

Process Design and Simulation of a Single Super Phosphate Plant



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Certificate

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Dedication

This thesis is dedicated to our parents and our friends, without whose support we could not have completed this project.

And, to our respected and esteemed supervisor and respected faculty without whose support and guidance we would not have been able to complete this thesis and degree.

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Abstract

Fertilizers are very important for the better growth of crops. Pakistan is an agricultural country, so better and cheap fertilizers are important for the country. Single superphosphate is a chemical fertilizer, which contains phosphorous as a major nutrient. It is suited to Pakistan's soils since they are relatively alkaline. It also contains some micronutrients like calcium, magnesium, aluminum, iron, sulphur and gypsum. Gypsum works as a soil conditioner. Single super phosphate is manufactured by the reaction of finely grounded Rock Phosphate with diluted sulphuric acid. The SSP is available in powder and granulated form. Tri calcium phosphate $\text{Ca}_3(\text{PO}_4)_2$ which is the major component of the natural phosphate rock is not soluble in water and plants only use phosphorus components when they are in soluble form. For this, tri-calcium phosphate is converted into mono-calcium phosphate, which is SSP. In this process, sulphuric acid is diluted to 70 percent before mixing it with grinded rock phosphate.

The process design and simulation of the single super phosphate plant in our final year project has been carried out to encompass an efficient material balance, energy balance, plant design, and economic analysis, and hazard & operability study, production of 500 tonnes per day of fertilizer. Following that, simulating and costing through Aspen Plus has been carried out to compare costing and overall feasibility of plant. The economic analysis of the plant calculated annual sales and overall installment costs to give us a payback period of approximately 2 years and 3 month

CHAPTER 1: Introduction

1.1 Introduction

Fertilizers are natural or synthetic materials containing nitrogen, phosphorus, and potassium compounds, spread on or worked into the soil to increase its capacity to support plant growth. They are essential for high yield crops. Mined inorganic fertilizers had been used for many centuries. While synthesized were developed during the industrial revolution. The use of inorganic fertilizers has widely helped the worldwide population. According to research, half of the world population is fed by the development of fertilizers. Fertilizers provide nutrients in varying proportions.

The history of commercial fertilizer is small as compared to the growth of crops. It is believed that crop production started about 6000 to 10000 years back. It was the 1840s afterward when first-time natural fertilizers like Peruvian Guano and Chilean Sodium Nitrate were used in western countries. Good agricultural techniques like manuring, liming and crop rotation were used in ancient Rome. Organic manure had been used in China. In old times, ashes of different plants were used as fertilizers. [1]

Fertilizers are used because they add nutrients and texture to the soil that needs to support trees, vegetables, herbs, shrubs, and flowers. The different type of fertilizers depends upon the nature of the soil, whether the soil is acidic, alkaline, sandy clay, rocky, weak or fertile. Fertilizers are used to improve the overall quality of the soil. Usually, a soil test is made to determine the number of nutrients in the soil. The soil test report will make recommendations for the amount and type of fertilizer to be used for optimum plant growth.

Sixteen basic nutrient elements are essential for the growth and reproduction of plants. The three most abundant nutrients are Carbon, Hydrogen and Oxygen from water and air.

The other elements are divided into two categories:

a. Primary Nutrients:

Nitrogen, Phosphorous, Potassium, Magnesium, Sulfur, and Calcium are six elements that are required in large quantities compared to other nutrients.

b. Secondary Nutrients:

Boron, Copper, Iron, Manganese, Molybdenum, Zinc, Chlorine, and Nickel are required by the plant in lesser quantities but are no less essential for good plant growth than the primary nutrients.

1.2 Types of Fertilizers

Fertilizers are classified into organic and inorganic or synthetic fertilizers.

1.2.1 Organic Fertilizers

Organic fertilizers are the most convenient forms of fertilizers. They are safe and easily available. Things like manure, slurry, worm castings, peat moss, seaweed, sewage and guano are good examples of organic fertilizers. Vegetation material called mulch, such as hay, peat moss, leaves, grass, bark, wood chips, seed hulls, and corn husks all help to aerate the soil, insulate the ground against temperature change, and add needed nutrients.

Apart from these naturally occurring minerals like sulfate of potash, limestone and rock phosphate are also considered very good organic fertilizers.

1.2.1.1 Advantages of Organic Fertilizers

The advantages of organic fertilizers are as follows:

1. Improve the structure of the soil.
2. Retain soil moisture.
3. Release nitrogen slowly and consistently.

4. Mobilize existing soil nutrients.
5. Do not burn the plants like some chemical fertilizers
6. Less subject to leaching

1.2.1.2 Disadvantages of Organic Fertilizers

1. Often organic fertilizers, especially those that contain animal and plant feces are contaminated with pathogens. Make sure they are properly composted to reduce the risk of pathogens.
2. The composition of organic fertilizers is variable thus; it becomes a very dilute and inaccurate source of nutrients compared to inorganic type of fertilizers. For profitable yields, significantly large amounts of fertilizers should be used to cope up with nutrient requirements.

1.2.2 Inorganic/Synthetic Fertilizers

Inorganic or synthetic fertilizers are primarily derived from chemical compounds such as ammonium nitrate, ammonium phosphates and potassium chloride. Mined rock phosphate and limestone are examples of inorganic fertilizers. Other synthetic fertilizers include:

- a. Single super phosphate
- b. Double super phosphate
- c. Triple super phosphate
- d. Urea
- e. Di-ammonium phosphate

1.2.2.1 Advantages of Inorganic/Synthetic Fertilizers

- **Measurable Nutrients:**

Unlike organic fertilizers, we know the nutrition content of synthetic fertilizers.

The three-digit numbers on the bag tell us the values of nutrients. This number

is called N-P-K value. N-P-K value shows the Nitrogen, Phosphorus and Potassium content present in that fertilizer.

- **Greater Concentration of Nutrients:**

Greater nutrition has its effects on the plant growth. Greater potassium content promotes protein synthesis. Phosphorus content allows storing energy. Nitrogen gives higher crop yields.

- **Quick Action:**

Synthetic fertilizers releases nutrients quickly and have rapid action on plant.

1.2.2.2 Types of Synthetic Fertilizers

1) Nitrogen Fertilizers:

- a. **Sodium Nitrate:**

First commercial fertilizer was sodium nitrate. It was mined from natural deposits in Chile.

- b. **Ammonium Sulfate:**

The next commercial source of the nitrogen fertilizer is ammonium sulfate. First, it was the by-product of the coal gas for the use in industry and domestic. The first synthetic production was introduced in 1920s.

- c. **Calcium Cyan amide:**

Nitric acid and calcium nitrate were produced in Norway around 1905. Before this, Germens have discovered how to synthesize calcium cyan amide and they know its importance as fertilizer. The first plant for the production of calcium cyan amide was set up in Italy.

- d. **Anhydrous ammonia:**

First commercial plant for the production of ammonia was set up in 1911 in Germany. However, the start of production from water gas as source of hydrogen started in 1920 in America.

e. Urea:

The present method for the production of urea was developed in Germany in 1920. It is the reaction between carbon dioxide and ammonia to form urea.

2) Phosphorus Fertilizers:

a. Phosphate Rock:

Phosphate rock is the only source for the production of phosphorus fertilizers. It is mined from various locations. Most of the deposits are marine phosphate deposits. Phosphate rock was first mined in 1940s.

b. Phosphoric Acid:

Phosphate is very stable compound to form very basic fertilizers from it we need some strong reactant or very high thermal energy. Reaction with sulphuric acid to form phosphorus acid is the most used technique.

c. Potassium (potash):

All the deposits of potash are in sedimentary rocks. The reserves of potassium are immense. They are immense enough to be used for centuries.

1.3 Single Super Phosphate

Single super phosphate is a chemical fertilizer, which contains phosphorus as a major plant nutrient. It is relatively very cheap and contains many micronutrients like calcium, magnesium, iron, aluminum, sulphur and gypsum. The gypsum works as a soil conditioner. The SSP is manufactured by the reaction of finely grounded rock phosphate with diluted sulphuric acid. The SSP is available in powder and granulated form. The powder form is called as PSSP and the granulated form is called as GSSP. The PSSP and GSSP are available in two grades. Grade A and grade B. The grading is based on water-soluble phosphorous pentoxide present in the product. The A grade contains 16% water-soluble phosphorous pentoxide and the B grade contains 14% water-soluble P_2O_5 . The granulated

super phosphate is somewhat costlier in comparison to PSSP because of the added cost of granulation. The GSSP dissolves slowly in the water present in the soil so it is available to the crops for a longer time. The free phosphoric acid present in the SSP is very useful for alkaline soils. The PSSP & GSSP are white, cream, gray, brown or black in color and packed in 50 Kg bags. The SSP is manufactured by the reaction of finely ground rock phosphate and diluted sulphuric acid. The rock phosphate is available in Jordan, Egypt, India, China, Israel, Morocco, Syria, Senegal and many other African countries. The sulphuric acid is produced by using sulphur as raw material [2].

1.3.1 Granulated Single Superphosphate

The granulated SSP is produced by granulation of SSP in the granulation plant. The granulation is done because the nutrients remain available for a longer time. The granule releases the nutrients very slowly. This process improves the nutrient utilization.

1.4 Raw Materials and their Resources

The raw materials for manufacturing SSP are as follows:

1. Rock Phosphate:

Rock phosphate is mined in many countries like India, China, Israel, Jordan, Syria, Morocco, Egypt, Malaysia, Indonesia and many African & American countries. The 31% plus grade is suitable for the production.

2. Sulphuric Acid:

The technical grade sulphuric acid that is 98.5% pure is suitable for the process. Use however spent sulphuric acid from other industries like textiles, Detergent and which is a waste, can also along with the technical grade sulphuric acid in the plants designed by us.

3. Fuel:

To dry GSSP any fuel like Furnace Oil, H.S. Diesel, LDO etc. may be used.

4. Fillers:

The filler used are Gypsum and Sand.

5. Bagging Material:

The bagging material used for Single Super Phosphate is High Density Polyethylene (HDPE) bags.

1.5 Benefits of SSP

Some major uses of single super phosphate (SSP) are listed below:

1. It increases the soil fertility.
2. It softens the soil and hence improves the soil capability to absorb water.
3. One of the major advantages of using single super phosphate is that phosphoric acid is fully water-soluble.
4. It also improves the quality of vegetables and fruits.
5. All soil types can get benefit from the application of single super phosphate as a fertilizer.
6. It also reduces pH value of soil.

CHAPTER 2: Process Description

2.1 Process Introduction:

The process of manufacturing single super phosphate includes 65-75% sulphuric acid and grinding rock phosphate mixed together for two to four hours. The percentage purity of sulphuric acid depends upon the composition of rock phosphate.

Rock phosphate is crushed using a hammer crusher and then grinded using a roller mill. Roller mill is used because it supports high feed rate. The particle size of rock phosphate after grinding required is approximately 57 microns.

