, $f = 95 \times 10^6 \text{ N/m}^2$

$J = \text{Joint Efficiency} = 0.85$

Corrosion allowance, $c = 2 \text{ mm}$

Hence, $t_s = 2.6 \text{ mm}$

Assuming thickness as 8 mm

Thus, outer diameter of shell $D_o = 0.996 \text{ m} + 2 \times 0.008 \text{ m} = 1.012 \text{ m}$

Axial Stress Due to Pressure:

Axial stress due to pressure, f_{ap}

$$\begin{aligned} f_{ap} &= [(p D_i) / \{4(t_s - c)\}] \\ &= 4.411 \times 10^7 \text{ N/m}^2 \end{aligned}$$

6.1.3.1 Stress due to Dead Load:

a) Compressive Stress due to weight of shell up to a distance x

Density of Shell material = $\rho_s = 7700 \text{ kg / m}^3$

$$f_{ds} = [\pi/4 (D_o^2 - D_i^2) \rho_s x] / \pi/4 (D_o^2 - D_i^2) \\ = 7.7 \times 10^4 \text{ X N/m}^2$$

[Where X is the distance measured from top of the vessel]

b) Compressive stress due to weight of insulation at height x:

Insulator used is asbestos

Thickness of insulation $t_{ins} = 3 \text{ cm}$

Diameter of insulation, D_{ins}

Density of insulation = 2200 kg / m^3

Mean diameter of vessel = D_m

For large diameter column

$$D_{ins} = D_m$$

$$f_{dins} = [\pi D_{ins} t_{ins} \rho_{ins} x] / [\pi D_m (t_s - c)] \\ = 0.11 \times 10^6 \text{ X N / m}^2$$

c) Compressive stress due to liquid in column up to height x:

Density of liquid, $\rho_l = 1519.59 \text{ kg/m}^3$

$$f_{dliq} = [(\pi/4) D_i^2 \times \rho_l] / [\pi D_m (t_s - c)] \\ = 0.625 \times 10^6 \text{ XN/m}^2$$

6.1.3.2 Stress due to Wind:

Stress due to wind is given by

$$f_{wx} = M_w / Z$$

Where,

$$M_w = [p_w X/2]$$

$$Z = [(\pi/4) D_o^2 (t_s - c)] = 0.4824 \times 10^{-3}$$

$$\text{Pressure due to wind} = p_w = 0.05 \text{ X } v_w^2$$

Considering velocity of wind to be 150 km/hr

$$p_w = 1125 \text{ XN /m}^2$$

$$f_{wx} = \{2 p_w X^2\} / \{\pi D_o^2 (t_s - c)\}$$

$$= 1.166 \times 10^6 X^2 \text{ N}$$

Determining the value of X:

$$f_{tmax} = 95 \times 10^6 \text{ N/m}^2$$

$$f_{tmax} = f_{wx} + f_{ap} - f_{dx}$$

$$95 \times 10^6 (0.85) = 1.166 \times 10^6 X^2 + 4.052 \times 10^6 - [7.7 \times 10^4 X + 0.11 \times 10^6 X + 0.625 \times 10^6 X]$$

Gives, $X = 5.93 \text{ m}$

As this value is greater than the design reactor height, hence the shell thickness
Can withstand the tensile and compressive stresses.

6.1.4 Design of the Cooling Water Jacket

Considering its height = $0.9 \times \text{height of CSTR} = h_j = 2.6892 \text{ m}$

So assuming volume of water needed = $0.1 \text{ m}^3/\text{hr}$

As the jacket surrounds the CSTR. So, it is assumed as hollow cylinder

$$\pi * h * (R_{22} - R_{12}) = 0.1 \text{ m}^3$$

R_1 is radius of CSTR = 0.483 m

$$R_{22} - 0.483 = 0.1 / (\pi * 2.988)$$

$$R_2 = 0.4936 \text{ m}$$

So annular thickness of jacket = $R_2 - R_1$

$$= 0.4936 - 0.483$$

$$= 0.01 \text{ m}$$

(Which is a permeable thickness of jacket)

6.2 DESIGN OF CRYSTALLIZER

Let v_1 = volume of crystallizer.

Sp_1 = Specific gravity of solution entering to the crystallizer.

Now for a crystallizer,

$$Sp_1 = ((m_1) * (m_2)) / (E(m_2) + (1-E)(m_1))$$

Where, E= efficiency of crystallizer.

m1 = Total mass of solution

m2 = Total mass of the component that needs to be crystallized.

Considering E= 80%

$$Sp_1 = 2077.4412 * 1000 / (0.8 * 1000) + (0.2 * 2077.4412) = 1709.1413$$

$$\text{Quality} = \text{mass of crystal obtained} / (1-E) = 1111.2002 / 0.2 = 5556.001 \text{ m}^3$$

$$\text{So volume } v_1 = \text{quality} / 6.24(sp_1) = 5556.001 / 6.24 * 1709.1413 = 0.5209 \text{ m}^3$$

Taking 30% clearance for the volume,

$$\text{Volume required for the crystallizer} = 1.3 * v_1 = 1.3 * 0.5209 = 0.67717 \text{ m}^3$$

6.2.1 Length and Diameter Calculation

$$V_1 = \pi * D^2 * H / 4$$

Where D = diameter, H = height.

