

Title of the Project
Production of Nitro Phosphate by Odda Process



KHWAJA FAREED
UEIT
RAHIM YAR KHAN

(Session2018-2022)

Submitted by:

Ahmad Ali	CHEN18111020
Farhan Hassan	CHEN18111008
Falak Shair	CHEN18111039

Submitted to:

Department of Chemical Engineering

Submission date:

1-Jun-22

**KHWAJA FAREED UNIVERSTY OF ENGINEERING AND INFORMATION
TECHNOLOGY**

(June 2022)

A PROJECT DESIGN REPORT

ON

Production of Nitro Phosphate by Odda Process

This Project is submitted to the Department of Chemical Engineering for the partial fulfillment of the bachelor's degree of BS chemical engineering.

PROJECT ADVISOR

APPROVED ON

INTERNAL EXAMINER

EXTERNAL EXAMINER

HEAD OF CHEMICAL ENGINEERING DEPARTMENT

**KHWAJA FAREED UNIVERSITY OF ENGINEERING AND INFORMATION
TECHNOLOGY**

Acknowledgement

The research project titled "Production of Nitro phosphate by Odda Process" was successfully completed in the Institute of Chemical and Environmental Engineering of the Khwaja Fareed University of Engineering and Information Technology under the Pakistan Engineering Council (PEC) Annual Award of Final Year Design Projects (FYDP) for the year 2022-2023. The Project was supervised by Engr. Dr. Tausif Ahmad and Engr. Aleena Naqvi.

Abstract

Nitro phosphate plant operating by using Odda process is designed to use different compositions of rock phosphate and dilute nitric acid. Such industrial method is used to manufacture ammonium, calcium hydro phosphate and calcium nitrate. Nitro Phosphate plant is design to use low grade different grades of rock phosphate without compromising on product quality to maximize profitability. Plant produce pilled fertilizer in which each prills has evenly distributed amounts of nitrogen (22%) and phosphorus (20%).plant process used urea solution to control emissions of NOx and antifoams prevent foaming

Table of Contents

Chapter no. 2 Literature review	4
2.67 Prilling:	14
3.1 Production capacity of plant = 135000 tons/year.....	23
3.2 Material balance around Acidulation/digester	23
3.3 Material balance around Crystallizer	25
3.4 Material balance around Filter Drum.....	26
3.5 Material balance around Neutralizer.....	27
3.6 Material balance around Evaporator	29
Chapter 5 Design section	42
5.1 Introduction of Reactor:	42
5.2 Continuous Stirrer Tank Reactor (CSTR):.....	44
5.3 Continuous Stirred Tank Reactor Design:	44
5.4 Material of Construction:	45
Continuous stirred tank reactor	45
Figure 4. Continous stirrer tank reactor	45
5.51 Design equation of C.S.T.R:	45
5.8 Design of Heat Exchanger:.....	54
Fluid Allocation	55
Design Steps:	55
Chapter 6 Instrumentation and Control.....	69
6.1 Instrumentation:	69
6.2 Control:	69
6.4 Fundamentals of Control System.....	70
6.5 Approaches to Control:	70
6.6 Combinations of Control Actions	70
6.7 Control Loops:	70
6.8 Reactor:	70
6.9 Heat Exchanger:.....	71
6.10 Pump:	72
6.11 Scrubber:	72
7.1 Economics Analysis:.....	74
7.2Purchase Equipment Cost:	74
7.21 Storage Tanks:	74
7.22 Continuous Stirred Tank Reactor.....	75
7.23 Shell and Tube Heat Exchanger.....	76
7.24 Evaporator.....	76
7.25 Centrifugal Pump:	77
7.3 Total Purchased Equipment Cost:.....	80

7.4 Direct Cost:	81
7.5 Indirect Cost:.....	81
7.6 Total Capital Investment:.....	81
7.7 Plant Cost:.....	81
7.8 Raw Material Cost:	81
Total Product Cost=?	81
7.9 Rate of Return:.....	82
7.9a Pay Back Period:.....	82
2. Conclusion	Error! Bookmark not defined.
Appendix.....	Error! Bookmark not defined.

List of Tables

List of Tables must be provided with their proper numbering and captions

Table 1. Analysis of Phosphate Rock from Several Sources	
Table 2. Pollutant Loads in Effluents, Nitro Phosphate Process.....	
Table 3. Emissions Loadings for Nitro Phosphate Process.....	
Table 4. Particle Strength of Prilled NPK Fertilizers.....	
Table 5. Caking Index for NPK with 0.5% H ₂ O.....	
Table 6. Adherence Properties of Different Anti Caking Agent.....	
Table 7. Material Balance Around the Acidulation/ Digester	
Table 8. Material Balance Around the Crystallizer.....	
Table 9. Material Balance Around the Filter Drum.....	
Table 10. Material Balance Around the Neutralizer	
Table 11. Material Balance Around the Evaporator	
Table 12. Available Data for Energy Balance	
Table 13. Energy Balance Around the Dissolving Reactor	
Table 14. Energy Balance Around the Heat Exchanger	
Table 15. Energy Balance Around the Crystallizer	
Table 16. Energy Balance Around the Neutralizer	
Table 17. Energy Balance Around the Evaporator	
Table 18. Reaction Kinetic Data	
Table 19. Specification of Heat Exchanger	
Table 20. Slandered Dimensions for Steel Tube	
Table 21. Thermal Conductivity of Material	

List of Figures

List of figures must be provided with their proper numbering and captions

Figure 1. Process and Instrumentation Diagram
Figure 2. Chemical Reaction Theory
Figure 3. Continuous Stirrer Tank Reactor
Figure 4. Dissolving Reactor
Figure 5. Control Loop for Reactor
Figure 6. Instrumentation and Controlled Diagram for Heat Exchanger
Figure 7. Instrumentation And Controlled Diagram For Pump

Chapter No. 1 Introduction

1.1 Fertilizer Introduction

We fulfill our agricultural needs from soil. Due to use of land often, it affects soil composition. In the past, farmers used manure as typical fertilizer for crops growth. They have noticed that the land where animals gathered has enhanced crop growth rather than other area. As time proceed, manufacturing of organic fertilizers starts, and it replace inorganic fertilizer means manure. Fertilizers were added to ground to give soil necessary elements. Fertilizers are basically chemical substances to the crops which provide essential elements to them to increase their yield. Essential nutrients for crops growth are nitrogen, potassium, phosphorus, calcium, magnesium etc.

Main nutrients are nitrogen, potassium, and phosphorus(NPK). Fertilizers and organic matter are rich of NPK.

1.2 Soil Composition

The composition of the soil is crucial to nutrient management. Soil water is what easily offers nutrients for plant uptake, whereas soil minerals and organic matter keep and store nutrients. Because many of the microorganisms that reside in the soil require air to carry out the biological processes that release extra nutrients into the soil, soil air plays an important function.

Minerals, organic matter, water, and air are the four fundamental components of soil. The average soil contains 45 percent minerals, 5 percent organic matter, 20-30 percent water, and 20-30 percent air. These figures are, at best, generalizations. In actuality, soil is a complex and dynamic system. The soil's composition can change on a daily basis, depending on a variety of factors such as water supply, agricultural techniques, and/or soil type.(Sachan 2017)

1.3 Fertilizer Suitability for Acidic Soil

PH scale of soils 0 to 14. Acidity is a property of soils with a pH less than 7. More than 7 represent alkaline nature of soil. 7 means natural. Nitrogen as main nutrient increase the acidity of soil. Fertilizers, manures, and legumes all contain or produce ammonium. Unless the plant takes the ammonium ions directly, this raises soil acidity. The higher the nitrogen fertilizer rate, the more acidic the soil.

Nitrogenous fertilizers are following.

- Ammonium nitrate N34.
- Calcium ammonium nitrate N27.
- Nitrogen fertilizers with calcium.
- Nitrogen fertilizers with Sulphur.
- Urea N46.
- Slow release nitrogen fertilizer LITFERT StabillioNR Premium.
- Urea with potassium humate.
- Liquid nitrogen fertilizers (UAN) N32.

Rock phosphate is more soluble in acidic soils (pH 5.5), it is more useful in acidic soils than in alkaline soils. The soluble form of phosphorus is available for plant roots to absorb.