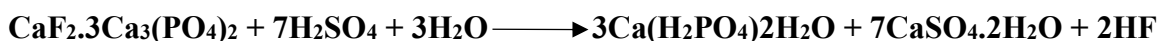
After mixing, the stream is fed to a rotating Den reactor where it is further allowed time to react. Gases are evolved from reactor that are corrosive in nature. To save the working environment, these corrosive gases are scrubbed in a vertical spray tower using water.

The scrubbed gases includes carbon dioxide and water vapors, which are then released into the atmosphere.

Fig. 2.1 shows the block flow diagram for manufacturing process.

2.2 Process of Manufacturing

The manufacturing process depends on reacting phosphate rock with sulphuric acid and the fertilizer contains about 32% P₂O₅. The net reaction proceeds as follows:



The process can be divided into two stages as follows:

1. The first step represents the diffusion of sulphuric acid to the rock phosphate particles accompanied by a rapid chemical reaction on the particle surface, which continues until the acid completely consumed, and crystallization of calcium sulfate.
2. The second stage represents the diffusion of the formed phosphoric acid into the pores of the rock phosphate, which did not decompose with sulphuric acid. This stage is accompanied by a second reaction.

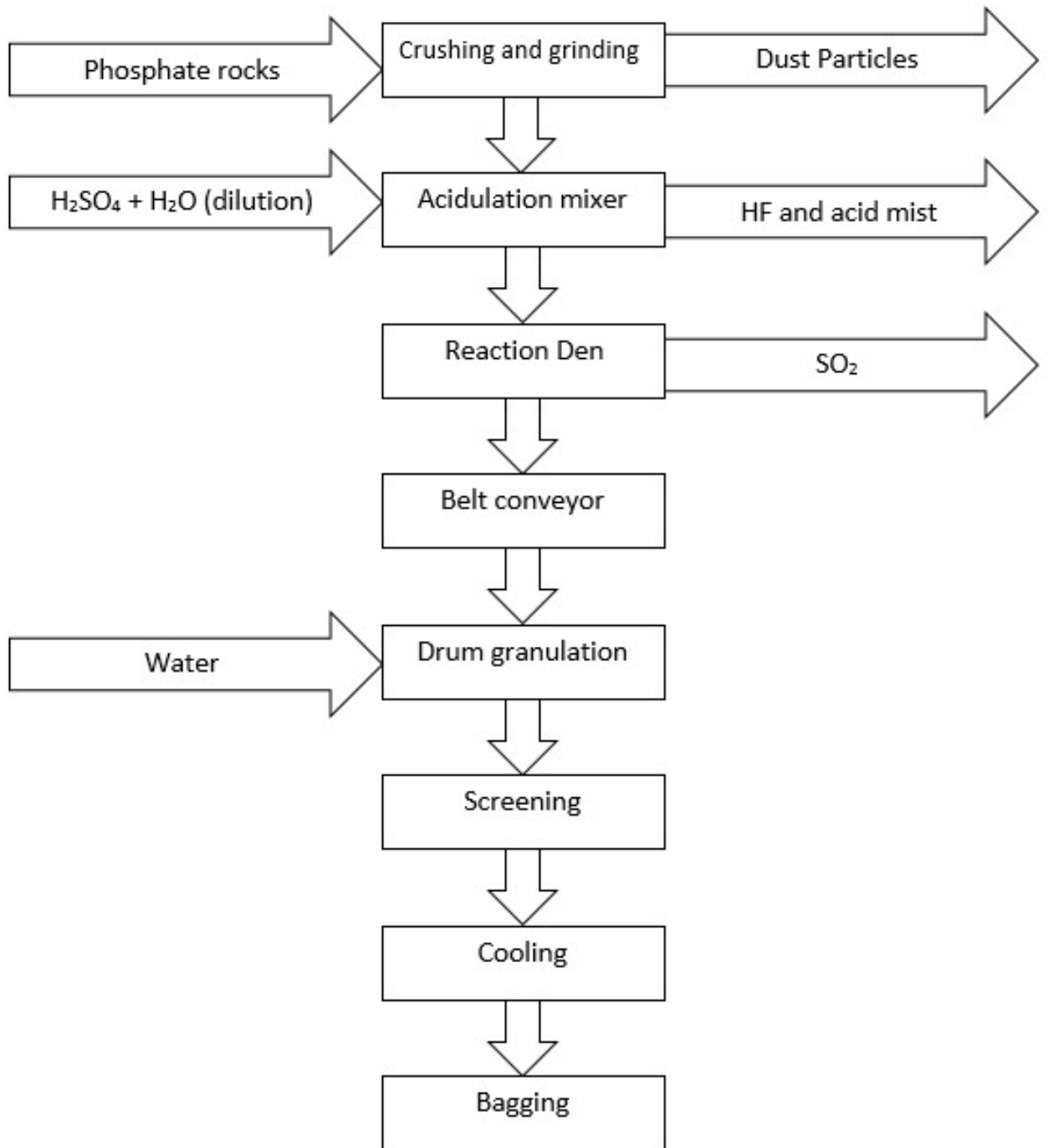


Figure 2.1: Block Flow Diagram of SSP Production

2.2.1 Phosphate rock blending and grinding

In this process, ground phosphate rock is transported from the storage site to automatic weight, by a system of belt and screw conveyors and elevators, which feed the continuous action double conical mixer. The sulphuric acid is continuously diluted with water in a batch mixer to a 70% concentration. After this, it is fed to a mixer to start reaction between ground rock phosphate and sulphuric acid where a first reaction takes place. This reaction ends in the first 1-2 hours, during the period of settling and hardening of the superphosphate slurry, which is caused by the relatively rapid crystallization of the low solubility calcium sulfate.

2.2.2 Superphosphate manufacture

The next stage of the process is ageing of the superphosphate i.e. the formation and crystallization of mono-calcium phosphate in the den. The formed slurry is transported to the continuous-action reaction den, which has a very low travel speed to allow for solidifying where formation of super phosphate takes place (settling and hardening of the slurry in the first stage of ageing). Considerable quantities of fluoride compounds are evolved from the acidulation, they are sent to the scrubbers.

The superphosphate powder, from the den, is transferred for ageing by a belt conveyor, located below the den, to the pile storage for curing, or completion of chemical reaction, which takes 2-6 weeks to P-Os availability acceptable for plant nutrient. The raw fertilizer is uniformly distributed by a scattering device and in order to accelerate the ageing operation, the superphosphate is agitated during storage by means of a grab-bucket crane. The end product still contains a certain amount of uncombined phosphoric acid, which makes the fertilizer more hygroscopic.

Neutralizing mixtures are used to remove the free acid of the superphosphate, or it is treated with gaseous ammonia. These procedures improve the physical properties of the superphosphate. They lower the moisture content, the hygroscopic and the tendency to cake. If ammonia treatment is used, an additional nutritional component (N₂) is also introduced into the fertilizer. During reaction of the phosphate with sulphuric acid in the

den, hydrogen fluoride evolves and reacts with the silica contained in the phosphates and forms gaseous silicon-tetra-fluoride (SiF_4).

The continuous den is, therefore, enclosed so that fumes of these compounds do not escape into the working place. The fluorine gases, containing SiF_4 vapors, are withdrawn through an opening in the den roof into a ventilation pipe to an absorption unit and are utilized for making sodium fluorosilicates.

2.2.3 Granulation

Superphosphate is granulated in drum granulators to improve its physical properties. In the granulator, the superphosphate powder (after being cured for 2-6 weeks) is wetted with water fed into the drum through nozzles, and rolled into granules of different size which are then dried, screened into size fractions cooled and the product is bagged in plastic (polyethylene) bags. The oversize granules are ground and recycled, with the undersize granules, to the den.

2.2.4 Utilities

A drying tower is used to dry the air in which the molten sulfur is burned. This is a packed tower in which air is blown counter-current against a stream of concentrated sulphuric acid, which absorbs any moisture.

The heat generated by the exothermic reaction between molten sulfur and oxygen is recovered in a waste heat boiler to produce steam which is then used for process heating and also to drive a steam turbine which produces electricity. The electricity is used on site and any excess is exported to the local power supplier. In addition, a gas scrubber is used to prevent any hydrofluosilicic acid from being released into the atmosphere.

2.2.5 The Role of Laboratory

The laboratory is responsible both for quality control and for research. Firstly, the laboratory monitors the exact composition of the phosphate rock blend (which varies

significantly depending on the source of the rock). This is important as the physical properties of the finished product (particle size, dryness, friability, etc.) which are critical if the fertilizer is to be easily spread and interact with the soil in a satisfactory way depend on these parameters. Secondly, research is conducted both on site and through scientists.

2.2.6 Environmental Applications

The most significant potential environmental hazards are dust (from the grinding of phosphate rock) and gaseous hydrofluosilicic acid (from the reaction between hydrofluoric acid and silica or quartz) emissions. These are both carefully monitored, and a dust catcher and gas scrubber are used.

2.3 Properties of Single Superphosphate:

a. Nature:

- Free flowing powder and granules
- Grayish to brownish white angle of repose 360°
- Slightly hygroscopic / caking at long storage
- Acidic in nature

b. Quality Specifications:

- Powder SSP (Green superphosphate)
- Moisture contents 12% max.
- Free acidity 4% max
- Water soluble P_2O_5 16% min

CHAPTER 3: Material Balance

3.1 Introduction to Material Balance

The raw materials used are:

- 1) Rock Phosphate
- 2) Sulphuric acid
- 3) Water

Basis: 100 kg/hr

Table 1: Composition of Rock Phosphate

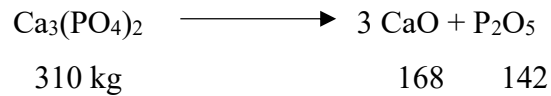
Components	Wt. percentage	Flowrate (kg/hr)
H ₂ O	0.16	49.43
CaO	39.9	31.5
P ₂ O ₅	28.35	3.8
F	3.0	5
CO ₂	1.4	0.25
MgO	0.25	0.7
Na ₂ O	0.31	5.69
SiO ₂	22.3	0.35
Al ₂ O ₃	2.4	0.2
Fe ₂ O ₃	1.5	0.03
K ₂ O	0.59	2.60
SO ₃	0.35	
Cl	125 ppm	

Assumptions

- All P₂O₅ is present as Ca₃(PO₄)₂
- All F is present as CaF₂
- All SO₃ present as CaSO₄.2H₂O
- All CO₂ present as CaCO₃

a. For $\text{Ca}_3(\text{PO}_4)_2$

142 kg of P_2O_5 are in 310 kg of $\text{Ca}_3(\text{PO}_4)_2$



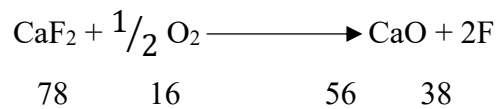
$$\text{Kg/hr of } \text{Ca}_3(\text{PO}_4)_2 = (310/142) * 28.35 = 61.89 \text{ kg/hr}$$

b. For CaCO_3 :