$$0.5209 = 3.14 * D^2 * H / 4$$

For a crystallizer = H = 2.5D

$$0.67717 = 3.14 * (H/2.5)^2 * (H * 0.25)$$

$$\Rightarrow H = 5.3914 \text{ m}$$

$$\text{So } D = 5.3914 / 2.5 = 2.1565 \text{ m}$$

6.3 DESIGN OF FLUIDIZED BED REACTOR

Diameter of catalyst particle = 40 μm

Assuming particles are spherical, sphericity = 1

Density of gas = $p_g = 1.37 \text{ kg/m}^3$

Density of catalyst = 3950 kg/m^3

Viscosity = $2.44 * 10^{-5} \text{ Pa.s}$

Porosity at minimum fluidization velocity = e_{mf}

$$\varepsilon_{mf} = 0.586\psi^{-0.7} \left(\frac{\mu^2}{\rho_g \eta d_p^3} \right)^{0.029} \left(\frac{\rho_g}{\rho_c} \right)^{0.021}$$

$$e_{mf} = 0.77$$

Minimum Fluidization velocity = U_{mf}

$$u_{mf} = \frac{(\psi d_p)^2}{150\mu} \underbrace{[\rho_c - \rho_g]}_{\eta} \frac{\varepsilon_{mf}^3}{1 - \varepsilon_{mf}}$$

$$U_{mf} = 0.034 \text{ m/s}$$

u_t is maximum fluidization velocity

$$u_t = \begin{cases} \frac{\eta d_p^2}{18\mu} & Re < 0.4 \\ \left(\frac{1.78 \times 10^{-2} \eta^2}{\rho_g \mu} \right)^{1/3} d_p & 0.4 < Re < 500 \end{cases}$$

$$U_t = 0.081 \text{ m/s}$$

6.4 DESIGN OF STEAM HEATER

Feed gas:

$$NO = 22.2216 \text{ Kmoles/hr}$$

$$= 666.648 \text{ Kg/hr}$$

Entering at 65.5°C, Pressure is 1 atm.

Flow rate, $m = 0.1852 \text{ Kg/s}$

$$Q' = 3.667 \text{ KW}$$

Using saturated steam as heater, flow rate of water required,

$$m_w = 5.8504 \text{ Kg/hr.}$$

For a counter current operation, LMTD:

$$\Delta T_{ln} = 23.07^\circ\text{C}$$

Calculation of Heat Transfer Area:

Assuming heat transfer coefficient,

$$U = 300 \text{ W/m}^2\text{-K}$$

Dirt factor = $0.003 \text{ ft}^2\text{h-K/Btu}$

Heat transfer Area, $A = 0.53 \text{ m}^2$

Choosing $\frac{3}{4}$ " OD, 8 ft long tubes,

H.T.A for one tube = 0.14593 m^2

$N_t = 60$ tubes

Choosing a 1-2 Heat exchanger, TEMA P or S type $15/16$ " triangular pitch,

Nearest tube count = 62 tubes

Shell I.D = 254 mm

Corrected heat transfer area, $A = 9.008 \text{ m}^2$

Corrected $U = 17.65 \text{ W/m}^2\text{-K}$

Fluid Velocities:

a) Tube side: Number of passes, $N_p = 2$

Flow area, $a_t = 6.04 \times 10^{-3} \text{ m}^2$

Tube side velocity, $v_t = 0.85219 \text{ m/s}$

For the tube side the acceptable velocity range is 1-2 m/s. Hence, this velocity is unacceptable.

Trial 2:

Choosing TEMA P or S type for 1 shell and 4 tube passes, 1" triangular pitch.

$N_t = 62$

Shell I.D = 305 mm

$v_t = 1.704 \text{ m/s}$, which is within the acceptable limits.

$a_t = 3.02 \times 10^{-3} \text{ m}^2$

b) Shell side:

Ds: Shell I.D

Cross flow area at the center of the shell, S_m :

$$S_m = [(P' - D_o) L_s] D_s / P'$$

$$= 0.01159 \text{ m}^2$$

Taking 25% baffle cut, $L_c = 76.25 \text{ mm}$

$$L_s = 0.5 D_i = 152.5 \text{ mm}$$

$$N_b + 1 = 16$$

$$N_b = 15 \text{ baffles}$$

6.4.1 Summary of shell and tube details:

Number of tube passes = 4

Number of shell passes = 1

Tube I.D = 15.75 mm

Tube O.D = 19.05 mm

Shell I.D = 305 mm

Number of tubes = 62

Tube pitch: 1" = 25.4 mm

Length of tube = 2.348 m

Baffle pitch: 152 mm

Number of baffles: 15

P_p : 21.99 mm

L_c : 76.25 mm

$U_{\text{corrected}}$: 17.65 W/m²-K

Film Transfer Coefficients:

a) Shell side (condensing vapors):

NO is heated from 65.5°C to 85.5°C

Wall temperature, $T_w = (1/2) [100 + (65.5 + 85.5)/2] = 87.75^\circ\text{C}$

Film temperature, $T_f = (100 + 87.75)/2 = 93.85^\circ\text{C}$

Using properties of steam at this temperature,

Thermal conductivity, $K = 0.15427 \text{ W/m-K}$

Density = 963.30 Kg/m³

Viscosity = 0.28 cP

Reynold's number (N_{Re}) = 164.67

$$h = 1.51 [(K^3 \rho^2 g) / \mu]^{1/3} Re^{-1/3}$$

$$= 60 \text{ W/m}^2\text{-K}$$

b) Tube side (NO):