$\text{Ca}_{10}(\text{PO}_4)_6(\text{X})_2$, where X is F, OH, or Cl, is the usual formula for pure rock phosphate. Apatites are the name for these minerals. Fluorapatite, which contains impurities such as CO_3 , Na, and Mg, is the most common rock phosphate mined. The phosphate concentration of phosphorite (or phosphate rock) varies dramatically, ranging from 4% to 20% phosphorus pentoxide (P_2O_5). Phosphate rock that is sold is enhanced (or "beneficiated") to at least 28%, and frequently more than 30% P_2O_5 . Washing, screening, de-liming, magnetic separation, and flotation are all methods for doing this.

Diammonium Phosphate (DAP), Monoammonium Phosphate (MAP), NPKs, and SSP are the most widely used phosphatic fertilisers. DAP is the most frequently used phosphorus fertilizer in the planet.

1.31 Diammonium Phosphate (DAP):

The most extensively used phosphorus fertilizer is diammonium phosphate (DAP). It's made up of two typical fertilizer ingredients ammonia and phosphoric acid and has a pretty high nutritional content. It's ideal for providing full phosphorus nutrition throughout crop growth and development, as well as a preliminary dosage of nitrogen and low Sulphur, to any agriculture crop. Before sowing, DAP is usually administered as a base dosage. The earliest seed germination occurs after 6 days, and roots begin to emerge. Nitrogen does not last longer than 36-48 hours in the soil.

The general formula for DAP is $(\text{NH}_4)_2\text{HPO}_4$. DAP is a fertilizer grade product that contains 18% nitrogen and 46% phosphorus (P^{2}O^5).

TVA began producing crystalline DAP in the late 1940s, and demonstration trials proved it to be an excellent high-analysis fertilizer. Others produced smaller quantities as a byproduct. When the TVA method for granular DAP of grade 18-20-0 (18-46-0) was invented in 1960-1961, it was relatively simple and dependable. (Young and Davis 2015)

1.32 Nitro phosphate(NP):

NP means nitro phosphate fertilizer having nitrogen and phosphorus as its main elements. About 1927, scientist name Erling Johnson invented the nitro phosphate process method on industrial scale production of nitrogen fertilizer. Such process was named as Odda process.

Phosphate rock + Nitric acid → Phosphoric acid + Calcium nitrate + Hydrofluoric acid

Both nutrients are required for crops growth for specific level. Nitro phosphate fertilizer has nitrogen 22% and phosphorus 20% in each of its grain. Naturally nitro phosphate fertilizer is acidic product having PH value of 3.5 is beneficial for soil that has required high PH.(European

Nitro phosphate fertilizer contain following properties.

- Such fertilizer contains 24 percent sulfur.
- This fertilizer is high explosive and hygroscopic. Therefore, it is packed in sealed bags until it is used.
- Urea and ammonium nitrate are less hygroscopic than nitro phosphate.
- Stored nitro phosphate tries to hard cake which is necessary to be broken.

- The molecular formula of nitro phosphate is NO_3P^{2-} having molecular weight of 140.98 g/mol. Nitro phosphates have ammonium nitrate as their main component.

Ammonium nitrate is mixed with different phosphate in a specific ratio in nitro phosphate. Mostly it is used in granular form. The chemical composition varies from product to product depending upon nutrients involved in the product. Because every product has its own requirements depending on soil nature or composition.

Nitro phosphate in granular form can be used directly to soil. Commonly it is spread over the entire soil surface. Ammonium nitrate in nitro phosphate fertilizer attracts moisture. Therefore, nitro phosphate containing ammonium nitrate is packed in watertight bags to prevent from moisture content. (Young and Davis 2015)

Why Nitro Phosphate is Preferable to Di-ammonium Phosphate Fertilizer?

NP fertilizer is superior to DAP in the following ways.

- The DAP fertilizer does not include potassium, but the NPK fertilizer does.
- It is debated if NPK fertilizer is preferable than DAP because the latter does not acidify soils.
- Raw material for nitro phosphate fertilizer are easily available in Pakistan than di-ammonium phosphate fertilizer which are imported.
- Nitro phosphate fertilizer has a cheaper manufacturing process than DAP fertilizer.

So the best option fertilizer for acidic soil is nitro phosphate fertilizer than other phosphorus fertilizers. (Nielsson n.d.)

Chapter No. 2 Literature review

2.1 General History of processed Phosphate Fertilizers:

Ground bones were the first phosphate fertilizer, and they were widely employed in Europe throughout the early nineteenth century. When animal bones became scarce, human bones were collected from battlefields and cemeteries. Sulfuric acid treatment of bones, as pioneered by Liebig, began around 1810 and quickly became mainstream practice. The product was made with diluted sulfuric acid. It was supplied in barrels made of wood. Later, other nutritious ingredients including ammonium sulphate, potash, and soda nitrate were added, resulting in the first fluid mixed fertilizer. Around 1830, the process evolved to include sulfuric acid treatment of the more available powdered phosphate rock. Superphosphate was the name of the solid product. Lawes created the first successful facility for the production of super phosphate in England in 1842.

By 1853, there were 14 manufacturers in the United Kingdom. By 1862, Lawes was producing 100 tonnes per day with a continuous super phosphate mixer. Ordinary superphosphate manufacturing (OSP).

It quickly spread to the United States and other parts of Europe. The history of concentrated or triple super phosphate (TSP) manufacturing coincided with the start of wet-process phosphoric acid production. The technique expanded to other European nations and the United States when the first commercial manufacturing was discovered in Germany. TSP was not widely used as a phosphate fertilizer until the 1950s, when many major factories were constructed. Beginning in the late 1930s, TVA began producing and distributing huge quantities of concentrated super phosphate manufactured using electric furnace acid, which led to widespread testing and commercial development of this higher analysis fertilizer.

Although ammonium phosphate had long been recognized as an efficient fertilizer, and modest amounts had been generated for fertilizer use on occasion, it was not widely used until TVA developed a feasible and cost-effective technology for granular diammonium phosphate (DAP) in 1961. This method became popular, and several big factories for producing the 18-20-0 (18-46-0) grade were erected in the United States, Europe, and subsequently Asia and South America.

In addition, TVA researchers devised a minimally modified technique for producing granular mono ammonium phosphate (MAP), which is widely used in Canada and Pakistan and is also manufactured elsewhere.

In the early 1930s, the development of nitric phosphate processes, in which phosphate rock is acidulated with nitric acid, nitric and sulfuric or phosphoric acid, began in Europe.

Several process improvements have been made over time. The popularity of nitric phosphates derived from the fact that they were more cost effective in many regions when compared to other process methods that used imported Sulphur. Although production in the United States has been limited, there are multiple nitric phosphate factories in Europe, Asia, and South and Central America. Since granular ammonium phosphates, ammonium polyphosphate (APP) fertilizers are the only new forms of economic relevance.

TVA began developing these liquid and solid materials in 1955. They are the backbone of the rapidly expanding liquid fertilizer business in the United States (about 2,800 plants in 1975). Several technologies for producing potassium phosphate fertilizers of various sorts were developed starting about 1970. In 1975, one modest facility opened in the United States. (Darwin 1895)

2.2 Phosphate Fertilizer and Process Technology:

The TVA method for granular ammonium phosphates uses a pre-neutralizer and a rotating drum ammoniator-granulator as critical equipment. Nitric phosphate manufacturing is expanding at a considerably faster rate across the world.

In the manufacturing of granular NPK grades, wet-process acid, OSP, TSP, and non-granular MAP are used as intermediates. Bulk blending, which is popular in the United States, uses granular DAP, MAP, and TSP.

The backbone of the fluid fertilizer industry is fluid and granular polyphosphates, which TVA developed and pioneered. Starting in the early 1970s, TVA pipe reactors allowed for the manufacture of liquid and solid polyphosphates from wet-process acid. Potassium phosphates appear to be the only truly novel fertilizers that might be commercially viable between 1977 and 1980.

Several countries are working on developing processes. In some places of the world, direct application of pulverized phosphate rock and partially processed phosphates may be investigated further. Melt-type granulation, which has the advantages of less energy, less investment, and less pollution, is gaining popularity. Several TVA pipe reactor plants are being built or renovated (from 1975 to 1976). (Darwin 1895)

2.3 why nitric acid replaces sulfuric acid in phosphate fertilizer production?