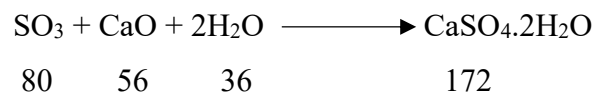
$$\text{Kg/hr of } \text{CaCO}_3 = (100/44) * 1.4 = 3.182 \text{ kg/hr}$$

c. For CaF_2 :



$$\text{Kg/hr of } \text{CaF}_2 = (78/38) * 3 = 6.158 \text{ kg/hr}$$

d. For $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$:



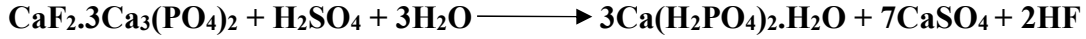
$$\text{Kg/hr of } \text{CaSO}_4 \cdot 2\text{H}_2\text{O} = (172/80) * 0.35 = 0.7525 \text{ kg/hr}$$

e. H_2O consumed:

$$\text{H}_2\text{O consumed} = 36/80 * 0.35 = 0.1575 \text{ kg/hr}$$

3.2 Reactions Involved

3.2.1 Main Reactions

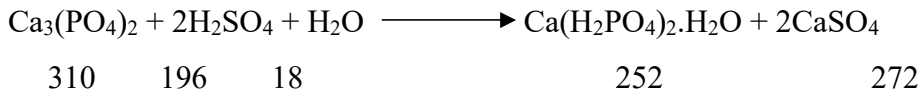


The main objective is to convert tri-calcium phosphate $\text{CaF}_2 \cdot 3\text{Ca}_3(\text{PO}_4)_2$ into mono-calcium phosphate $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ because tri-calcium phosphate is insoluble in water and unavailable to plant till it dissolves in water. So mono-calcium phosphate is produced by reaction of tri-calcium phosphate with 70% H_2SO_4 so soluble phosphate fertilizer is produced which can be taken up by the plant.

Alongside of mono-calcium phosphate, some di-calcium phosphate is also produced and some of the tri-calcium phosphate is left unreacted:

Reaction # 1:

97% conversion



Kg/hr of $\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ produced = $(252/310) * 61.9 * 0.97 = 48.81$ kg/hr

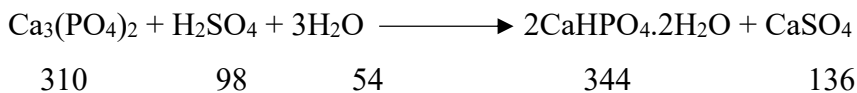
Kg/hr of CaSO_4 produced = $(272/310) * 61.9 * 0.97 = 52.68$ kg/hr

Kg/hr of sulphuric acid used = $(196/310) * 61.9 * 0.97 = 37.96$ kg/hr

Water used to dilute sulphuric acid to 70% = $(18/310) * 61.9 * 0.97 = 3.48$ kg/hr

Reaction # 2:

1.5% conversion



Kg/hr of $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$ produced = $(344/310) * 61.9 * 0.015 = 1.03$ kg/hr

Kg/hr of CaSO_4 produced = $(136/310) * 61.9 * 0.015 = 0.41$ kg/hr

Kg/hr of sulphuric acid used = $(98/310) * 61.9 * 0.015 = 0.29$ kg/hr

Water used to dilute sulphuric acid to 70% = $(54/310) * 61.9 * 0.015 = 0.162$ kg/hr

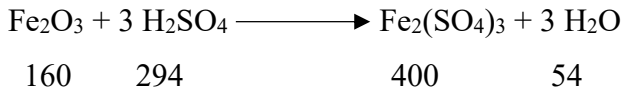
Reaction # 3:**0.5% conversion**

Kg/hr of H_3PO_4 produced = $(392/620) * 61.9 * 0.005 = 0.1 \text{ kg/hr}$

Kg/hr of CaSO_4 produced = $(816/620) * 61.9 * 0.005 = 0.41 \text{ kg/hr}$

Kg/hr of sulphuric acid used = $(588/620) * 61.9 * 0.005 = 0.29 \text{ kg/hr}$

1% of BPL is unreacted so $61.9 * 0.01 = 0.62 \text{ kg/hr}$

3.2.2 Side Reactions**Reaction # 4:**

Kg/hr of $\text{Fe}_2(\text{SO}_4)_3$ produced = $(400/160) * 1.5 = 3.75 \text{ kg/hr}$

Kg/hr of sulphuric acid used = $(294/160) * 1.5 = 2.75 \text{ kg/hr}$

Kg/hr of H_2O formed = $(54/100) * 1.5 = 0.51 \text{ kg/hr}$

Reaction # 5:

Kg/hr of $\text{Al}_2(\text{SO}_4)_3$ produced = $(342/102) * 2.4 = 8.05 \text{ kg/hr}$

Kg/hr of sulphuric acid used = $(294/102) * 2.4 = 6.92 \text{ kg/hr}$

Kg/hr of H_2O formed = $(54/102) * 2.4 = 1.27 \text{ kg/hr}$

Reaction # 6:

Kg/hr of Na_2SO_4 produced = $(142/62) * 0.31 = 0.71 \text{ kg/hr}$

Kg/hr of sulphuric acid used = $(98/62) * 0.31 = 0.49 \text{ kg/hr}$

$$\text{Kg/hr of H}_2\text{O formed} = (18/62) * 0.31 = 0.09 \text{ kg/hr}$$

Reaction # 7:

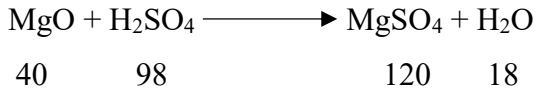


$$\text{Kg/hr of K}_2\text{SO}_4 \text{ produced} = (174/94) * 0.59 = 1.09 \text{ kg/hr}$$

$$\text{Kg/hr of sulphuric acid used} = (98/94) * 0.59 = 0.61 \text{ kg/hr}$$

$$\text{Kg/hr of H}_2\text{O formed} = (18/94) * 0.59 = 0.11 \text{ kg/hr}$$

Reaction # 8:

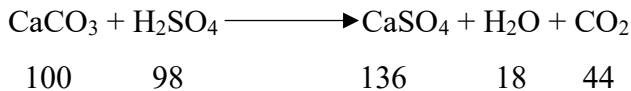


$$\text{Kg/hr of MgSO}_4 \text{ produced} = (120/40) * 0.25 = 0.75 \text{ kg/hr}$$

$$\text{Kg/hr of sulphuric acid used} = (98/40) * 0.25 = 0.61 \text{ kg/hr}$$

$$\text{Kg/hr of H}_2\text{O formed} = (18/40) * 0.25 = 0.11 \text{ kg/hr}$$

Reaction # 9:



$$\text{Kg/hr of CaSO}_4 \text{ produced} = (136/100) * 3.18 = 4.32 \text{ kg/hr}$$

$$\text{Kg/hr of CO}_2 \text{ produced} = (44/100) * 3.18 = 1.4 \text{ kg/hr}$$

$$\text{Kg/hr of H}_2\text{O produced} = (18/100) * 3.18 = 0.57 \text{ kg/hr}$$

$$\text{Kg/hr of sulphuric acid used} = (98/100) * 3.18 = 3.121 \text{ kg/hr}$$

Reaction # 10:



$$\text{Kg/hr of CaSO}_4 \text{ produced} = (136/78) * 6.16 = 10.74 \text{ kg/hr}$$

$$\text{Kg/hr of sulphuric acid used} = (98/78) * 6.16 = 7.74 \text{ kg/hr}$$

$$\text{Kg/hr of HF produced} = (40/78) * 6.16 = 3.16 \text{ kg/hr}$$

Reaction # 11:

This reaction is proceeded by HF and SiO₂ but since HF is limiting reactant (3.16 kg/hr) and SiO₂ is 22.31 kg/hr in rock phosphate. So, all SiF₄ calculations are based on HF.

Kg/hr of SiF₄ produced = (104/80) * 3.16 = 4.11 kg/hr

Kg/hr of SiO₂ consumed = (80/60) * 3.16 = 4.21 kg/hr

Kg/hr of SiO₂ left = 22.31 – 4.21 = 18.09 kg/hr

Kg/hr of H₂O formed = (36/80) * 3.16 = 1.42 kg/hr

3.3 Calculation**3.3.1 Total Products Produced from 100 kg/hr of Rock Phosphate****Table 2: Formation of Products from Rock Phosphate**

Product	Production Reaction	Quantity (kg/hr)
CaSO ₄	1, 2, 3, 9, 10	68.56
Ca(H ₂ PO ₄) ₂ .H ₂ O	1	48.81
CaHPO ₄ .2H ₂ O	2	1.03
H ₃ PO ₄	3	0.19
Fe ₂ (SO ₄) ₃	4	3.75
Al ₂ (SO ₄) ₃	5	8.05
K ₂ SO ₄	7	1.09
MgSO ₄	8	0.75
Na ₂ SO ₄	6	0.71
SiO ₂		18.09
CaSO ₄ .2H ₂ O		0.7525

3.3.2 Total amount of 100% H₂SO₄ required for 100 kg/hr ROP

From Reaction # 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 is 60.68 kg/hr

98% H₂SO₄ required = $60.68/0.98 = 61.92$ kg/hr

70% H₂SO₄ required = $60.68/0.70 = 86.69$ kg/hr

So, water required for dilution = $86.69 - 61.92 = 24.77$ kg/hr

Water is also produced in various reactions; it is calculated to be

$$0.57 + 0.11 + 0.09 + 1.42 + 1.27 + 0.51 + 0.11 = 4.24 \text{ kg/hr}$$

Water consumed during different reactions = $3.48 + 0.16 = 3.64$ kg/hr

Hence, free available water = total water + water formed – water consumed

$$= 24.77 + 4.24 - 3.64 = 25.37 \text{ kg/hr.}$$

3.3.3 Composition of SSP

Table 3: Composition of Single Super Phosphate

Components	Amount Produced (kg/hr)	Wt.% on dry basis
Ca(H ₂ PO ₄) ₂ .H ₂ O	48.81	32
CaHPO ₄ .2H ₂ O	1.03	0.67
H ₃ PO ₄	0.19	0.124
CaSO ₄	68.56	44.98
Fe ₂ (SO ₄) ₃	3.75	2.46
Al ₂ (SO ₄) ₃	8.05	5.28
K ₂ SO ₄	1.09	0.715
MgSO ₄	0.75	0.492
Na ₂ SO ₄	0.71	0.46
SiO ₂	18.09	11.87
CaSO ₄ .2H ₂ O	0.75	0.49
Unreacted	0.62	0.406

3.3.4 Calculation for 500 TPD of SSP

∴ 500 tonnes per day \approx 20800 kg/hr

For our capacity, raw material required:

173.18 kg/hr of SSP is obtained from 100 kg/hr of rock phosphate.

$$\begin{aligned} 20800 \text{ kg/hr of SSP requires rock} &= (100/173.18) * 20800 \\ &= 12010.6 \text{ kg/hr} \end{aligned}$$

H₂SO₄ required:

$$\begin{aligned} 98\% \text{ purity} &= (61.92/100) * 20800 \\ &= 7340.36 \text{ kg/hr} \end{aligned}$$

$$70\% \text{ purity} = (86.69/100) * 20800 = 10325 \text{ kg/hr}$$

$$\begin{aligned} \text{Water required for dilution} &= 10325 - 7340.36 \\ &= 2985 \text{ kg/hr} \end{aligned}$$

3.3.5 Rock Phosphate composition and flow rates of 500 TPD

Table 4: Rock Phosphate used for 500 TPD

Components	Wt.% of components	Flowrate (Kg/hr)
Ca ₃ (PO ₄) ₂	61.891	7434.55
CaCO ₃	3.182	381.9
CaF ₂	6.158	739.6
CaSO ₄ .2H ₂ O	0.7525	90.35
MgO	0.25	30
Na ₂ O	0.31	37.2
SiO ₂	22.3	2678.35
Al ₂ O ₃	2.4	288.15
Fe ₂ O ₃	1.5	180.15

K ₂ O	0.5	60.05
H ₂ O	0.16	19.2

3.3.6 SSP composition and flow rates of 500 TPD

Table 5: SSP manufactured for 500 TPD

Components	Wt.% on dry basis	Flowrate (kg/hr)
Ca(H ₂ PO ₄) ₂ .H ₂ O	32	5857.25
CaHPO ₄ .2H ₂ O	0.67	112.55
H ₃ PO ₄	0.124	22.65
CaSO ₄	44.98	8233.1
MgSO ₄	0.492	90.05
Fe ₂ (SO ₄) ₃	2.46	450.25
Al ₂ (SO ₄) ₃	5.28	966.45
Na ₂ SO ₄	0.465	85.1
K ₂ SO ₄	0.715	130.85
CaSO ₄ .2H ₂ O	0.493	90.2
SiO ₂	11.87	2172.65
Unreacted	0.406	74.3

3.3.7 Gases Evolved

HF evolved in reaction # 10 = $(3.16 * 20800) / 100 = 657.28$ kg/hr

SiF₄ evolved during the reaction # 11 = $(4.11 * 20800) / 100 = 854.88$ kg/hr

CO₂ evolved during the reaction # 9 = $(1.4 * 20800) / 100 = 291.2$ kg/hr

Water in the form of steam = $2978 - (20800 * 0.12) = 479$ kg/hr

3.4 Balance around Dilution Tank and Cooler

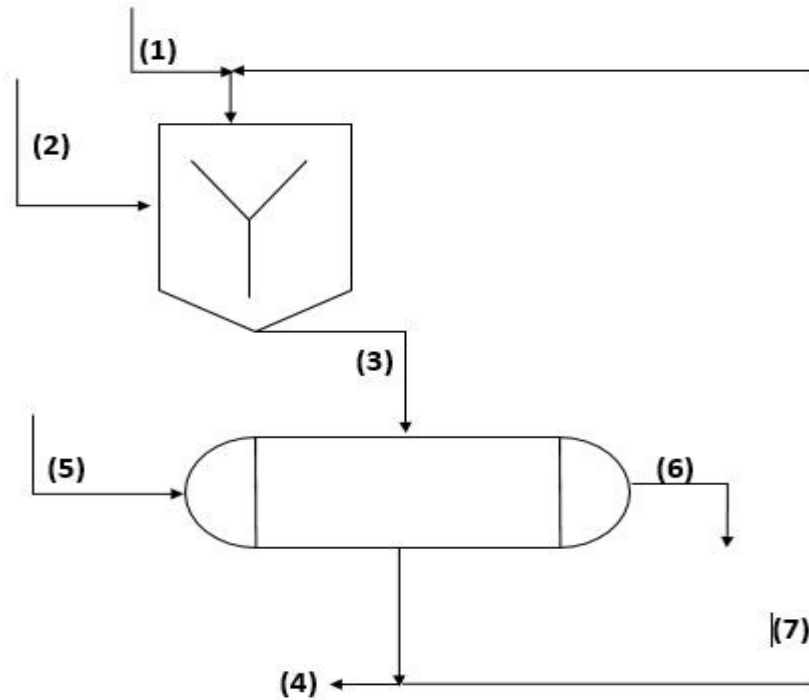


Figure 3.1: Dilution Tank and Cooler

Stream 7 is a recycle stream carrying 70% of sulphuric acid to avoid overheating in dilution tank because overheating can cause decomposition of sulphuric acid.

Table 6: Material Balance around Dilution Tank and Cooler

Stream	Component	Composition	Flowrate
1	H ₂ SO ₄	0.98	12879.3
2	H ₂ O	1.00	2975
3	H ₂ SO ₄	0.70	15854
4	H ₂ SO ₄	0.70	433.45
5	Cooling H ₂ O (in)	1.00	-
6	Cooling H ₂ O (out)	1.00	-
7	H ₂ SO ₄ recycle	0.70	-

3.5 Material Balance around Mixer

We assume that no reaction is taking place in mixer and residence time is 2-4 minutes.

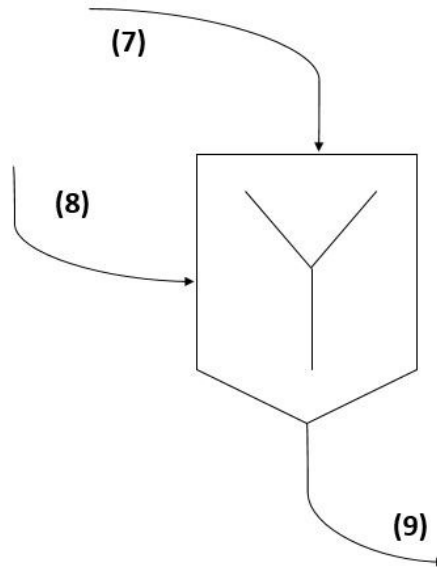


Figure 2.2: Mixer

Table 7: Material Balance around Mixer

Stream	Component	Flowrate (kg/hr)
7	H ₂ SO ₄	10325
8	Rock Phosphate	12010
9	Mixture of ROP and H ₂ SO ₄	22335

3.6 Material Balance around Den Reactor

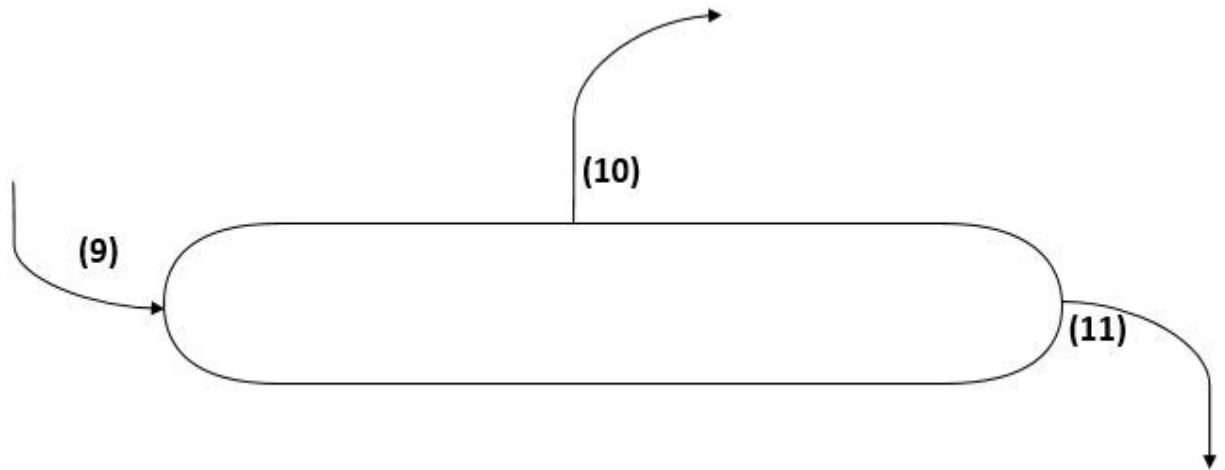


Figure 3.3: Den Reactor

Table 8: Material Balance around Den Reactor

Stream	Component	Flowrate (kg/hr)
9	Mixture of ROP and H ₂ SO ₄	22335
10	Gases Evolved	1535
11	SSP Produced	20800

3.7 Material Balance around Spray Tower:

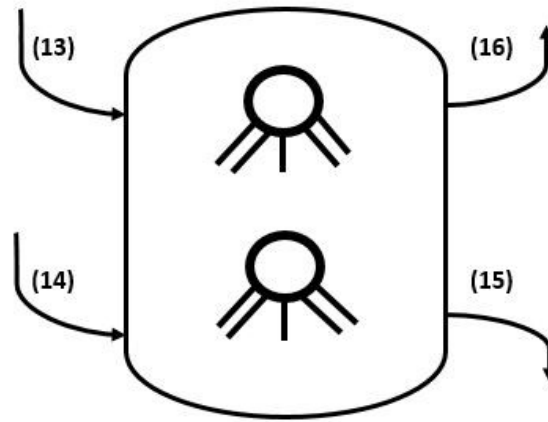


Figure 3.4: Spray Tower

Table 9: Material Balance around Spray Tower

Stream	Water	SiO ₂	CO ₂	SiF ₄
13	13500	-	-	-
14	481	-	290	850
15	13881	163.5	0.35	845
16	135	-	138	5

All values are taken in kg/hr [3].

CHAPTER 4: Energy Balance

Reference Temperature = 25°C = 298 K

Reference Pressure = 1 atm.

4.1 Energy Balance around Dilution Tank

Calculation of Exit Diluted Acid:

Here, the purpose of using dilution tank is to dilute the sulphuric acid from 98% purity to 75% purity with water. Both liquids are entering at 25°C; our aim is to find the exit temperature of 70% sulphuric acid if no cooling is provided. This will be calculated with the help of enthalpy-concentration diagram.

- i. Draw a perpendicular line at 98% concentration of sulphuric acid to the point, which this line cuts the temperature curve at 25°C.
- ii. Similarly, draw another line at 0 concentration that is pure water then extend it up to 25°C.
- iii. Join these two points.
- iv. Find out the value of temperature the perpendicular line from 70% concentration cuts the temperature curve. This gives the final temperature, which comes out to be 162°C.