Flow area, $a_t = 3.02 \times 10^{-3} \text{ m}^2$

Density $\rho = 1.34 \text{ Kg/m}^3$

Viscosity $\mu = 0.1 \text{ cP}$

Thermal conductivity, $K = 0.03 \text{ W/m-K}$

Prandtl's number (N_{Pr}) = 3.3167

$$h_i d_i / K = 0.023 (N_{Re})^{0.8} (N_{Pr})^{0.3}$$

Hence, $h_i = 6.958 \text{ W/m}^2\text{-K}$

$$\begin{aligned} \text{Wall resistance} &= (x_w / k_w) \times [d_o / (d_i - d_o)] \times \ln(d_o / d_i) \\ &= 4.02646 \times 10^{-5} \end{aligned}$$

Overall Film Heat Transfer Coefficient:

$$U_{od} = 6.2344 \text{ W/m}^2\text{-K}$$

6.5 DESIGN OF DRYER

Assuming Air humidity = 0.015

Air inlet temperature = 565°C

Maximum Solid Temperature = 150°C

Maximum air velocity = 1.5m/s

Feed inlet = 1010.82 kg/hr

Specific heat of solid = 1.61 kJ/Kg K

Basis: 1hr

Mass of dry solid = $L_s = 1000 \text{ kg}$

Moisture in wet solid = $10.182 / 1000 = 0.011 = X_1$

$$\text{Moisture in dry solid} = 0.001 = X_2$$

$$\begin{aligned}\text{Water evaporated} &= m_s = L_s * (X_1 - X_2) \\ &= 1000 * (0.011 - 0.001) \\ &= 10 \text{ kg}\end{aligned}$$

$$T_{s1} = 25^\circ\text{C} \quad T_{G2} = 565^\circ\text{C} \quad y_2 = 0.015$$

Assumptions:

- 1) Exit gas temperature = $T_{G1} = 200^\circ\text{C}$
- 2) Exit solid temperature = $T_{S2} = 140^\circ\text{C}$
- 3) $T_{\text{ref}} = 0^\circ\text{C}$

$$\begin{aligned}H_{s1}^1 &= (C_{ps} + 4.187 X_1) * (T_{s1} - T_{\text{ref}}) \\ &= 41.4014 \text{ kJ/kg of dry solid}\end{aligned}$$

$$\begin{aligned}H_{s2}^1 &= (C_{ps} + 4.187 X_2) * (T_{s2} - T_{\text{ref}}) \\ &= 22.9861 \text{ kJ/ kg of dry solid}\end{aligned}$$

$$\begin{aligned}H_{G1}^1 &= (C_{p\text{AIR}} + C_{p\text{MOISTURE}} * y_2) * (T_{\text{hot air}}) + (y_2 * 2500) \\ &= (1.006 + 1.88 * 0.015) * 565 + 0.015 * 2500 \\ &= 621.258 \text{ kJ/kg}\end{aligned}$$

$$\begin{aligned}H_{G1}^1 &= (C_{p\text{AIR}} + C_{p\text{MOISTURE}} * y_1) * (T_{\text{hot air}}) + (y_1 * 2500) \\ &= 201 + 2876 * y_1\end{aligned}$$

Overall Mass Balance:

$$\begin{aligned}G_s * (y_1 - y_2) &= L_s * (X_1 - X_2) \\ G_s * (y_1 - 0.015) &= 1000 * (0.011 - 0.001) \\ \text{Hence, } G_s &= 10 / (y_1 - 0.015)\end{aligned}$$

Overall Energy Balance:

$$L_s * (H_{S2} - H_{S1}) = G_s * (H_{G2} - H_{G1})$$

$$1000 * (225.986 - 41.4014) = [10 / (y_1 - 0.015)] (621.258 - 201 - 2876 * y_1)$$

$$18458.47 * y_1 - 276.877 = 420.258 - 2876 * y_1$$

$$y_1 = 0.0326$$

$$G_s = 568.1818 \text{ kg/hr}$$

Calculation of Shell Diameter

Humid Volume

$$v_H = \left(\frac{1}{28.97} + \frac{1}{18.02} \right) \times 22.4 \times \left(\frac{T_G + 273}{273} \right) \text{ m}^3/\text{kg dry air}$$

For inlet gas, $V_{H2} = 2.4414 \text{ m}^3/\text{kg dry air}$

For exit gas, $V_{H1} = 1.416 \text{ m}^3/\text{kg dry air}$

Maximum volume of gas flow rate (at end 2)

$$\begin{aligned} &= G_s * V_{H2} \\ &= 568.1818 * 2.4414 \\ &= 1387.159 \text{ m}^3/\text{hr} \end{aligned}$$

Maximum superficial air velocity = $0.8 * (\text{max. allowable velocity})$

$$= 0.8 * 1.5 = 1.2 \text{ m/s}$$

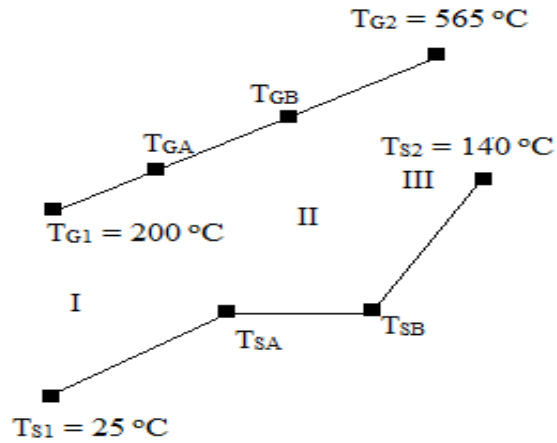
$$(\pi/4) * d^2 * 1.2 = 1387.159 / 3600$$

$$d^2 = 0.409$$

$$d = 0.6395 \text{ m}$$

For clearance, select diameter = 0.7m

$$\text{Length of Dryer} = L = N_{tg} * L_t$$



Zone III

Taking boundary between I and II

Solid is at T_{sB}

Assume $T_{sA} = 80^\circ\text{C} = T_{sA}$ (needs to be checked)

$$\begin{aligned} H_{sB}^I &= (C_{ps} + 4.187 X_1) * (T_{sB} - T_{ref}) \\ &= 129.135 \text{ kg/ kg of dry air} \end{aligned}$$