In 1950, there was a global Sulphur crisis, limiting supply of sulphuric acid for superphosphate production. The Agricultural Research Council launched a field trial to see whether there were any phosphate fertilizers that didn't require sulphuric acid or used it sparingly. Several types of nitro phosphate were compared to superphosphate plus equivalent nitrogen in half of the field studies. Cooke described the findings of the research (1955).

When sulphuric acid is substituted with nitric acid in the usual superphosphate process, the result is mono-calcium phosphate and calcium nitrate, both of which are too hygroscopic to store and use on the farm. The majority of phosphate rock treatment procedures with nitric acid to produce nitro phosphate have been adjusted to eliminate calcium nitrate from the end product. Cooke (1954) identified a number of economically viable techniques. Phosphate rock was treated with nitric acid, then ammonia was added, and ammonium nitrate, di-calcium phosphate, and calcium nitrate were created, according to d'Leny (1953).

Calcium sulphate was precipitated by adding ammonium sulphate, and the resultant product contained no calcium nitrate. (Cooke and Widdowson 1956)

2.4 Raw Material:

Nitro phosphate fertilizer by Odda process required following raw materials.

- Nitric acid (Formula HNO_3).
- Phosphate rock.
- Urea Solution.

- Ammonia (NH₃).
- Antifoams or defoamer.

2.41 Nitric Acid:

Nitric acid with formula of (HNO₃) is a very corrosive mineral acid also known as aqua Fortis and spirit of niter. The pure chemical is colorless, but breakdown into nitrogen oxides and water causes older samples to turn yellow.

2.41a Manufacturing process:

Nitric acid is made by reaction of nitrogen dioxide (NO₂) with water.



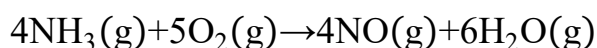
The net reaction is:



The nitric oxide produced by the reaction is normally deoxidized by oxygen in the air to produce more nitrogen dioxide. Increased acid output can be achieved by releasing nitrogen dioxide via hydrogen peroxide.



The concentration of nitric acid in commercial grade nitric acid solutions is normally between 52 and 68 percent. The Ostwald procedure, named after German chemist Wilhelm Ostwald, is used to make nitric acid. At a high temperature of around 500 K and at 9 atmospheric pressure, anhydrous ammonia is oxidized to nitric oxide in the presence of a platinum or rhodium gauze catalyst.



$$\Delta H = -905.2 \frac{\text{Kj}}{\text{mol}} \quad \text{Eq - 2.4}$$

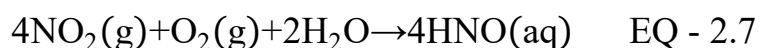
Nitrogen dioxide is foamed when nitric acid react with oxygen in air.



This is subsequently absorbed in water to form nitric acid and nitric oxide.



The nitric oxide is returned to the system for re-oxidation. Alternatively, if the final step is done in the air.



Distillation can concentrate the aqueous HNO₃ to a mass concentration of about 68 percent. Dehydration using concentrated H₂SO₄ can increase the concentration to 98 percent. The end product can be made from nitrogen, hydrogen, and oxygen derived from air and natural gas as the sole feedstocks by employing ammonia derived from the Haber process. (Britannica Encyclopedia 2016; Johnson 1927)

2.41 Other Uses of Nitric Acid:

Nitric acid is further use in following processes.

- Nitric acid is used for the production of ammonium nitrate which is a major component of fertilizers.
- Glass can be cleaned with nitric acid. It accomplishes this by removing ions from the glass' surface.
- It's also used to make nitroglycerin and trinitrotoluene, which are both explosives (TNT).
- It is also used for oxidizing metals.(Britannica Encyclopedia 2016)

2.42 Phosphate Rock:

Phosphate of Rock is a mineral that is found in rocks. Rock phosphate, also known as phosphorite, is a sedimentary rock that contains a lot of phosphorus.

$\text{Ca}_{10}(\text{PO}_4)_6(\text{X})_2$, where X is F-, OH-, or Cl-, is the usual formula for pure rock phosphate. Apatites are the name for these minerals. Fluor apatite, which contains impurities such as CO_3 , Na, and Mg, is the most common rock phosphate mined. Clay and limestone are also found in the rock, which is mined. The majority of phosphate rock resources are sedimentary, meaning they come from the remains of marine life in areas that were previously covered by seas and have been converted over millennia into significant phosphate deposits. The origins of other resources are igneous or volcanic.

The phosphate concentration of phosphorite (or phosphate rock) varies dramatically, ranging from 4% to 20% phosphorus pent oxide (P_2O_5). Phosphate rock for sale is enhanced to at least 28%, and sometimes more than 30% P_2O_5 .

Phosphate rock was discovered in central Florida in the 1880s, and because of the extensive, high-quality deposits and lower mining cost, it quickly became the leading phosphate-producing state. Currently, phosphate rock is mined in Florida, Idaho, North Carolina and Utah.(Chan et al. 2012)

This vital resource may be found in many locations across the world. Early deposits were found in England, Ireland, France, Spain, Germany, and the United States of America (South Carolina). Because of their poor quality or because they have been exhausted, most of these deposits are no longer mined.

Morocco, the United States, the Soviet Union, the Spanish Sahara, and Tunisia are the main international suppliers at the moment (1976). On a smaller scale, deposits are being mined in Togo, South Africa, Australia, Israel, Brazil, Jordan, Senegal, the Ocean Islands, Algeria, and Egypt. Peru, Saudi Arabia, Venezuela, Colombia, India, Pakistan, Turkey, Iraq, Iran, and Mexico have all discovered or mined deposits that might be significant.

According to current surveys and expeditions in various places, the globe should have enough supplies of this vital resource.

However, it is anticipated that lower-grade phosphate with substantial quantities of undesirable impurities will be required. TVA began research in 1975 with the goal of creating techniques for the exploitation of low- or marginal-quality phosphate rock.(Darwin 1895)

2.42a Analysis of Phosphate Rock from Several Sources.

Following analysis are by weight of phosphate rock.

Table 1. several sources of rock phosphate

sources	P2O5	CaO	MgO	R2O3	F	SiO2	CO2	Na2O
Central Florida USA	32.1	46.5	0.5	2.8	4.0	4.7	4.0	0.6
North Caroline USA	32.8	52.8	0.6	1.2	4.0	2.5	–	1.0
Taiba	37.4	51.2	0.1	2.1	4.0	2.9	1.7	0.2
Togo	36.6	52.3	0.1	1.8	4.0	1.8	1.8	0.3
Morocco	32.1	51.6	0.4	0.6	4.1	1.4	5.3	0.8
Algeria	29	49.3	0.8	0.7	3.6	1.0	7.4	2.0
Kola	38.2	52	0.06	1.8	3.1	2.0	0.2	0.5
Oron Israel	31.3	52.7	0.24	0.5	3.6	0.2	7.5	0.8
Gafsa	28.8	48.3	0.6	1.2	3.4	1.8	6.3	1.3
India Jhamar Kotra	40.1	54.2	0.04	0.7	3.6	1.2	0.7	0.1
Spanish Sahara	35.8	51.9	0.14	1.2	3.8	3.4	2.3	0.4
Jordan	33.8	53	0.18	3.4	4.0	5.6	4.9	0.5

Some phosphate ores, such as those from the Spanish Sahara, have a chloride concentration of roughly 0.3 percent. High Cl levels can cause substantial corrosion in the production process and increase the concentration of wet process acid.

The addition of a little quantity of ammonia to the acid before concentration has reduced corrosion.(Darwin 1895)

(Orris et al. 2015)

2.42b Phosphate Rock Resources in Pakistan:

Phosphate rock deposits in Pakistan are found in the Hazara division and are sedimentary in origin. They can be found in the form of mica schist, shale, or limestone in Tarnawai village; dolomites or phosphorite at Kakul; and orthquartzites and lime stones near Thandiani.