Heat Absorbed to Cool the Acid:

Specific heat of sulphuric acid = 3.50 J/g. °C

Enthalpy of sulphuric acid:

Given that the standard state heats of formation for $\text{H}_2\text{SO}_4 (l)$, $\text{H}^+ (aq)$, and $\text{HSO}_4^- (aq)$ are -813.989 kJ/mol, 0 kJ/mol (defined), and -885.75 kJ/mol, respectively.

$$\begin{aligned}\Delta H^{\circ} &= [\Delta H^{\circ}_f, \text{HSO}_4^- + \Delta H^{\circ}_f, \text{H}^+] - \Delta H^{\circ}_f, \text{H}_2\text{SO}_4 \\ &= [(-885.75 + 0) - (-813.989)] \\ &= -71.76 \text{ kJ/mol}\end{aligned}$$

Unit conversion:

Mass of sulphuric acid = 7430 kg/hr

Molar mass of sulphuric acid = 98 kg/kmol

Mass of water added of dilution = 2985 kg/hr

Moles of sulphuric acid per hour = $\frac{7430 \text{ kg/hr}}{98 \text{ kg/kmol}} = 75.8 \text{ kmol/hr}$

Enthalpy of stream = $75.8 \times 10^3 \text{ mol/hr} * -71.76 \text{ kJ/mol}$
= -543940 kJ/hr (151 kW)

4.2 Energy balance around Den Reactor

In reactor, there are multiple reactions going on with sulphuric acid and rock phosphate. All these reactions are exothermic in nature, heat is evolved which raises the temperature of rock phosphate. Grinded rock phosphate enters the reactor and it is at a slightly high temperature because of milling and crushing. The sulphuric acid comes from heat exchanger and they both mix in a pre-mixer, which makes a slurry before entering the reactor.

Reactor has a 4-blade impeller inside which rotates and mixes the slurry uniformly as well as it pushes the slurry out of the reactor before it solidifies inside the reactor. This eliminates the risk of choking the reactor.

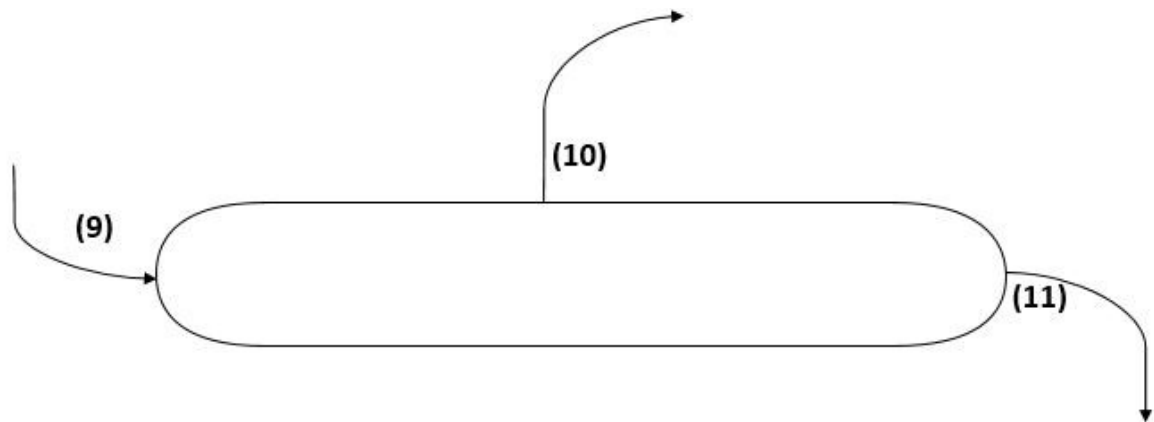


Figure 4.1: Den Reactor

Stream 9 is slurry coming from pre-mixer.

Stream 10 is gases evolving from reactor as rock phosphate reacts with acid.

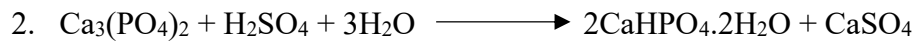
Stream 11 is the slurry exiting the reactor.

Temperature of gases = 90°C

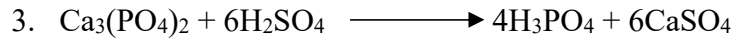
4.2.1 Reactions occurring in reactor



$$\Delta H = -171064 \text{ kJ/kg}$$

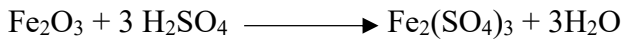


$$\Delta H = -404436 \text{ kJ/kg}$$

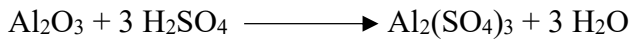


$$\Delta H = -459392 \text{ kJ/kg}$$

4.2.1.1 Side reactions



$$\Delta H = -1089.10 \text{ kJ/kg}$$



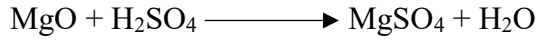
$$\Delta H = -178.94 \text{ kJ/kg}$$



$$\Delta H = -438.1 \text{ kJ/kg}$$



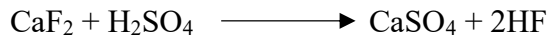
$$\Delta H = -543.6 \text{ kJ/kg}$$



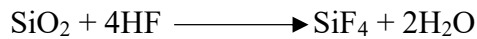
$$\Delta H = -149.67 \text{ kJ/kg}$$



$$\Delta H = -231.3 \text{ kJ/kg}$$



$$\Delta H = +842.48 \text{ kJ/kg}$$



$$\Delta H = -234.16 \text{ kJ/kg}$$

These are the reactions taking place in reactor when sulphuric acid comes in contact with rock phosphate [4].

HF instantly reacts with SiO_2 and forms SiF_4 and along with CO_2 , it is liberated from reactor.

Enthalpies of Components present in rock phosphate:

Table 10: Enthalpies of components present in rock phosphate

Components	Mass flowrate (kg/hr)	Enthalpy (kJ/kg)	Heat flow (kJ/hr)
$\text{Ca}_3(\text{PO}_4)_2$	1600	-105.0	-168000
CaCO_3	264	-121.1	-31944
CaF_2	181	-155.5	-28055
$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	130	-116.4	-15080
MgO	5.8	-150.3	-870
Na_2O	16.25	-67.0	-1088
SiO_2	132	-141	-18612
Al_2O_3	8.15	-163.5	-1328.4

Fe ₂ O ₃	4.65	-518.6	-2408.7
K ₂ O	0.14	-383.0	-53.62

Total Enthalpy = -267440 kJ/hr

4.3 Enthalpy of Sulphuric acid

Mass of sulphuric acid = 7430 kg/hr

Molar mass of sulphuric acid = 98 kg/kmol

Moles of sulphuric acid per hour = $\frac{7430 \text{ kg/hr}}{98 \text{ kg/kmol}} = 75.8 \text{ kmol/hr}$

Enthalpy of stream = $75.8 \times 10^3 \text{ mol/hr} * -71.76 \text{ kJ/mol}$

= -543940 kJ/hr

Enthalpies of components present in SSP:

Table 11: Enthalpies of components present in SSP

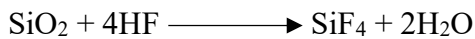
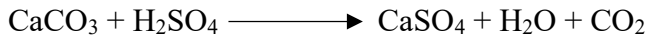
Components	Mass Flow (kg/hr)	Enthalpy (kJ/kg)	Heat Flow (kJ/hr)
Ca(H ₂ PO ₄) ₂ .H ₂ O	5857.25	-134.85	-789850.16
CaHPO ₄ .2H ₂ O	112.55	-129.28	-14550.46
H ₃ PO ₄	22.65	-150.30	-3404.29
CaSO ₄	8233.1	-929.6	-7653489.76
MgSO ₄	90.05	-106.22	-9565.11
Fe ₂ (SO ₄) ₃	450.25	-682.6	-307340.65
Al ₂ (SO ₄) ₃	966.45	-100.34	-96973.59
Na ₂ SO ₄	85.1	-97.28	-8278.52
K ₂ SO ₄	130.85	-82.31	-10770.26
CaSO ₄ .2H ₂ O	90.2	-126.01	-11366.102
SiO ₂	2172.65	-121.00	-262890.65

unreacted	74.3	-	-
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Total Enthalpy = -9168480 kJ/hr

4.4 Enthalpy of gases

Enthalpy of gases stream:



These are the gases evolved and the amount is given as:

CO₂:

Mass flowrate = 138 kg/hr

Specific heat capacity = 0.846 kJ/kg. K

Enthalpy = $\Delta H_f = -393.5$ kJ/mol CO_{2(g)}

Heat flow = - 45880 kJ/hr

SiF₄:

The production of SiF₄ is governed by the production rate of HF as evident from equations above; HF produced is consumed in the production of SiF₄.

Mass flowrate = 50 kg/hr

Specific heat capacity = 0.704 kJ/kg. K

Enthalpy = -1614 kJ/mol = -15520 kJ/kg (480 mol/hr = 50 kg/hr)

Heat flow = - 54600 kJ/hr

Water vapors:

Mass flow = 135 kg/hr

Heat flow = 35230 kJ/hr

Total Enthalpy of stream = -65170 kJ/hr

Considering that the reactor is perfectly insulated and no heat is being lost to the atmosphere, then all the heat generated is being used in raising the temperature. Hence, the output temperature of SSP produced coming from reactor is calculated to be 90°C.

4.5 Energy Balance around Spray Tower

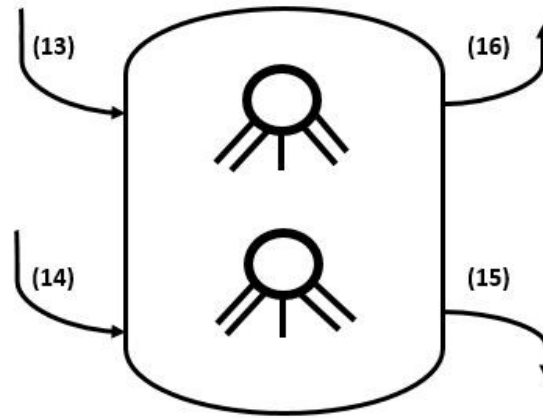


Figure 4.2: Spray Tower

Stream 13 is water sprayed from top.

Stream 14 is the gases coming from reactor.

Stream 15 is acidic water that includes all hazardous gases dissolved in it.

Stream 16 is the non-hazardous gases venting in atmosphere.

Total Enthalpy of gases stream = -65170 kJ/hr

Mass flow of water = 13500 kg/h

Heat flow = 3523000 kJ/hr

Enthalpy of exhaust gases = 5437.86 kJ/kg

Enthalpy of acidic water = 4333.82 kJ/kg

CHAPTER 5: Equipment Design

5.1 Heat Exchanger

Introduction:

A heat exchanger device is a heat transfer device used for the transfer of internal thermal energy between two or more fluid streams available at different temperatures. In most heat exchangers, the fluids are separated by heat transfer surfaces and ideally, they do not mix. Heat exchangers are used in process, power, petroleum, transportation, air conditioning, heat recovery and other industries.

Common examples of heat exchangers familiar to use in day-to-day life are automobile radiators, condensers, evaporators and oil coolers.

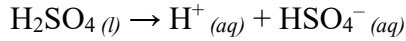
Based on our heavy-duty heat transfer requirements, we have selected shell and tube type heat exchanger.