Humid heat of gas entering zone II

$$C_{HB} = (1.005 + 1.88 * 0.015) = 1.033 \text{ kJ/kg K [remains constant in zone III]}$$

Heat balance for zone III

$$\begin{aligned} L_s * (H_{s2} - H_{sB}) &= G_s * (C_{HB})_{III} * (T_{G2} - T_{GB}) \\ 1000 * (225.9861 - 129.135) &= 568.1818 * (565 - T_{GB}) \\ T_{GB} &= 394.5421^\circ\text{C} \end{aligned}$$

$$\Delta T_B = 310.5421^\circ\text{C}$$

$$\Delta T_2 = 425^\circ\text{C}$$

$$\Delta T_{LMTD,III} = (425 - 310.5421) / \ln(425/310.542) = 364.7827^\circ\text{C}$$

$$\begin{aligned}(\text{Ntg})_{\text{III}} &= (565-394.5421)/364.7827 \\ &= 0.5672\end{aligned}$$

Zone II

Heat Balance for Zone II

$$L_s^*(H_{\text{SB}} - H_{\text{AS}}) = G_s^*(H_{\text{GB}} - H_{\text{GA}})$$

$$H_{\text{GB}} = (1-005 + 1.88Y_{\text{B}}) (394.5421) + (2500Y_{\text{B}})$$

$$Y_{\text{B}} = 0.015$$

$$\text{Hence, } H_{\text{GB}} = 445.141 \text{ kJ/kg}$$

$$H_{\text{AS}} = (1.61 + 4.187*0.011) (80) = 132.4845 \text{ kJ/kg}$$

Solving the heat balance,

$$H_{\text{GA}} = 439.245 \text{ kJ/kg} = (1.005 + 1.88*0.0326) (T_{\text{GA}}) + (0.0326*2500)$$

$$T_{\text{GA}} = 335.6^\circ\text{C}$$

$$\Delta T_{\text{A}} = 255.6^\circ\text{C}$$

$$\Delta T_{\text{B}} = 310.5421^\circ\text{C}$$

$$\Delta T_{\text{LMTD,II}} = (310.5421 - 255.6)/ \ln(310.542/255.6) = 282.18^\circ\text{C}$$

$$\begin{aligned}(\text{Ntg})_{\text{II}} &= (394.5421-335.6)/282.18 \\ &= 0.5088\end{aligned}$$

Zone I

$$\Delta T_1 = 200-25 = 175^\circ\text{C}$$

$$\Delta T_{\text{A}} = 255.6^\circ\text{C}$$

$$\Delta T_{\text{LMTD,II}} = (255.6-175)/ \ln(255.6/175) = 199.2302^\circ\text{C}$$

$$\begin{aligned}(\text{Ntg})_{\text{I}} &= (335.6-200)/199.2302 \\ &= 0.6806\end{aligned}$$

$$\text{Total (Ntg)} = 0.6806 + 0.5088 + 0.5672 = 1.7566$$

Length of Transfer Unit

$$L_T = (G' C_H / u_a)$$

G' = average gas mass flow rate

$$= [568.1818(1.015+1.0326)]/2 = 581.7045 \text{ kg/hr}$$

$$G' = (581.7045/3600)/(3.14*(0.7)^2)/4 = 0.42 \text{ kg/m}^2\text{s}.$$

$$u_a = 237*(G')^{0.67}/D_s = 189.33 \text{ W/m}^3\text{K}$$

C_H = Average Humid Heat

$$C_{H1} = 1.005 + (1.88*0.0326) = 1.0662 \text{ kJ/kgK}$$

$$C_{H2} = 1.005 + (1.88*0.015) = 1.0332 \text{ kJ/kgK}$$

$$C_H = 1049.7 \text{ J/kgK}$$

$$L_T = 0.42*1049.7/189.33 = 2.3286\text{m}$$

$$\textbf{Length of Dryer} = 1.7566*2.3286 = 4.1\text{m}$$

CHAPTER 7: SITE SELECTION AND PLANT LAYOUT

7.1 SITE SELECTION

The location of the plant can have a turning effect on the overall viability of a process plant, and the scope for future expansion. Many factors must be considered when selecting a suitable plant site. The most important factors are as follows:

1. Location, with respect to the marketing area.
2. Raw material supply.
3. Transport facilities.
4. Availability of labour.
5. Availability of suitable land.
6. Environmental impact and effluent disposal.
7. Local community consideration.
8. Climate
9. Political and strategic consideration.

Location with Respect to marketing area:

For materials that are produced in bulk quantities where the cost of the product per tonne is relatively low and the cost of transport a significant fraction of the sales price, the plant should be located close to the primary market. This consideration will be less important for low volume production, high-priced products like Pharmaceuticals.

Raw materials:

The availability and price of suitable raw materials will often determine the site location. Plants producing bulk chemicals are best located close to the source of the major raw material where this is also to the marketing area.