A huge number of phosphate deposits may be found in the Delolaha area of Garhi Habibullah. These reserves are estimated to be 9.1 million tons, according to prior estimates. However, it comprises low-grade materials that do not exceed the industry's standards of fertilizer manufacturing industries. These deposits are geologically and miner logically distinct from the Kakul deposits.

The beds of the Kakul deposits are continuous in both dip and length. Strike in the hundredth and tenth feet, respectively.

Calcium oxide (40-50%), hematite (1-2%), and quartz (10-25%) make up a considerable portion of gangue in mineralogical deposits. The deposits area of Ghari Habib Ullah (Delolaha) is faulted and heavily fractured. Formations of the overall Dolomitization occur in the sediments. The deposit's stratigraphy is not consistent due to a fault in the area's bed. The principal gangues in the Garhi Habibullah phosphate deposits are quartz (35-50%) and calcium oxide (15-20%) and hematite (5-10 percent). (Orris et al. 2015)

2.43 Urea Solution:

A transparent aqueous solution of urea and water is known as urea solution. This substance is also available in concentrations ranging from 30 to 60%, depending on the final application. In a variety of cropping systems, urea solutions are utilized as a source of nitrogen (N) fertilizer.

The addition of urea solution to dissolving reactors began in 2005 to decrease NO_x emissions in the stack. Before the addition of urea, NO_x emissions in the stack were 200–300 ppm. Urea Solutions are also utilized to manage emissions in a variety of industrial settings and in other industrial chemical applications. During digestion of rock phosphate in dissolving reactor, urea solution of about 0.2% of mass of rock phosphate is added to control emissions of NO_x. Urea solution of 50% has nitrogen of 23% is high pure solution which is used in variety of industrial applications. PH of such solution is 6.5 to 7.5 and measured weight per gallon is 9.5lbm/gallon. (Hussain 2012)

The finished product contains tiny levels of urea as a result of hydrolysis, and no adverse impacts can be observed in the product attributes. By adding urea to digestion, a simple scrubber may lower NO-X levels in the environment to below 150 ppm. An automated control system will guarantee that the solution has the proper amount of urea. (Nielsson n.d.)

2.44 Ammonia (NH₃):

Ammonia is a nitrogen-hydrogen chemical with the formula NH₃. Ammonia is a colorless gas with a pungent odour. It is a stable binary hydride and the simplest pnictogen hydride. Ammonia neutralize nitric acid (HNO₃), phosphoric acid (H₃PO₄) and calcium nitrate into CaHPO₄ and ammonium nitrate.

Few companies are providing ammonia gas in Pakistan. Some are Premier tubular quality inspection services, Lubricants oils, and non-destructive testing services by NDT Pakistan etc. (Anon n.d.)

2.45 Antifoams or Defoamers:

A defoamer, also known as an anti-foaming agent, is a chemical additive that prevents foam formation in industrial process liquids. The phrases defoamer and anti-foam agent are frequently used interchangeably. To avoid foam production, a range of chemical formulations are available.

Certain alcohols, insoluble oils (castor oil), stearates, polydimethylsiloxanes and other silicone derivatives, ether and glycols are all often used antifoaming agents.

Foam can be a concern in downstream NP solution granulation processes, just as it can be in the nitro phosphate manufacturing process. The addition of a suitable defoaming agent can increase capacity utilization and optimize fertilizer output.

To comply with emission requirements, exhaust gases from the reaction of phosphoric acid with ammonia must be scrubbed from the air stream during fertilizer manufacture. To remove entrained gases and particles, a scrubber liquid is collided with the airstream at the right velocity. The high-speed impact traps air inside the liquid, resulting in a very frothy solution that reduces pumping efficiency and scrubbing capacity. If left unchecked, it will become a limiting factor in ammonium phosphate fertilizer output, as well as a source of environmental regulatory non-compliance.

In the more dilute liquid phase found in the granulation scrubbing system, an ideal granulation defoamer should have a superior knock-down impact on the slightly different forms of surface foam bubbles. Understanding the characteristics of the phosphate rock, the specific type of granulation manufacturing processes, and the expected operating parameters is required for the design and selection of an appropriate defoamer.

The use of effective defoamers necessitates a wealth of knowledge and access to the operational and feedstock variables that determine the nature of the process issue.(Nielsson n.d.)

2.6 Process Description of Odda Process:

The term "nitro phosphate process" is usually reserved for procedures that use nitric acid alone for the acidulation of phosphate rock.

The elimination of calcium by crystallization as calcium nitrate tetra hydrate is a distinguishing aspect of this procedure.

The first technique in this category was developed into a commercially effective technology circa 1930, based on Erling B. Johnson's ideas in Odda, Norway. The Odda technique, as it is known, was quickly adopted across continental Europe and Norway. Since then, several variations of this method have been created.

The process consists of following main stages.

- Rock Phosphate Storage and Handling.
- Phosphate rock digestion
- Calcium nitrate tetra hydrate crystallization
- Filtration to remove the calcium nitrate tetra hydrates crystals.
- The mother liquid is neutralized (with the addition of ammonium nitrate if necessary).
- Evaporation of the neutralized liquid's water.
- The evaporated liquid is combined with recycled off-spec product and, if necessary, potassium salts and micronutrients.
- In an air-cooled pilling tower, the mixture is pilled.
- The final product is screened, cooled, and coated.

- Ammonia and carbon dioxide are used to convert calcium nitrate tetra hydrate to ammonium nitrate and calcium carbonate. (Alternatively, the calcium nitrate can be converted into a calcium nitrate-ammonium nitrate fertilizer, which is primarily composed of $5Ca(NO_3)_2 \cdot NH_4NO_3 \cdot 10H_2O$).
- Liquid effluent management.
- Dust and gaseous effluents are reduced.(Hussain 2012)

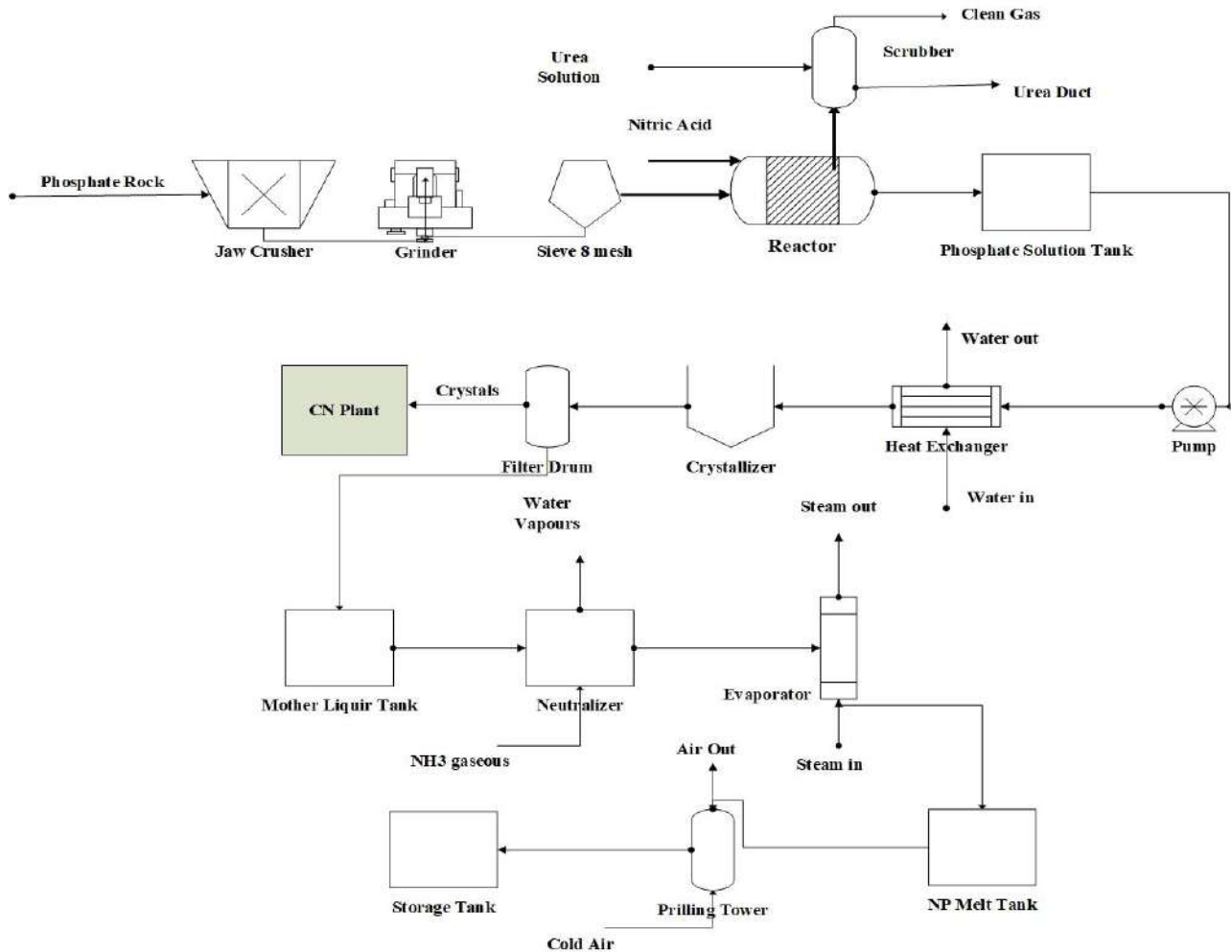
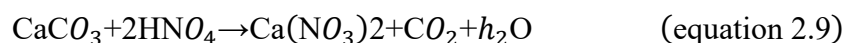
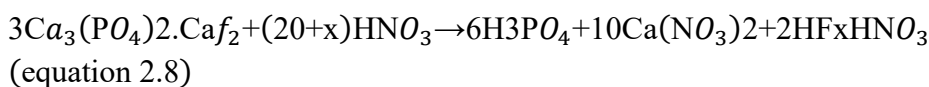
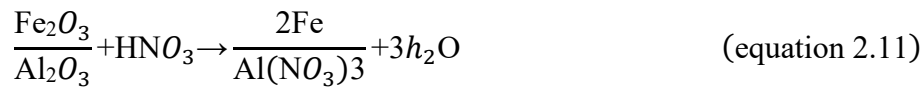