Shell and Tube Heat Exchanger Designing:

In process industries, more than 90% of heat exchangers used are shell and tube type. The shell and tube heat exchangers are “work horses” of industrial process heat transfer. They are the first choice because of well-established procedures for design and manufacture from a wide variety of materials, many years of satisfactory service, and availability of codes and standards for design and fabrication. They are produced in widest variety of sized and styles.

5.1.1 Designing

Designing of a shell and tube heat exchanger depends upon the heat removed from a hot stream using a cold stream. In our process of dilution of sulphuric acid from 98% to 70%, the reaction carried out and enthalpy values are as follows. When we add concentrated sulphuric acid to water the reaction, following reaction takes place:



Given that the standard state heats of formation for $\text{H}_2\text{SO}_4(l)$, $\text{H}^+(aq)$, and $\text{HSO}_4^-(aq)$ are -813.989 kJ/mol, 0 kJ/mol (defined), and -885.75 kJ/mol, respectively.

$$\begin{aligned}\Delta H^o &= [\Delta H^o_{f, \text{HSO}_4^-} + \Delta H^o_{f, \text{H}^+}] - \Delta H^o_{f, \text{H}_2\text{SO}_4} \\ &= [(-885.75 + 0) - (-813.989)] \\ &= -71.76 \text{ kJ/mol}\end{aligned}$$

Mass of sulphuric acid = 7430 kg/hr

Molar mass of sulphuric acid = 98 kg/kmol

Mass of water added of dilution = 2985 kg/hr

$$\text{Moles of sulphuric acid per hour} = \frac{7430 \text{ kg/hr}}{98 \text{ kg/kmol}} = 75.8 \text{ kmol/hr}$$

Enthalpy of stream = 75.8×10^3 mol/hr * -71.76 kJ/mol = -543940 kJ/hr (151 kW)

LMTD:

$$\text{LMTD} = \frac{(T_i - t_o) - (T_o - t_i)}{\ln \frac{T_i - t_i}{T_o - t_i}}$$

LMTD = 64 °C

From Figure below, for 1 shell pass and 2 tube passes using values of S and R, [5]

$$R = \frac{T_{hi} - T_{ho}}{T_{co} - T_{ci}}$$

$$S = \frac{T_{co} - T_{ci}}{T_{hi} - T_{ci}}$$

Figure 5.1: Formulas for calculation of R and S values

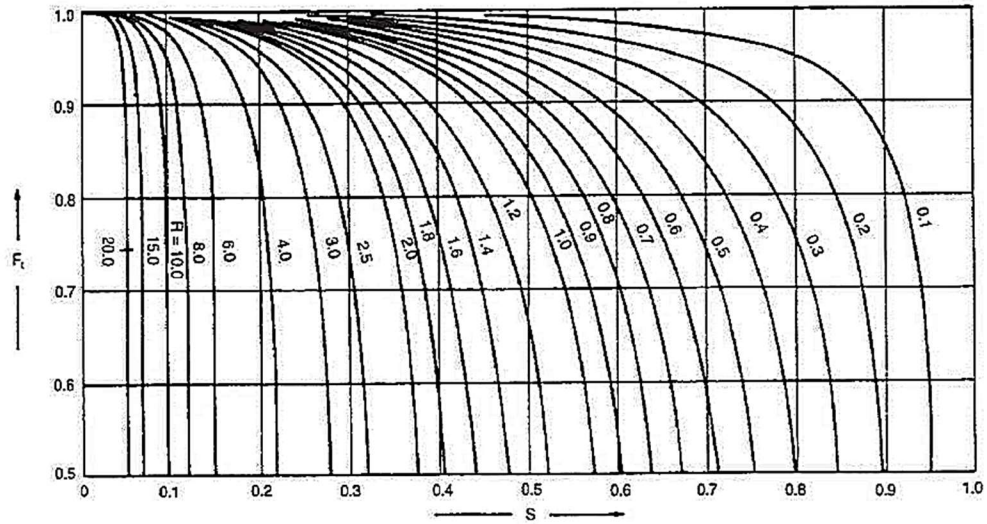


Figure 5.2: F_T Values using R and S

We get $F_t = 0.905$

$$\Delta t_m = F_t * \text{LMTD} = 0.905 * 64$$

$$= 57.9 \text{ } ^\circ\text{C}$$

From table (8) of D.Q.Kern, overall U_D of aqueous solution and water system is in the range of $800\text{-}1500 \text{ W/m}^2 \cdot ^\circ\text{C}$ [6].

Table 12: U_D values for hot and cold fluids

Hot fluid	Cold fluid	U ($W/m^2\text{ }^\circ C$)
<i>Heat exchangers</i>		
Water	Water	800–1500
Organic solvents	Organic solvents	100–300
Light oils	Light oils	100–400
Heavy oils	Heavy oils	50–300
Gases	Gases	10–50
<i>Coolers</i>		
Organic solvents	Water	250–750
Light oils	Water	350–900
Heavy oils	Water	60–300
Gases	Water	20–300
Organic solvents	Brine	150–500
Water	Brine	600–1200
Gases	Brine	15–250
<i>Heaters</i>		
Steam	Water	1500–4000
Steam	Organic solvents	500–1000
Steam	Light oils	300–900
Steam	Heavy oils	60–450
Steam	Gases	30–300
Dowtherm	Heavy oils	50–300
Dowtherm	Gases	20–200
Flue gases	Steam	30–100
Flue	Hydrocarbon vapours	30–100
<i>Condensers</i>		
Aqueous vapours	Water	1000–1500
Organic vapours	Water	700–1000
Organics (some non-condensables)	Water	500–700
Vacuum condensers	Water	200–500
<i>Vaporisers</i>		
Steam	Aqueous solutions	1000–1500
Steam	Light organics	900–1200
Steam	Heavy organics	600–900

Assuming U_D of our system to be $1400 \text{ W/m}^2 \cdot \text{ }^\circ C$

Now, applying formula to find out the area required for required heat transfer:

$$Q = U_D \cdot A \cdot \Delta t_m$$

Where

Q = Heat Duty

A = Area of heat transfer

$\Delta t_m = \text{Corrected LMTD}$

$U_D = \text{Overall design heat transfer co-efficient}$

Putting the values

$$-543940 = 1400 * A * 57.9$$

$$A = 6.71 \text{ m}^2$$

So, area of heat transfer required = 6.71 m^2

Selecting

1 shell pass and 2 tube passes

Outer Diameter of tube = $d_o = 15 \text{ mm}$

Length of tube = $L = 3.5 \text{ m}$

Area of one tube = $\pi \times L \times d_o = 0.16 \text{ m}^2$

Number of tubes = Heat transfer area / area of one tube = $6.71/0.16 = 42 \text{ tubes}$

Use triangular pitch,

Pitch = $P_t = 1.25 \times d_o = 18.75 \text{ mm}$

Bundle diameter = $D_b = d_o (N_t/K_1)^{1/n_1}$

K_1 and n_1 are constants. For 1 shell pass their values are,

$$K_1 = 0.249, n_1 = 2.207$$

$$D_b = 151.17 \text{ mm}$$

Clearance for shell diameter = 0.088 m (assumption)

Shell Diameter = Bundle Diameter + Clearance

$$\text{Shell Diameter} = D_o = 151.17 + 88 = 2410.53 \text{ mm}$$

$$\text{Baffle spacing} = 0.5 \times D_o = 0.5 \times 241.53 = 1200.58 \text{ mm}$$

$$\text{No of baffles} = \text{length of tube/baffle spacing} = 29$$

5.2 Den Reactor Design

Classification of Reactors:

Reactors can be classified in two kinds:

- 1) Depending upon the nature of the reactants and products
 - a) Homogenous Reactors (only one phase is present usually gas or liquid)
 - b) Heterogeneous Reactors (two or possibly three phases)
- 2) Depending upon the mode of reaction
 - a) Batch Reactors (for small scale)
 - b) Continuous Reactors (for large scale)

Reactor Selection Criteria:

1. Conversion
2. Selectivity
3. Productivity
4. Yield
5. Heat exchange
6. Mixing
7. Catalyst distribution
8. Hold up time
9. Availability
10. Compatibility
11. Energy utilization
12. Safety
13. Economics

Nature of Material Handled:

- The slurry has a nature to go continuously thicken.
- There is danger of solidification of slurry inside reactor.
- To prevent solidification, slurry has to be removed continuously

Why Den Reactor:

- In this process, intense mixing is required due to high density differences
- Semi solid formation is required during the reaction so centrifugation require for movement of semi solid
- Autogenously size reduction due to tumbling, centrifugation and smearing
- More effective for highly viscous slurries
- High heat and mass transfer coefficients
- Useful for slow reaction
- Uniform, good temperature control

5.2.1 Design Calculations

Table 13: Volumetric flowrate in Den Reactor

Components	Flowrate (kg/hr)	Density (kg/m ³)	Volumetric flowrate (m ³ /hr)
Ca ₃ (PO ₄) ₂	7434.55	2220	3.34
CaCO ₃	381.9	2710	0.140
CaF ₂	739.6	3180	0.232
CaSO ₄ .2H ₂ O	90.35	2300	0.039
MgO	30	2800	0.0107
Na ₂ O	37.2	2700	0.013
SiO ₂	2678.35	2634	1.01
Al ₂ O ₃	288.15	4000	0.072
Fe ₂ O ₃	180.15	5277	0.00341
K ₂ O	60.05	2560	0.023
H ₂ O	3000	1000	3
H ₂ SO ₄	7430	1840	4.038

Total volumetric flowrate = 11.91 m³/hr

Residence time = 2 hours

a. Volume of Reactor:

$$\begin{aligned}\text{Volume of reactor} &= \text{residence time} * \text{volumetric flowrate} \\ &= 2 * 11.91 \\ &= 23.82 \text{ m}^3\end{aligned}$$

As the volume of the reactor is not fully filled and 30% is kept as vacant space, which is called, fill rate. [7]

$$\text{So, total volume of reactor} = 23.82 * 1.30 = 30.966 \approx 31 \text{ m}^3$$

b. Diameter of Reactor:

$L/d = 4$ (Ref. from the cost curves and formula of vessel design handbook)

$$\text{Volume} = \text{Area} * \text{Length}$$

$$\text{Volume} = 31 \text{ m}^3$$

$$\text{Diameter} = 2.4 \text{ m}$$

$$\text{Length} = 6.8 \text{ m}$$

c. Wall Thickness:

$$T = [P * r_i / S e_j - 0.62P] + C_c$$

Where,

T= thickness

P=pressure design

r_i =internal diameter

e_j =efficiency for joint

C_c =corrosion allowance

S=maximum allowable stress

Design pressure=1.5 atm

Inside diameter = 2.4 m = 2400 mm

Allowable working stress = S = 932.64 atm (carbon steel)

Efficiency of joint (if spot examined) = $e_j = 0.85$

Corrosion allowance = 0.3 mm

Putting all the values, thickness = 6.6 mm

Allowance for material of construction of reactor is 300%

Therefore, thickness = 19.8 mm

d. Rotation of reactor:

From literature round per minute (rpm) of Den Reactor is 1 rpm.