Transport:

The transport of materials and products to and from the plant will be an overriding consideration in site selection. If practicable, a site should be selected such that is close to at least two major forms of transport: road, rail, waterway (canal or river), or a sea port. Road transport is being increasingly used, and is suitable for long distance transport of bulk

chemicals. Air transport is convenient and efficient for the movement personnel and essential equipment and supplies, and the proximity of the site to a major airport should be considered.

Availability of labour:

Labour will be needed for construction of the plant and its operation. Skilled construction workers will usually be brought in from outside the site area, but there should be an adequate pool of unskilled labour availability locally and labour suitable for training to operate the plant. Skilled tradesmen will be needed for plant maintenance. Local trade union customs restrictive practices will have to be considered when assessing the availability and suitability of the local labour for recruitment and training.

Availability of Utilities:

Chemical processes invariably require large quantity of water for cooling and general process use, and the plant must be located near a source of water of suitable quality. Process water may be drawn from a river, from wells, or purchased from a local authority. At some sites, the cooling water required can be taken from a river or lake, or from the sea; at other locations cooling towers will be needed.

Electrical power will be needed at all sites. Electrochemical processes that require large quantities of power; need to be located close to a cheap source of power. A competitively priced fuel must be available on site for steam and power generation.

Environmental Impact and Effluent Disposal:

All industrial processes produce waste products, and full consideration must be given to the difficulties and cost of their disposal. The disposal of toxic and harmful effluents will be covered by local regulations, and the appropriate authorities must be consulted during the initial site survey to determine the standards that must be met. An environmental impact assessment should be made for each new project or major modification or addition or an existing process.

Local community considerations:

The proposed plant must be fit in with and be acceptable to the local community. Full consideration must be given to the safe location of the plant so that it does not impose a significant additional risk to the community. On a new site, the local community must be able to provide adequate facilities for the plant personnel: Schools, banks, housing and recreational and cultural facilities.

Availability of land:

Sufficient suitable land must be available for the proposed plant and future expansion. The land should ideally be flat, well drained and have suitable load-bearing characteristics. A full site evaluation would be made to determine the need for piling or other special foundations.

Climate:

Adverse climatic conditions at a site will increase costs. Abnormally low temperature will require the provision of additional insulation and special heating for equipment and pipe runs. Stronger structures will be needed at locations subject to high winds (cyclones, hurricanes) or earthquakes.

Political and Strategic Considerations:

Capital grants, Tax concessions, and other inducements are often given by governments to direct new investment to preferred locations such as areas of high employment. The overriding of such grants can be the overriding considerations in site locations.

In addition to the main plant, we also have to consider the associated services which have to be amalgamated within a particular plant site. Canteens, parks, general utilities, emergency medical services and places for storage must also be taken into consideration while deciding on a particular site.

Actual site selection:

The plant has to be located at a place where cost of raw materials is less. Another factor which has to be considered for product in vicinity of production area.

Availability of Raw materials:

The huge reserves of Natural gas at the Krishna river Basin in Andhra Pradesh, provides a suitable source of our major raw material.

Availability of transport, labour & Utilities:

As the area is itself an industrial area, so infrastructure for all these is sufficient. Cheap labour is easily available. Power and other utilities are also available at a reduced rate.

Availability of Land:

The area being an industrial areas, the procurement of land should not be a problem.

7.2 PLANT LAYOUT

The economic construction and operation of a process unit will depend on how well the plant equipment specified on the process flow sheet and laid out. The principal factors to be considered are:

1. Economic considerations, construction and operation cost.
2. The process requirement.
3. Convenience of operation.
4. Convenience of maintenance
5. Safety
6. Future Expansion

Costs:

The cost of construction can be minimized by adopting a layout that gives shortest run of connecting pipes between equipment and adopting the least amount of structural steel work. However, this will not necessarily be the best arrangement for operation and maintenance.

Process requirement:

All the required equipment have to be placed properly within process. Even the installation of the auxiliaries should be done in such a way that it will occupy the least space.

Operations:

Equipment that needs to have frequent operation should be located convenient to the control room. Valves, sample points, and instruments should be located at convenient position and height. Sufficient working space and headroom must be provided to allow easy access to equipment.

Maintenance:

Heat exchangers need to be sited so that the tube bundles can be easily withdrawn for cleaning and tube replacement. Vessels that require frequent of catalyst or packing should be located on the outside of buildings. Equipment that requires dismantling for maintenance, such as compressors and large pumps, should be placed under cover.

Safety:

Blast walls may be needed to isolate potentially hazardous equipment, and confine the effects of an expansion. At least two escape routes for operator must be provided from each level in the process building.

Plant expansion:

Equipment should be located so that it can be conveniently tied in with any future expansion of the process. Space should be left on pipe alleys for future needs, service pipes oversized to allow for future requirements.

Main selection:

A complex chemical plant is divided into selection to limit the capital at risk; some of these areas should be located in areas where there is no fire risk. Care should be taken to give enough vacant space to avoid a chain reaction during the times of fire any other emergencies.

Pipe Bridges:

Equipment can be under pipe bridges but avoid large inventories of flammable materials or say pumps, moving liquids above atmospheric temperature. Enough bends should be given for the lines carrying hot liquids; otherwise, thermal expansion may damage the lines.

Loading and Off-loading:

Locate the loading and off loading facilities at the terminal parts of the plant so that traffic through the plant is minimized. Road tanker loading or offloading should be classified as danger areas and should be located at the periphery of the plant. These facilities should be so located that they are close to the railway terminal.

Plant roads:

Plant roads should give access to plants and should be arranged so that vehicles do not pass through process area. Dead end roads should be avoided while access from any point to any part of the plant should be from two directions. Hazardous areas should not overlap the plant limits.