Figure1. Process flow diagram of Nitro phosphate by Odda process

2.61 Digestion of Rock Phosphate in Dissolving reactor:

In dissolving reactor, phosphate rock is digested with the help of nitric acid of 60%. Calcium nitrate and phosphoric acid are produced when phosphate rock is treated with 60 percent nitric acid. Other minerals are dissolved in addition to the calcium phosphate. Following main reactions take place there.





The reactions are exothermic and take place in two stainless steel vessels with agitators at temperatures ranging from 50 to 65°C.

A portion of the nitric acid is utilized to wash the crystals on the calcium nitrate filters before being reintroduced to the digesting tanks. The pace of phosphate rock dissolving is determined by the amount of nitric acid present, the degree of agitation, and the rock's exterior surface. Even for the most unreactive rocks, a residence time of less than one hour is adequate to dissolve more than 99 percent of the phosphorous with particle sizes less than 8 meshes.

As a result, additional grinding of commercial rocks is rarely necessary. The phosphate rock dissolves, releasing minor amounts of HF and SiF₄ as well as nitrogen oxides (NO₂ + NO). The formation of too many oxides due to the presence of reducing agents in the rock can be effectively prevented by adding urea solution.

The amount of urea needed is usually between 0.1 and 0.3 percent of the rock feed. The use of vigorous agitation reduces foaming caused by the CO₂ content and organic material in the rock. Antifoaming agents may be required as well.

Separation of undissolved particles such as silica, for example, can be done after acidulation. Otherwise, equipment erosion may occur, as well as a reduction in the concentration of nutrients in the product. Silica can be removed via a hydro cyclone or settling cone system, with the particles then being washed in a spinning drum. Because some phosphate rocks contain relatively little acid-insoluble material, no cleanup is necessary. (Nielsson n.d.)

2.62 Crystallization of calcium nitrate:

Batch crystallizers are run in a series, resulting in a continuous operation that produces big, uniformly sized crystals (about 1 mm).

The crystallizers have cooling coils that use a 20% aqueous ammonia solution as a coolant. The coolant enters the vessel in the most advanced state of cooling from the refrigeration section, and then runs sequentially through each crystallizer to the warmest unit. The suspension is discharged to a buffer tank and fed to the calcium nitrate filters at a continuous rate once the coolest crystallizer has achieved the end temperature. Warm digestion liquid is put into this crystallizer, which melts the clinging crystals and regenerates the coil surface. The crystallizers' operation can be totally automated. The degree of calcium removal impacts the water solubility of

P₂O₅ in the final product, which is determined by the end temperature. As more calcium is eliminated, nutritional concentrations and water solubility rise. In the past, cooling to around 15 to 20°C was normal procedure, resulting in a product with 25 to 30% P₂O₅ in a water soluble state. It is possible to cool to -5 to -10°C and achieve a water solubility of 80 to 85 percent. (Nielsson n.d.)

2.63 Filtration:

Centrifuges are used in the Odda process to separate the calcium nitrate crystals from the mother liquid. Norsk Hydro's nitro phosphate process employs a filter that the company designed and built. Each filter features two rotary drums that are both vacuum-operated.

The first drum's mother liquor is transferred to a mother liquor tank. The mother liquor-containing calcium nitrate cake is put to suspend in wash acid, filtered on the second filter drum, and then washed with nitric acid and water. The wash acid that results is collected in a wash acid tank. A portion of the acid is recycled to suspend the calcium nitrate filter cake in the calcium nitrate filters, while the rest is returned to the acidulation stage. The calcium nitrate crystals are flushed from the filters with molten calcium nitrate and sent to a surge tank, where they are melted and sent to the conversion section. The crystallizer coolant can be precooled in the cold mother liquor before being pumped to the neutralization section.(Nielsson n.d.)

2.64 Neutralization of mother liquor:

In two or three processes, the filtrate, or mother liquid, is neutralized with gaseous ammonia. The ammonia comes from a crystallizer refrigeration system evaporator.

Calcium is precipitated as calcium fluoride and di-calcium phosphate during the neutralization stage. Except for small amounts of aluminum, iron, and magnesium phosphates, the rest of the phosphorus stays in solution as ammonium phosphates. In addition, ammonium nitrate is generated. The main reactions are following:



Each step's pH is automatically adjusted, resulting in a final value of around 5.6 (measured in a 1:9 (w/w) dilution with water). The ammonium phosphates have a high solubility as a result of this. The final step involves adding 10 to 30% of the total ammonia. The heat of neutralization evaporates a significant portion of the water content of the mother liquor.

The neutralization of the mother liquid appears to be aided by increased calcium nitrate elimination. As a result of the enhanced elimination, it is now possible to formulate goods with lower N/P₂O₅ ratios than before.

The neutralized liquor has a minimum N/P₂O₅ ratio of roughly 0.6/1. By adding ammonium nitrate during neutralization, it can be increased to any desired amount. A product with an N/P₂O₅ ratio of around 2/1 is obtained by returning the complete amount of ammonium nitrate from the conversion plant. Additional ammonium nitrate from another source must be added for larger ratios. The ammonium nitrate solution is generally concentrated to above 90% before being added.(Nielsson n.d.)

2.65 Evaporation of neutralized liquid:

Evaporation of water from a liquid phase consumes far less energy than drying a solid product to remove the same amount of water. Low pressure evaporation reduces the water content of the neutralized liquid to 0.5 percent at around 180°C. It is so possible to avoid the installation of huge drying equipment. The evaporation section has a high degree of uniformity. The evaporation section can function automatically with relatively little monitoring thanks to the instrumentation and process control. No potassium chloride is added to the liquor before evaporation to reduce the likelihood of thermal breakdown of the liquor in the evaporators.(Nielsson n.d.)

2.66 Mixing with potassium salts and other additives:

Regular commercial grade KCl or K₂SO₄ can be used as a potassium source. When these salts are mixed with hot melt, chemical reactions cause the viscosity of the combination to rise to the point where pilling is severely hampered.(Johnson 1927)

2.67 Prilling:

A revolving perforated pilled bucket forms the NP melt or NPK mixture into "drops" at the top of an air-cooled pilled tower. The bucket is meant to prevent clogging and ensure that the prills are distributed evenly. The bucket must be cleaned on a regular basis. A second, clean bucket is then used to replace it. Buckets are quickly changed using unique equipment. The prills are scraped off the tower with a spinning bottom and a stationary scraper, or with a rotating scraper if a stationary bottom is utilized.