Rotation of reactor is necessary so that mixing can be done and a 4 turbine blade cutter impeller is also used so that the material could be mixed well and can be removed from reactor before it solidifies inside the reactor and chokes the reactor.

To calculate rpm of this impeller, we use formula:

$$N_1 d_1 = n_2 d_2$$

Inner diameter= 2.4 m

Outer diameter=2.42 m

$$n_2 = \frac{1 \cdot 2.4}{2.42} = 0.991 \text{ rpm}$$

5.3 Spray Tower Design

A scrubber or spray tower is used after den reactor so that all corrosive gases evolving from reactor can be dissolved into water before venting the gases into atmosphere. [8]

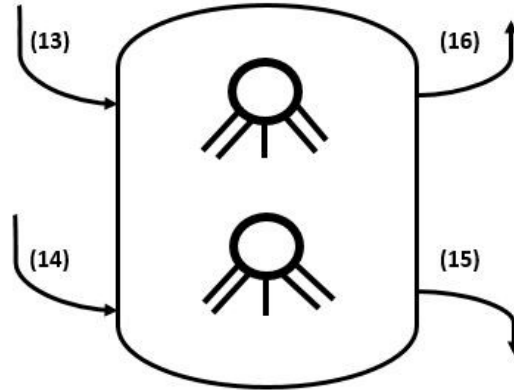


Figure 5.3: Spray Tower

Stream 13 is water sprayed from top.

Stream 14 is the gases coming from reactor.

Stream 15 is acidic water that includes all hazardous gases dissolved in it.

Stream 16 is the non-hazardous gases venting in atmosphere.

Table 14: Flowrates of streams of spray tower

Stream	Water (kg/hr)	CO ₂ (kg/hr)	SiF ₄ (kg/hr)
13	13500	-	-
14	481	290	850
15	13881	90	845
16	100	200	5

5.3.1 Steps involved in Designing

The major steps involved in the design of spray tower are:

- Perform material and energy balance across the tower.

- Collect the data available.
- Calculate the diameter.
- Calculate the circulation rate of spray tower.
- Calculate the number of overall spray units.
- Calculate the mass transfer coefficient of existing unit.
- Calculate the volume of tower.
- Calculate the height of tower.
- Calculate the pressure drop of tower.

Type of scrubber:

Spray tower

Position:

Vertical

a. Tower Diameter:

For vertical Spray Tower:

Flowrate of gases from reactor = 1621 kg/hr

Volumetric flowrate = 658 m³/hr = 10.9 m³/sec

Maximum permissible velocity through vertical spray tower = 2.3 m/s

In order to avoid excessive entrainment, we take the 60% of maximum velocity:

So, $u_g = 0.6 * u_{gmax} = 1.38$ m/s

$$D_i = (4 * q_v / \pi * u_g)^{1/2}$$

$$= 3.17 \text{ m}$$

b. Number of gas phase transfer units:

$$NG = \frac{y_1 - y_2}{y_2}$$

y_1 = mole fraction of solute in feed

y_2 = mole fraction of solute in product

NG = 9

c. Circulation Rate of water:

For vertical spray tower:

From an existing unit, following data is available:

Flowrate of gases = 12,000 m³/hr

Temperature of incoming gases = 45 -55 °C

Circulation rate of water = 25 m³/hr

Tower Height = 6.10 m

Tower diameter = 1.52 m

Pressure drop max = 130 mm WC

Based on these calculations, rate required for new tower will be calculated as:

SiF₄ concentration in inlet = 3000 ppm

SiF₄ concentration in outlet = 300 ppm

Liquid flowrate required = 13.5 m³/hr

d. Height of Tower:

For vertical spray tower:

$$V = (NG * GM / K_{ga})$$

For existing tower,

$$NG = 3600 - 300 / 300$$

$$= 11$$

$$V = 11 \text{ m}^3$$

$$Gm = 12000 / 22.414$$

$$= 535.38 \text{ kmol/hr}$$

$$K_{ga} \text{ for existing tower} = 11 * 0.1487 / 11$$

$$= 0.147 \text{ kmol/m}^3 \cdot \text{s}$$

Volume of spray section required for new tower:

$$V = (NG * GM / K_{ga})$$

$$Gm = 654 / 22.414 = 29.17 \text{ kmol/hr}$$

Putting the values:

$$V = 1 \text{ m}^3$$

$$\text{Volume} = \pi * d^2 / 4 * h$$

$$H = \text{height of tower} = 5.84 \text{ m}$$

e. Pressure drop calculations:

For vertical spray tower:

$$\Delta P = 130 * G_m^{1.84} * D_i^{-4.84} * L$$

Putting the values:

$$\Delta P = 54 \text{ mm WC}$$

So, our pressure drop is within the acceptable limits

f. Drift and Evaporation losses:

Loss of water = drift losses + evaporation losses

$$= 1.2\% \text{ of circulation rate}$$

$$= 0.012 * 13.5$$

$$= 0.162 \text{ m}^3/\text{hr}$$

$$= 162 \text{ kg/hr}$$

Results:

Vertical spray tower:

Diameter of tower = 3.17 m

Height of tower = 5.84 m

Volume of spray section = 1 m³

Pressure drop = 54 mm WC = 0.629 kPa

Circulation rate = 13.5 m³/hr

CHAPTER 6: Economic Analysis

6.1 Introduction

Cost assessment is an independent profession. But the design engineer must be able to determine between alternative designs to evaluate the project quickly and estimate the rough cost. Chemical plants are constructed to generate profit, and before a profitability of a project can be assessed an estimate is made of the necessary investment and cost of production. An acceptable plant design must be able to operate under conditions that produce profit.

Direct plant costs, such as those for raw materials, work, and equipment, must be allocated with the capital. In addition to direct expenditure there are a number of other indirect expenditures, which have to be included in order to obtain a full analysis of the total cost. For example, administrative wage, product distribution costs, and interplant communication costs are some of these indirect expenditures.

Specific funding for the purchase and installation of equipment must be provided before the plant is operated. Fixed capital investments are the capital necessary to supply the necessary installations, the work principal and a total capital investment for the operation of the installation, are the total capital investments.

6.2 Capital Investment

The accuracy of the assessment depends on the number of available design details. Estimates on capital costs can be widely classified according to their correctness and purpose in three types:

1. Preliminary (approximate) predictions, typically 30% precision, used to make tough choices among the design alternatives in initial feasibility studies. The cost data and design details are limited.
2. The estimates for approval (budgeting), typically 10-15% accuracy. These are used to authorize funds to carry out the design to the extent to which a detailed and

accurate assessment is possible. Approval may also include money to cover cancellation charges for any lengthy delivery equipment ordered at this design stage in order to avoid project delays. Such an estimate could be used in a contracting organization, with a large contingency factor, to get the tender price. However, it would normally be necessary, if time allowed, to achieve accuracy of around 5% and to make a more detailed estimate. Where an enterprise has cost data and experience.

3. Detailed (quotation), accuracy of 5-10% used for project cost control and fixed price contract estimates. These are based on the process design, firm equipment quotations and a detailed division and cost estimation (or almost complete). The estimate costs rise from around 0.1% of the total cost to 30% accuracy, to approximately 2% for a detailed estimate with $\pm 5\%$ accuracy.

6.3 Fixed Capital Investment

The total cost of the plant is fixed capital ready for launch. It is the costs paid to the entrepreneurs.

It covers the costs of:

1. Design, engineering and other supervisory services.
2. All equipment items and their assembly.
3. All systems of piping, equipment, and control.
4. Structures and buildings.
5. Helping facilities like utilities, land and civil engineering.

Only once the costs are recovered, other than the scrap value, at the end of the life of the project. Working capital is the additional investment necessary to start and operate the plant, in addition to fixed capital, in order to earn revenue.

It includes the cost of:

1. Launching.
2. Catalyst loads initial.
3. In this process, raw materials and intermediates.

4. Inventories of finished product.

At the end of the project, most work capital is recovered. For a project, the total investment needed is the amount of fixed and operating capital.

The working capital of a single product can range from 5% of fixed capital for a simple process with little or no finished product storage to 30% for a process which produces a wide variety of product classes for an advance market, such as synthetic fibers. A typical figure of 15% of fixed capital is used for petrochemical plants. Will be using a figure of 20 percent.

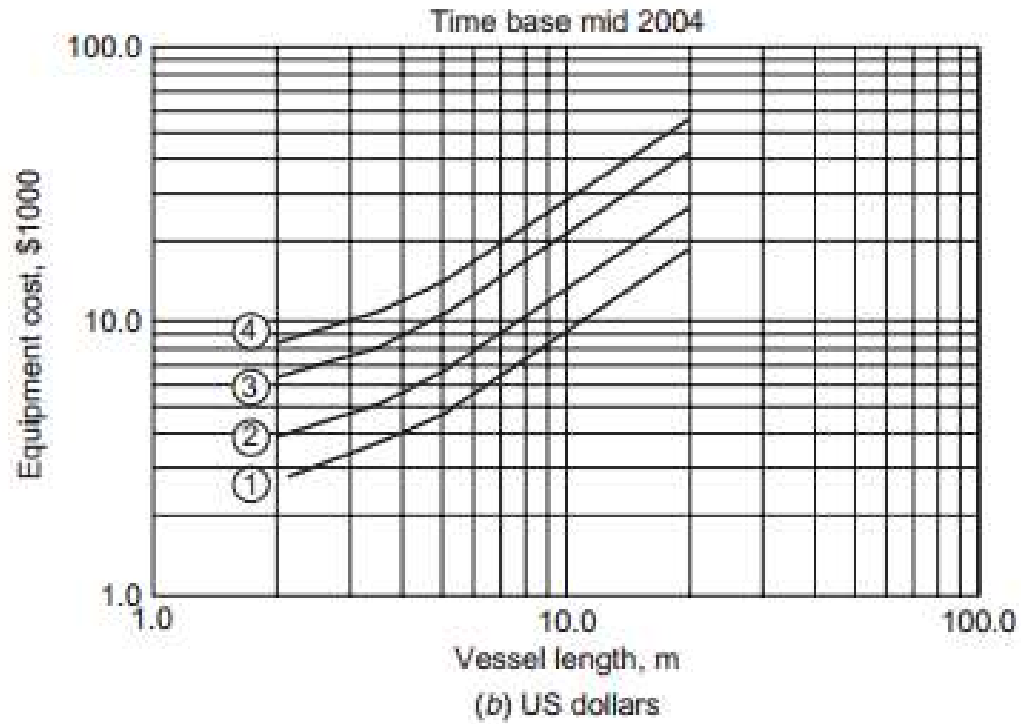
6.4 Cost Estimates for the Proposed Design