Nomenclature of the Plant Layout shown in Figure:

1. Administrative Block
2. Fire & Safety Department
3. Plant Utilities
4. Emergency Water
5. Packaging Plant
6. Process Plant
7. Expansion
8. Laboratory
9. Store
10. Stray Yard
11. Canteen
12. Storage Vessels
13. Workshops

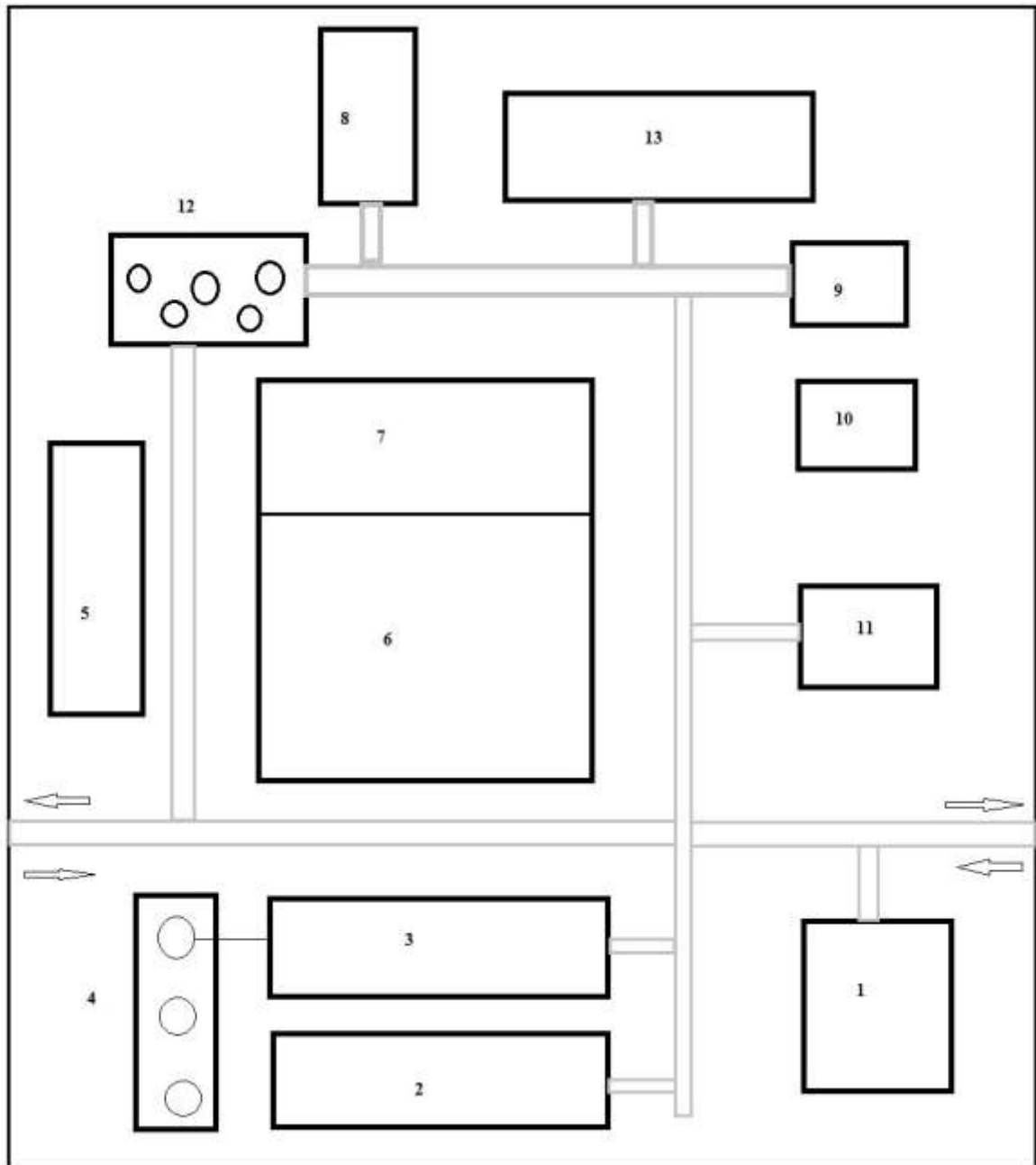


Figure 5: Plant Layout

CHAPTER 8: COST ESTIMATION

No plant and process design is complete without a cost analysis of the project, which gives in addition to the cost of the produce, also an idea of the initial investment needed to start and run the plant. The ultimate aim of any plant is the maximization of profit in relation to the investment employed. This can be achieved by a careful study of the market and critical estimation of the cost. The total amount invested is employed for purchasing or hiring of the following, land and building, machinery and equipment, raw materials. Labour, Supervisors and administrators, etc. of this land, machinery etc. are classified as **fixed capital** and raw materials, power, wages etc., are classified as **working capital**.

The income to the company is by the sales of the product at a price would fetch a good profit and at the same time is capable of competing with other companies.