The pilling process has several advantages, including high capacity in a single train, low recycle rates and equipment hold-up, no requirement for product drying, and minimal investment and maintenance expenses.

Normally, the quantity of off size is less than 10%. The pilling technique is particularly beneficial for high-capacity facilities, but it can also be employed cost-effectively for small units. Although normal pug mill/drum granulation can be utilized in this procedure, pilling is the preferable method. Because of the low hold up, it is very straightforward to manage the product composition. In addition, the spray pattern in the tower can be monitored and controlled easily and fast.

The huge volume of fertilizer material in the subsequent dryer may start to deteriorate when nitro phosphates containing potassium chloride are granulated in standard pug mills or rotating drum granulators. Because the retention time is short and the temperature is easily regulated, this is unlikely to happen during the pilling process.(Nielsson n.d.)

2.68 Screening, Cooling and Coating:

Before coating, the prills are screened and chilled. Fluid-bed or rotating-drum coolers can be used for cooling. Oversized particles are crushed and rescreened before being sent to the pilling tower's top with the undersized fraction. The material, together with the potassium salt and any micronutrients, is incorporated into the evaporated melt.(Hussain 2012)

2.7 Conversion of calcium nitrate tetra hydrate:

Calcium nitrate tetra hydrate can be used in the manufacturing of lime fertilizer nitrate or transformed into ammonium nitrate and calcium carbonate. Lime fertilizer nitrate is a great product that contains 15.5 percent nitrogen and 19 percent water soluble calcium. It is hygroscopic, but in addition to $5\text{Ca}_3(\text{NO})_2 \cdot \text{NH}_4\text{NO}_3 \cdot 10\text{H}_2\text{O}$

It contains $\text{Ca}(\text{NO}_3)_2 \cdot 0\text{H}_2\text{O}$ and $\text{Ca}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ and which can "bind" the absorbed water and keep the product dry. It has excellent storage qualities when coated with the appropriate coating agents.

A conversion plant produces ammonium nitrate, which is the chosen product. The conversion of calcium nitrate to ammonium nitrate and calcium carbonate can be done in a variety of ways.

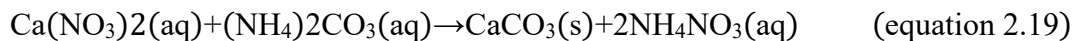
To prevent scaling difficulties, the absorption of ammonia and carbon dioxide, on the one hand, and the precipitation of calcium carbonate, on the other hand, take place in distinct units.

In a recirculation OF ammonium nitrate solution, ammonia and carbon dioxide react to produce ammonium carbonate.



The absorption is done in a packed tower. The crystallizer refrigeration system feeds ammonia directly to the column from an evaporator, while CO_2 is compressed before being delivered to the column. An external cooling circuit removes the heat of reaction, which is 36 Kcal/mole CO_2 . Absorption occurs at a pressure of roughly 2.0 bars. The pressure is regulated to match the temperature of the cooling water in order to keep the ammonium carbonate solution in the reactor at 40°C . The heat released is reduced by approximately 6 Kcal/mole CO_2 when liquid ammonia is employed.

The calcium nitrate melt and ammonium carbonate solution are put into a tank reactor, where they are converted to ammonium nitrate and calcium carbonate.



In comparison to the heat generated in the absorption unit, the heat of reaction is low (about 7.5 Kcal/mole calcium). This means that no additional cooling in the reactor is necessary by managing the temperatures of the ammonium carbonate solution and the calcium nitrate melt.

To ensure full conversion, the reactor temperature should be kept below 70°C . Before the suspension is sent to a filter, it grows more crystals in a following filter feed tank.

The conversion efficiency is greater than 99.9%. The typical size of calcium carbonate crystals is 80 microns.

The calcium carbonate suspension is filtered and rinsed with water using a revolving filter. In a polishing filter unit, the ammonium nitrate solution is further purified. A little amount of alcohol is present in the solution. To avoid loss, an excess of ammonia is neutralized using nitric acid.

The solution contains around 60 percent ammonium nitrate.

About two-thirds of the liquid is re-circulated to the absorption column, while the remainder is concentrated by evaporation and used for other purposes.

N/P 2O5 -ratio adjustment in the NPK-process any excess can be eliminated. Other ammonium nitrate-based fertilizers are produced as a result of the processing. By making use of Calcium carbonate and ammonium nitrate limestone can also be produced.(Nielsson n.d.)

2.8 Liquid Effluents:

It is conceivable to release less than 1.5 m³/t P₂O₅ as effluent, however the utilization of holding ponds allows for a discharge near nil.

The pollutant loads in effluents reported for NPK fertilizers generated by the nitro phosphate method are shown in the table below.(Description and Characteristics 2007)

Table 2. Pollutant loads in Effluents, Nitro phosphate process

Kilograms per ton of NPK fertilizer produced	
Parameter	Loading
P ₂ O ₅	0.06
NH ₄ -N	0.012
Nitrate nitrogen (NO ₃ -N)	0.03
Fluoride (as fluorine)	0.05

2.9 Control of Liquid Effluents:

Since 1977, a commercial plant for the treatment of liquid effluents has been in operation. All effluent streams from the NPK-plant that are caused by any leakage, overflow, floor cleaning, equipment cleaning, and so forth. To prevent a constant supply of new washing water, a closed water system is employed in this technique. The water in this system is the liquid effluent is treated to generate tem. In this case, mint, as well as essential nutrients like as phosphorus, nitrogen, and potassium are resurrected and reintroduced into the process this wash can be used to include fresh-water make-up. water as ordinary process water in the nitro phosphate industry process.

Water contaminated with Cl must be collected in a separate system for safety reasons. As a result, two circuits are established: one containing Cl and the other carrying P₂O₅. Water contaminated with Cl must be collected in a separate system for safety reasons. As a result, two circuits are established: one containing Cl and the other carrying P₂O₅.

A regulated flow from the Cl circuit to the P₂O₅ circuit containing circuit maintains a safe minimum Cl level in the water re-entered the process.(Nielsson n.d.)

2.10 Air emissions:

The first table displays the emissions values that have been published for the nitro phosphate pathway in the manufacturing of NPK fertilizers and that should be achieved in a well-run facility. The emissions loadings shown in the second table are achievable for NPK fertilizers generated by the mixed-acid approach.(Description and Characteristics 2007)

Table 3. Emissions loadings for Nitro phosphate process

Kilograms per tons of NPK fertilizer produced	
Parameter	Loading
Ammonia (NH ₃ as N)	0.3
Nitrogen Oxides (as NO ₂)	0.2
Fluoride (as fluorine)	0.02
PM	0.3

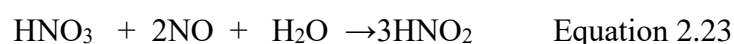
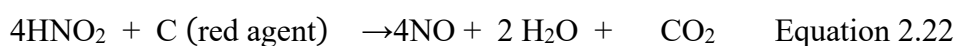
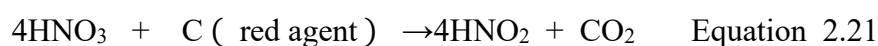
Table 4. Emissions loadings for Nitro phosphate process

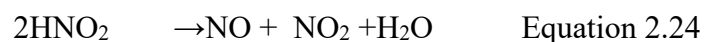
Kilograms per ton of NPK fertilizer produced	
Parameter	Loading
Ammonia nitrogen (NH ₄ -N, including free ammonia)	0.01
Fluoride (as fluorine)	0.01
PM	0.2

2.11 Reduction of gaseous Effluents and Dust:

Fluorine compounds, nitrogen oxides, and nitric acid vapor are the primary components in exhaust air from digestion, crystallization, and filtering. The amount of fluorine compounds released is a small percentage of the total a sulfuric acid procedure that accounts for only 0.5 to 1% of fluoride the rock provides contentment. The generation of nitrogen oxides occurs during the digestive process the decrease of nitric acidduction of Gaseous Effluents and Dust.