The table for base costs, size units, and indices for different equipment was used from Richardson Coulson's Chemical Engineering Volume 6. The table is as follows:

Table 15: Purchase cost of industrial equipment and cost factors

Equipment	Size unit, S	Size range	Constant C,£	C,\$	Index n	Comment
<i>Agitators</i>						
Propeller	driver	5-75	1200	1900	0.5	
Turbine	power, kW		1800	3000	0.5	
<i>Boilers</i>						
Packaged						oil or gas fired
up to 10 bar	kg/ft steam	$(5-50) \times 10^3$	70	120	0.8	
10 to 60 bar			60	100	0.8	
<i>Centrifuges</i>						
Horizontal basket	dia., m	0.5-1.0	35,000	58,000	1.3	carbon steel
Vertical basket			35,000	58,000	1.0	$\times 1.7$ for ss
<i>Compressors</i>						
Centrifugal	driver power, kW	20-500	1160	1920	0.8	electric, max. press. 50 bar
Reciprocating			1600	2700	0.8	
<i>Conveyors</i>						
Belt	length, m	2-40				
0.5 m wide			1200	1900	0.75	
1.0 m wide			1800	2900	0.75	
<i>Crushers</i>						
Cone	t/h	20-200	2300	3800	0.85	
Pulverisers	kg/h		2000	3400	0.35	
<i>Dryers</i>						
Rotary	area, m ²	5-30	21,000	35,000	0.45	direct
Pan		2-10	4700	7700	0.35	gas fired
<i>Evaporators</i>						
Vertical tube	area, m ²	10-100	12,000	20,000	0.53	carbon steel
Falling film			6500	10,000	0.52	
<i>Filters</i>						
Plate and frame	area, m ²	5-50	5400	8800	0.6	cast iron
Vacuum drum		1-10	21,000	34,000	0.6	carbon steel
<i>Furnaces</i>						
Process						
Cylindrical	heat abs, kW	10^3-10^4	330	540	0.77	carbon steel
Box		10^3-10^5	340	560	0.77	$\times 2.0$ ss
<i>Reactors</i>						
Jacketed, agitated	capacity, m ³	3-30	9300	15,000	0.40	carbon steel
			18,500	31,000	0.45	glass lined
<i>Tanks</i>						
Process	capacity, m ³					
vertical		1-50	1450	2400	0.6	atmos. press.
horizontal		10-100	1750	2900	0.6	carbon steel
Storage						
floating roof		50-8000	2500	4350	0.55	$\times 2$ for
cone roof		50-8000	1400	2300	0.55	stainless

For tanks and horizontal pressure vessels, we use the following table from Richardson Coulson's Chemical Engineering, Volume 6 [5]:



Diameter, m		Material factors	Pressure factors
①—0.5	③—2.0	C.S. × 1.0	1–5 bar × 1.0
②—1.0	④—3.0	S.S. × 2.0	5–10 × 1.1
		Monel × 3.4	10–20 × 1.2
		S.S. clad × 1.5	20–30 × 1.4
		Monel × 2.1	30–40 × 1.6
		clad	40–50 × 1.8
			50–60 × 2.2

Temperature up to 300°C

Figure 6.1: Graph for Cost of Vessels

6.4.1 List of Equipment

The list of equipment in the plant and their quantities are as follows. These costs attribute to the total cost of equipment and are further used in calculations.

Table 16: Details of Equipment

List of Equipment	Number of Equipment
Crusher	1
Roller Mill	1
Pre-Mixer	1
Heat Exchanger	1
Mixer	1
Den Reactor	1
Scrubber	1
Settler	1

6.4.2 Total Capital Investment

The Fixed Capital Cost of the Plant is made up of the Plant Direct Cost and the Plant Indirect costs, which are as follows.

Fixed Capital Cost = Plant Direct Costs + Indirect Cost

Table 17: Total Plant Direct Cost

Costs	Amount
Equipment Cost	Rs. 194957831
Delivery Cost	Rs. 214453614
Installation Cost	Rs. 83636323
Processing and Piping	Rs. 21445359
Instrumentation	Rs. 55757941
Electrical Facilities	Rs. 21445359
Building Cost	Rs. 62191547
Total Plant Direct Cost	Rs. 653888570

Total Plant Indirect Cost:

Table 18: Total Plant Indirect Cost

Costs	Amount
Engineering and Supervision	Rs. 68625155
Construction Expenses	Rs. 72914229
Contractor Fee	Rs. 40746185
Legal Expenses	Rs. 8578143
Contingency	Rs. 79347837
Total Plant Indirect Cost	Rs. 124405681

Thus, the Fixed Capital Investment = Plant Direct Cost + Plant Indirect Cost

$$= \text{Rs. } 949,834,559$$

Fixed Capital Investment = Working Capital (20 % of Total Capital Investment)

$$= 0.2 * (\text{TCI})$$

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

Total Capital Investment = Rs. 1, 187, 293, 200

6.4.3 Production Cost

Total Production Cost = Manufacturing Cost + General Expenses

1) Manufacturing Cost:

Raw Material Cost

Rock Phosphate = Rs. 1, 736, 822

Sulphuric Acid = Rs. 2, 293, 135

Total Raw Material Cost = Rs. 4, 029, 371

Total Direct Production Cost= Rs. 319, 028, 391

Total Fixed Charges = Rs. 99, 230, 221

Manufacturing Cost = Rs. 467, 300, 000

2) General Expenses:

Administration rate + Distribution and Marketing Cost + R&D Cost = Rs. 23, 113, 924

Total Production Cost = Manufacturing Cost + General Expenses = Rs. 490, 413, 394

6.4.4 Annual Sales

Cost of Single Super Phosphate per Ton: Rs. 7350 (In Pakistan)

Total Annual Sales = Rs. 7350 x 500 (per day) x 365 days = Rs.134,1375,000

Gross Earning = Total Sales – Total Production Cost

= Rs. 850, 961,006.

Payback Period = Total Investment/Annual Cash Flow

= 2 years and 3 months

CHAPTER 7: HAZOP Analysis

7.1 Introduction

The Hazard and Operability Study (HAZOP) is a standard hazard analysis technique used in the preliminary safety assessment of new systems or modifications to existing ones. The HAZOP study is a detailed examination by a group of specialists, of components within a system to determine what would happen if that component were to operated outside its normal design mode. The results of this study is then assessed and noted down on study forums so that the information can come handy in the hour of need to the industry workers. The categories of information entered on these forms can vary from industry to industry.

The HAZOP technique was initially developed to analyze chemical process systems, but has later been extended to other types of systems and also to complex operations. A HAZOP is a qualitative technique based on guidewords and is carried out by a multi-disciplinary team (HAZOP team) during a set of meetings and aims to stimulate the imagination of participants to identify potential hazards and operability problems [9].

7.2 Objectives of HAZOP

1. To familiarize the study team with the design information available.
2. To ensure that a systematic study is made of the areas of significant hazard potential.
3. To identify and study features of the design that influence the probability of a hazardous incident occurring.
4. To identify pertinent design information not available to team.
5. To provide a mechanism for feedback to the client of the study team detailed comments.

7.3 Keywords used in HAZOP Study

Keywords are divided into two sub-sets. These are used to focus the attention of the team upon deviations and their possible causes

7.3.1 Primary Keywords

Focus attention upon a particular aspect of the design intent or an associated process condition or parameter.

Table 19: Primary Keywords for HAZOP Study

Flow	Pressure
Separate (settle, filter, centrifuge)	React
Reduce (grind, crush, etc.)	Corrode
Temperature	Level
Composition	Mix
Absorb	Erode

7.3.2 Secondary Keywords

Secondary keywords is a list of keywords which when combine with a primary keyword suggest possible deviations.

Table 20: Secondary Keywords for HAZOP Study

Word	Meaning
No	The design intent does not occur or the operational aspect is not achievable
Less	A quantitative increase in the design intent occurs (e.g. pressure is less)
More	A quantitative increase in the design intent occurs (e.g. temperature is more)
Reverse	The opposite of the design intent occurs (e.g. flow reverse)
Also	The design intent is completely fulfilled, but in addition some other related activity occurs (e.g. indicating contamination in a product stream, or level/also meaning

	material in a tank or vessel which should not be there)
Other	The activity occurs but not in the way invented (e.g. flow other could indicate a leak or product flowing where it should not)
Fluctuations	The design intention is achieved only part of the time (e.g. an air lock in a pipeline might result in flow/fluctuation)
Early	Used when studying sequential operations, this would indicate that a step is started at the wrong time or done out of sequence

7.4 Conducting HAZOP Study:

The steps involved in conducting a HAZOP study are as follows:

1. Specify the objective of study
2. Selection of team for HAZOP study
3. Make preparatory work
4. Assemble the data
5. Understanding the subject
6. Planning the sequence
7. Mark the drawings
8. Devise a list of appropriate keywords
9. Preparing node heading and agenda
10. Running a HAZOP study
11. Summarizing the results
12. The Report

7.5 HAZOP Analysis on equipment

We performed HAZOP analysis on our pre-mixer inlet and heat exchanger outlet. The results are as follows:

7.5.1 Pre-Mixer

Table 21: HAZOP Analysis on Pre-Mixer Inlet

Deviation	Causes	Consequences	Recommendations
More flow	<ul style="list-style-type: none"> Level control valves (LCV) fails open 	<ul style="list-style-type: none"> Required rock and Sulphuric acid ratio not achieved Tank overflows 	<ul style="list-style-type: none"> Level indicator transmitter (LIT) installation at critical points Regular checking and maintenance
Less flow	<ul style="list-style-type: none"> Blockage or probable leakage 	<ul style="list-style-type: none"> Required rock and Sulphuric acid ratio not achieved Material loss 	<ul style="list-style-type: none"> Install alarm on LIC Regular checking and maintenance

7.5.2 Heat Exchanger Outlet

Table 22: HAZOP Analysis on Heat Exchanger Outlet

Deviation	Causes	Consequences	Recommendations
More Temperature	<ul style="list-style-type: none"> More flow rate of Sulphuric acid (valve/ flow controller fails) Summer conditions Less flowrate of cooling water (valve/ 	<ul style="list-style-type: none"> High pressure in line Cooling objective not met 	<ul style="list-style-type: none"> Install high temperature warning alarm Install Temperature sensor

	flow controller fails/ leakage)		<ul style="list-style-type: none"> • Regular checking for pipe leakage/ valves/ controller
Less Temperature	<ul style="list-style-type: none"> • Winter conditions • Less flowrate of hot fluid (valve/ flow controller fails/ leakage) • Inadequate heat transfer due to corroded tubes • More flowrate of cooling water (valve/ flow controller fails) 	<ul style="list-style-type: none"> • Freezing of lines • Loss of Sulphuric acid due to leakage 	<ul style="list-style-type: none"> • Install TC • Regular maintenance and cleaning of heat exchanger • Regular checking for leakage/ valves/ controller)

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