8.1 PURCHASED EQUIPMENT COST

Equipment	Cost
Reactor Vessel	17,02,000
Steam Heaters (2)	3, 84,000
Centrifugal Compressor	60, 00,000
Absorption Column	20, 00,000
Storage Tanks	53, 00,000
Fluidized bed reactor	20, 50,000
Crystallizer (2)	10, 00,000
Centrifuges (2)	15, 00,000
Dryer	15, 00,000
Filter	8, 00,000
TOTAL	2, 22, 36,000

8.2 DIRECT FIXED COST

Details	Cost (Rs.)
Equipment installation cost including insulating cost and painting (30% of cost total equipment cost).	66, 70,800
Instrumentation and control (20% of total equipment cost)	44, 47,200
Electrical equipment (15% of total equipment cost)	33, 35, 400
Building cost (50% of land cost)	1, 95, 09,500
Piping cost (20% of total equipment cost)	44, 47, 200
TOTAL	3, 84, 10, 100

8.3 INDIRECT FIXED COST

Details	Cost (Rs.)
Engineering and Supervision (30% of total equipment cost)	66, 70,800
Construction cost (10% of direct fixed cost)	38, 41, 010
Contractors costs (5% of direct fixed cost)	19, 20,505
Contingency(8% of direct fixed cost)	30, 72, 808
TOTAL	1, 55, 05,426

8.4 VARIABLE COST

Raw Materials	Annual Requirements (kg)	Cost (Rs.)
Molasses	7683150.72	76,831,507
Nitric Acid	16186572.9	1,618,657,290
Catalyst	157359.384	118,019,538
TOTAL		1,81,35,08,340

8.5 UTILITIES

Utilities	Cost (Rs.)
Cooling Water	7,202,000
Steam	63,759,000
Power requirement (Electricity)	11,000,000
Cooling Air	9,966,000
TOTAL	91,927,000

8.6 WORK FORCE

Work Force	No.	Salary per month	Salary per annum (lacs)
1. Chief executive	2	30,000	7.20
2. Plant manager	2	20,000	4.80
3. Assistant plant manager	5	16,000	9.60
4. Supervisor	20	15,000	36.00
5. Shift engineer (chemical)	5	18,000	10.80
6. Shift engineer (mechanical)	4	18,000	8.64

7. Chief chemist	1	15,000	1.80
8. Laboratory chemist	3	12,000	4.32
9. Operators	10	10,000	12.00
10. Lab inspectors	1	9,000	1.08
11. Accountant	1	16,000	1.92
12. Store keeper	1	10,000	12.00
13. Clerk	4	9,000	4.32
14. Typist	3	2,500	0.90
15. Skilled workers	30	5,000	18.00
16. Unskilled workers	30	1,500	5.40
17. Watchman	3	1,200	0.43

Total labour cost = Rs. 139.21 lakhs

Total operating cost per annum = Rs. 60 lakhs

Maintenance per annum = Rs. 20 lakhs

Supervision and labour cost = Rs. 4 lakhs (15% of operating cost)

8.7 DEPRECIATION:

Plant life = 10 years

Amount of straight line depreciation = $(P-L)/n$

Where,

P = total equipment cost

L = salvage value (10% of total purchased equipment)

N = no of years

$$\begin{aligned}\text{Straight line depreciation amount} &= (2,22,36,000 - 22,23,600)/10 \\ &= \text{Rs. } 20,01,240\end{aligned}$$

$$\begin{aligned}\text{Building cost} &= 3\% \text{ of initial building cost} \\ &= \text{Rs. } 5,85,285\end{aligned}$$

$$\begin{aligned}\text{Depreciation value} &= \text{Rs. } 20,01,240 + 5,85,285 \\ &= \text{Rs. } 25,86,525\end{aligned}$$

Taxes:

$$\begin{aligned}\text{Local tax} &= 3\% \text{ of total fixed capital investment} \\ &= 0.03 \times 76,151,526 \\ &= \text{Rs. } 2,284,546\end{aligned}$$

$$\begin{aligned}\text{Insurance (1\% of TFCI)} &= 0.01 \times 76,151,526 \\ &= \text{Rs. } 761,515\end{aligned}$$

$$\begin{aligned}\text{Plant overhead} &= 50\% \text{ of operating labour cost} + \text{maintenance} + \text{supervision} \\ &= 50\% \text{ of } 60 \text{ lakhs} + \text{Rs. } 20 \text{ lakhs} + \text{Rs. } 4 \text{ lakhs} \\ &= \text{Rs. } 54 \text{ lakhs}\end{aligned}$$

$$\begin{aligned}\text{Total taxes} &= \text{local taxes} + \text{insurance} + \text{plant overhead} \\ &= 2,284,546 + 761,515 + 5,400,000 \\ &= \text{Rs. } 8,446,061\end{aligned}$$

$$\text{Total product cost per annum} = \text{amount of product per batch} \times \text{no of batches}$$

$$\text{Produced in a day} \times \text{price of product/ kg} \times 365 \text{ days.}$$

$$\begin{aligned}\text{Cost of Oxalic acid} &= 24000 \times 365 \times 150 \\ &= \text{Rs. } 1.314 \times 10^9\end{aligned}$$

8.8 GENERAL EXPENSES

Expenses	Cost (Rs.)
Administrative cost (15% of operating cost)	9,00,000
Distribution and marketing cost (5% of product cost)	6,57, 00,000
R & D cost (5% of operating cost)	3,00,000
TOTAL	6,69,00,000

$$\begin{aligned}\text{Total variable cost} &= \text{manufacture cost} + \text{utilities cost} + \text{labour costs} + \\ &\quad \text{Maintenance cost} + \text{supervision and labour cost} + \\ &\quad \text{General expenses} \\ &= 18135.08 + 919.27 + 139.21 + 20 + 4 + 76.06 + 669\end{aligned}$$

$$\text{Total variable cost} = \text{Rs. } 199,626,200$$

$$\begin{aligned}\text{Total investment} &= \text{TVC} + \text{TFCI} \\ &= \text{Rs. } 2.75 \times 10^8\end{aligned}$$

$$\begin{aligned}\text{Profit after taxation} &= \text{sales per annum} - \text{total variable cost} - \text{depreciation} - \text{taxes} \\ &= 1.314 \times 10^9 - 2.75 \times 10^8 - 2,284,546 - 2,586,525 \\ &= \text{Rs. } 1.03 \times 10^9\end{aligned}$$

$$\begin{aligned}\text{Payback period} &= \text{TFCI} / \text{profit after tax} \\ &= 76,151,526 / (1.03 \times 10^9) \\ &= 0.074 \text{ Years}\end{aligned}$$