The probable chain reactions are





The amount of nitrogen oxides produced in the process is greatly influenced by the phosphate rock type. However, most rocks include considerable contaminants that inhibit nitric acid production. (Nielsson n.d.)

2.12 Scrubbing:

Ammonia is present in the vent air from the alkaline portion (neutralization and conversion). Scrubbing is done by re-covering the ammonia as an ammonium nitrate solution with nitric acid. For enhanced nitrogen recovery, another top portion with final water washing is frequently employed. An additional mist eliminator can be added in places with very stringent criteria to collect any ammonium nitrate fume. In addition to ammonia, vapour from NP-evaporation contains tiny quantities of fluorine compounds. Scrubbing with an ammonium nitrate solution removes the fluorine before recovering the ammonia with nitric acid. Condensing the vapour and stripping the ammonia with steam is an alternate process. Prilling of NPK fertilizer creates relatively little fume in effluent air, in contrast to prilling plain nitrogen fertilizers like urea and ammonium nitrate, which causes a lot of pollution. Dust and fume concentrations are often less than 15 mg/Nm, therefore air cleaning is not necessary. The reason for this is that before prilling, recycled material and potassium salt are added to the melt to keep the prilling temperature low. Other equipment's dust is collected and processed in cyclones and bag filters before being reintroduced to the mixer as dry material. (Nielsson n.d.)

2.13 Process control:

The general goal is to create a fertilizer with a precise composition of three primary elements: nitrogen, phosphorous, and potassium, as well as a uniform size and content for each prill.

For its NPK fertilizer product, the process is essentially a mixing operation involving five primary components: nitric acid, ammonia, ammonium nitrate, phosphate rock, and potassium salt. Any error in one of these several components will have an impact on the others. Additionally, nitrogen is added at different times throughout the process. The majority of the automated functions are achieved via the employment of well-designed electric-recorder controller systems and electric interlocks. Microprocessor-based digital-control devices have also been produced in recent years, typically integrating with existing instrumentation. (Johnson 1927)

Manual analyses are still an important aspect of the recording system, and they might take a long time. Usually, two to three hours are necessary. Furthermore, buffer capacities allow longer retention durations. This means that a change in raw material intake will take several hours to notice. Adjustments by the operators are frequently done on an ad hoc basis, and overreactions are common. This iterative strategy does not yield the most cost-effective solution, therefore any reduction in response time is welcome.

As a result, technologies that could gather analytical data automatically and quickly would be quite useful. In many process sectors, such automatic systems are already in use or in development.

In order to better control of raw material feed ratios, an automated sampler/analyzer system is being tested for nitrogen, calcium, and phosphorus in digestion liquid and mother liquid. The flow of NH₃ to the neutralizers is regulated by automated liquid sampling and pH monitoring. The conversion reactor's pH and CO₂ levels are automatically monitored. The temperature profile for prills on the prill tower bottom is provided by an infrared scanner. The profile is calculated throughout the radius of the tower and provides useful information about the prill bucket's spray pattern. An automated sampler/analyzer provides extremely quick and reliable information on the nitrogen, phosphorus, and potassium levels of the end product. The rapid response time (approximately 10 minutes) allows for a prompt response to valuable nutrient overdose. These are instances of analytical advancements that will be critical components of any computer-controlled process in the future.(Nielsson n.d.)

2.14 Mechanical Resistance:

To reduce the formation of crushed particles and dust, high mechanical resistance is necessary to endure pressures during storage, loading, transit, and discharge. The chemical makeup of NP and NPK prills affects their crushing strength. It will be shown that NP prills have the maximum strength, and that chloride-based type parti-cles are weaker than those with potassium sulphate as the potassium source. The higher the nitrogen level, the greater the propensity for lower crushing strength.

The data also demonstrates that by absorbing a significant quantity of extra water, the crushing strength of NPK is reduced only somewhat. (Nielsson n.d.)

Table 5. particle strength of Prilled NPK Fertilizers

Crushing strength, kg			
Fertilizer N- P ₂ O ₅	With normal moisture content	After about 1% moisture absorption	Remarks
16-16-16	6.3	4.9	KCl - based
20-11-11	5.6	4.4	
21-9-12	3.9	3.1	
25-7-11	3.2	3.1	
14-9-9	6.9	5.7	
15-15-15	7.0	6.3	
15-9-15	6.0	5.6	
21-16-17	5.6	4.0	

20-20	8.2	8.2	K ₂ SO ₄ - based
23-23	7.4	7.3	
26-14	6.0	6.0	

2.15 Caking Tendency:

Bulk material must have exceptional storage characteristics if it is to be transported over long distances and by enormous carriers with holds that are frequently 15 metres or more deep. As a result, surface active organic compounds have grown increasingly popular as anticaking agents in recent years. They can be used alone or in conjunction with a parting agent (fine powder).

The surface active chemicals alone have relatively little anticaking impact in piled and granulated NPK and piled AN. However, if these items are treated with cationic surfactants in addition to powder, they provide a satisfactory result.

When goods have a significant propensity to cake, this combination may be important to keep fertilizer particles free-flowing throughout extended sea shipment by bulk ships.

The table below illustrates typical caking index values for an NPK fertilizer product treated with various anticaking agents. A commercial product should have a caking index of 300 or less. Fertilizer is most susceptible to caking in the days following manufacture, owing to the fact that it is still warm and post reactions are occurring.

Table 6. Caking Index for NPK with 0.5 % H₂O

Anticaking agent	Caking index
Untreated product	1000
0.5% fatty amine	950
0.4% Kieselguhr	650
0.4% Kieselguhr + 0.5% fatty amine	300
0.4 % amorphous SiO ₂	360
0.4% amorphous SiO ₂ + 0.05% fatty amine	170

During shipment and subsequent storage in the consignee's warehouse, a bulk product that is free-flowing after 3 to 4 weeks in the producer's warehouse is very seldom caked.(Nielsson n.d.)

2.16 Hygroscopicity:

The majority of fertilizers are hygroscopic, meaning they collect moisture from the air easily. This feature is required to some extent if the particles are to disintegrate quickly following fertilizer application. The absorption of moisture, on the other hand, has a negative impact on the storage and handling properties of complex fertilizers. Most NPK fertilizers have lesser mechanical strength, storage qualities, a lower rate of moisture absorption, and a lower critical relative humidity than NP particles. As a result, even in the most adverse climatic circumstances, NP-products should be quite acceptable for bulk handling and shipment.

Most NP fertilizers, on the other hand, are made up of hard particles with a compact surface and little porosity. When exposed to humid air, the majority of the moisture absorbed condenses on the particles' surfaces. Wet and sticky particles with poor flow qualities might come from a low degree of absorption, which is generally not visible for particles with better absorption ability. With rising NH₄NO₃ levels, warmth, and humidity, this trend will increase. This is owing to NH₄NO₃'s high solubility. A hydrofobe coating has been created that renders the surface of particles water resistant, reducing the rate of moisture absorption significantly. This coating on NP fertilizers allows them to be carried in bulk without problem, even to extremely hot and humid climates.(Johnson 1927; Nielsson n.d.)

Table 7. Adherence properties of different Anticaking Agents

Powder	Dust quantity (mg per kg NPK)	
	0.4%	0.8%
Amorphous SiO ₂	64	353
Kaolin	1790	5000
Kieselguhr	2730	5080
Talc	1700	6550
Danish powdering chalk	1627	2135
Attacote	2680	4120

2.17 Dusting Tendency:

Dust can form at any point during bulk handling. Fine dust will be carried by air currents and pollute the atmosphere, especially while screening, moving from one conveyor to another, and during phases when fertilizer particles fall freely. Many port authorities now only allow a very low amount of dust; therefore, a lot of effort has gone into lowering dust levels. Industrial processes had to be upgraded, and transportation equipment had to be replaced or enhanced. Dust-removal equipment has been created and integrated into loading processes. Finding anti-caking compounds with good adhesion qualities is critical. (Nielsson n.d.)