CHAPTER 9: INDUSTRIAL SAFETY

Risk of Accidents and Harmful Exposures: Areas of Concern

- i) Dangerous Materials
- ii) Hazards of Pressure Vessels
- iii) Hazardous Chemical Reactions
- iv) Hazardous of Unit Operations
- v) Flammable Gases, Vapors and Dust Hazards
- vi) Health Hazards
- vii) Hazards due to corrosion
- viii) Entry in To Confined Spaces
- ix) Working with Pipelines
- x) Plant Alteration and modification
- xi) Sampling and Gauging
- xii) Hazards due to Instrument Failures.

Dangerous Materials

- i) Explosives
- ii) Gases
- iii) Inflammable Liquids
- iv) Inflammable Solids
- v) Oxidizing substances
- vi) Toxic and Infectious substances
- vii) Radio Active Substances
- viii) Corrosive Substances
- ix) Miscellaneous Dangerous Substances

Hazards of Pressure Vessels

1. Leakage or Bursting of Pressure Vessels
2. Design defects

3. Failure of Relief Systems
4. Lack of hydraulic testing.
5. Lack of Proper Instrumentation or Instrumentation Failure
6. Lack of ND Tests
7. Corrosion of Vessels.
8. Lack of routine inspections
9. Attempt of Pneumatic testing

Hazards of Unit Operations

Understanding the hazards inherent in each unit operation and adopting precautionary and emergency measures. Examples

Heat Transfer	Surface Fouling & Leakage, Miscalculation in scaling, Mixing of fluids etc.
Size Reduction	Dust Explosions, Dust release, etc.

Flammable Gases, Vapors and Dust Hazards

- Identification of potential areas, where possibility of flammable mixture are possible.
- Efforts to avoid hazardous mixtures, by inert gas purging and other methods.
- Declaring hazard zones and providing flameproof electrical fittings and equipment.
- Providing Explosion Vents in spaces with possibility of air-vapour mixtures.
- Explosive meter testing.
- Providing adequate fire control devices.
- Providing arrangements to avoid static sparks. Etc.

Health Hazards

- Identification of potential health hazards.
- Assessment of levels of physical and chemical health hazards.
- Control of hazards by various techniques
- Adequate awareness among the workers.
- Periodic medical examination of the workers.
- Personal protection for occasional exposures.
- Proper hygiene and decontamination facilities. etc.

Hazards Due To Corrosion

- Weakening and falling of structures and sheds.
- Falling of workers from height due to breaking of raised platforms, handrails, toe boards, stairs and ladders.
- Spills and toxic releases from pipelines due to corrosion.
- Leakages and bursting of vessels due to corrosion.
- Corrosion monitoring and control.
- Testing and inspection of vessels and structures to ensure safety

Entry into Confined Spaces

1. Oxygen Deficiency
2. Toxic Contamination
3. Flammable Environment
4. Possibility of Electrocutions through electric equipment
5. Possibility of Toxic gas generation during the work
6. Lack of Ventilation

7. Difficulty in welfare monitoring
8. Failure to escape on emergency.
9. Combustible Substances

Safety While Entry into Confined Spaces

- Thorough cleaning and purging before hot work.
- Safety belt with one end outside.
- Lifeline to monitor welfare.
- Ongoing ventilation.
- A person to watch the welfare.
- Low voltage electric appliances.
- Self-contained breathing apparatus.
- Environmental monitoring for oxygen, toxic gases and flammable gases before entry.
- Pipeline isolation before entry
- Electric isolation before entry.
- Proper ladder for entry.

Safety in Use of Pipelines

- Hazards due to inadequate Identification of Pipelines.
- Hazards due to leakages and bursting of pipelines.
- Hazards due to collision of vehicles with pipelines.
- Hazards due to improper materials of construction of pipelines.
- Hazards while breaking of pipeline

Plant Alterations and Modifications

Alteration in plant, equipment, component, process, operating procedure,

etc. due to some difficulty. Followed by failure in some unforeseen aspect of the system.

- If any alteration is inevitable,
- Design intention of each component of the system should be well understood by every person concerned.
- Refer the matter to the designers.
- Carry out HAZOP study by expert team.
- Pass it through plant modification approval committee

Sampling and Gauging

- Exposures of gases, vapours and dust while collecting samples.
- Approaching odd locations.
- Splashes and spillages while collectingsamples.
- Exposures due to breaking of sight glasses and glass level indicators.
- Dip gauging of flammable liquids.
- Dip gauging of corrosive liquids.

Hazards Due To Instrument Failures

- Absence of fail safety instruments.
- Lack of interlocks and trip systems.
- Human failures in manual and semi-automatic operations.

CHAPTER 10: CONCLUSION

The process described above leads to considerable improvement in the yield of oxalic acid in a single step. In this case, production efficiency was as high as 76% and this process has an advantage of producing Nitric acid and Nitrogen oxide gas which can be used in other industrial process or upon treatment of the by-products, also it can also be used in the same plant. The purity of oxalic acid is 98%, which is best available in the market.

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