Chapter No. 3 Material Balance

3.1 Production capacity of plant = 135000 tons/year

Total number of working days in year = 300

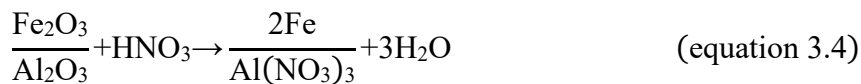
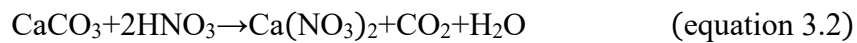
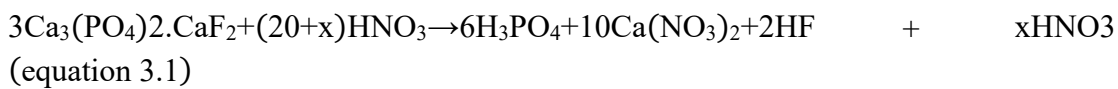
Operating hours in a day = 24

$$\text{Capacity of plant} = \frac{135000 \times 1000}{300 \times 24} \text{ kg/hr} = 18750 \text{ kg/hr}$$

3.2 Material balance around Acidulation/digester



Following reactions take place in reactor.



Composition of rock:

Compounds	Percentages
$\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2$	35
CaCO_3	25
MgCO_3	25
Fe_2O_3	15
Total	100

Mass of rock taken = 12267.28 kg/hr

Table 8. Composition of rock phosphate

Compounds	Mass (kg/hr)	Molar mass (kg/kmol)	Moles (kmo/hr)
$3\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2$	4293.548	1008.605	4.256
CaCO_3	3066.82	100	30.66
MgCO_3	3066.82	84	36.509
Fe_2O_3	1840.092	160	11.50
Total	12267.28		82.935

Moles of nitric acid required for reaction 1:

1 moles of calcium phosphate require = 20 moles of nitric acid
 4.2193 moles of calcium phosphate require = a moles of nitric acid
 $a = 20 \times 4.256 = 85.138 \text{ kmol/hr}$

Moles of nitric acid required for reaction 2:

1 moles of calcium carbonate require = 2 moles of nitric acid
 30.3978 moles of calcium carbonate require = b moles of nitric acid
 $b = \frac{2}{1} \times 30.668 = 61.33 \text{ kmol/hr}$

Moles of nitric acid required for reaction 3:

1 moles of magnesium carbonate require = 2 moles of nitric acid
 36.1879 moles of magnesium carbonate require = c moles of nitric acid
 $c = \frac{2}{1} \times 36.509 = 73.019 \text{ kmol/hr}$

Moles of nitric acid required for reaction 4:

1 moles of iron oxide require = 6 moles of nitric acid
 11.399 moles of iron oxide require = d moles of nitric acid
 $a = \frac{6}{1} \times 11.50 = 69.00 \text{ kmol}$

Total moles of nitric acid required = a + b + c + d = 288.497 kmol/hr

Total of 10% excess nitric acid is required for reaction 1

So total nitric acid = $0.1 \times a + b + c + d = 297 \text{ kmol/hr}$

Urea is added into the fertilizer which is only 0.2 % of the initial rock mass

Urea added = $0.002 \times 12267.28 \text{ kg/hr} = 24.534 \text{ kg/hr}$ (unreacted)

For the calculations of the species involved in the reaction, stoichiometric ratios are used to calculate the moles of each specie formed

H₃PO₄

Using reaction 1:

1 moles of calcium phosphate produce 6 moles of phosphoric acid

4.2569 kmol of calcium phosphate will produce e kmol of phosphoric acid

$$e = \frac{6}{1} \times 4.2569 = 25.54 \text{ kmol/hr}$$

Mass of phosphoric acid = moles \times molar mass = 25.3163 \times 98 = 2503 kmol/hr

All of the species are found using the same technique and the results are summarized below in the table

Compounds	Inlet			Outlet			
	molar mass (kg/kmol)	Moles (kmol/hr)	Mass (kg/hr)	Scrubber (Kmol/hr)	Scrubber (Kgl/hr)	Moles (kmol/hr)	Mass (kg/hr)
3Ca ₃ (PO ₄) ₂ .CaF ₂	1008.605	4.25	4293.54	0	0	0	0
CaCO ₃	100	30.66	3066.82	0	0	0	0
MgCO ₃	84	36.50	3066.82	0	0	0	0
Fe ₂ O ₃	160	11.50	1840.09	0	0	0	0
H ₃ PO ₄	98	0	0	0	0	25.54	2503.067
Ca(NO ₃) ₂	164	0	0	0	0	73.23	12010.92
HF	20	0	0	8.343558	166.9546	0.1702767	3.407236549
CO ₂	44	0	0	67.17796	2956.502	0	0
Mg(NO ₃) ₂	148	0	0	0	0	36.50	5403.44
Fe(NO ₃) ₃	242	0	0	0	0	23	5566.27
HN ₃	63	297.01	18711.72	0	0	8.513	536.371
H ₂ O	18	0	0	0	0	101.679	1830.23
total		379.947	30979	75.52152	3123.457	268.67	27853.73

1. Table 9. Material balance around dissolving reactor

.3 Material balance around Crystallizer

In crystallizer, the calcium nitrate is crystallized using the cool ammonia gas passing through the coils inside crystallizer

In crystallizer, molar ratio of calcium phosphate/phosphoric acid must be between 0.6 to 0.7. The ratio assumed in this project is 0.67.

Moles of calcium nitrate present in solution = 73.237 kmol/hr'

Moles of phosphoric acid = 25.54 kmol/hr

Moles of calcium nitrate present in solution after crystallization = $0.67 \times 25.54 = 17.11$ moles

Moles of calcium nitrate crystallized = $73.237 - 17.11 \text{ kmol/hr} = 56.124 \text{ kmol/hr}$

Here, the composition of the solution does not change, just the physical form of the calcium nitrate changes, so there will be no change in the inlet and outlet composition



Table 10. Material balance around

crystallizer

Compounds	From buffer tank			To filter drum	
	molar mass (kg/kmol)	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)
$3\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2$	388	0	0	0	0
CaCO_3	100	0	0	0	0
MgCO_3	84	0	0	0	0
Fe_2O_3	160	0	0	0	0
H_3PO_4	98	25.54	2503.067	25.54	2503.067
$\text{Ca}(\text{NO}_3)_2$	164	73.23	12010.92	73.23	12010.92
HF	20	0.170276689	3.405534	0.170276689	3.405534
CO_2	44	0	0	0	0
$\text{Mg}(\text{NO}_3)_2$	148	36.50	5403.44	36.50	5403.44
$\text{Fe}(\text{NO}_3)_3$	242	23	5566.27	23	5566.27
HNO_3	63	8.513	536.371	8.513	536.371
H_2O	18	101.679	1830.23	101.679	1830.23
total		268.65	27853.73	268.65	27853.73

3.4 Material balance around Filter Drum

Here, the crystallized calcium nitrate is removed from the solution sent from the crystallizer. Water is also added to wash the filtrate such that the water: mixture from crystallizer is 9:1 (by weight)

Total water added = $27853.7 \times 9 = 250683.57 \text{ kg/hr}$

Moles of water = $250683.57/18 = 13926.86 \text{ kmol/hr}$

The crystals are separated in the filter and then sent to CAN purification section

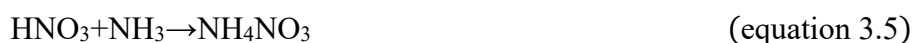
The summarized table of material balance around the filter drum is shown below

Table 11. Material balance around Filter drum

Compounds	stream to filter drum		to mother liquor		after filtration	
	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)
3Ca ₃ (PO ₄) ₂ .CaF ₂	0	0	0	0	0	0
CaCO ₃	0	0	0	0	0	0
MgCO ₃	0	0	0	0	0	0
Fe ₂ O ₃	0	0	0	0	0	0
H ₃ PO ₄	25.54	2503.067	25.54	2503.067	0	0
Ca(NO ₃) ₂	73.23	12010.92	73.23	12010.92	56.124	9204.428
HF	0.170276689	3.405534	0.170276689	3.405534	0	0
CO ₂	0	0	0	0	0	0
Mg(NO ₃) ₂	36.50	5403.44	36.50	5403.44	0	0
Fe(NO ₃) ₃	23	5566.27	23	5566.27	0	0
HN ₃	8.513	536.371	8.513	536.371	0	0
H ₂ O	14043.87	252789.7	14043.87	252789.660	0	0
total	14195.5	278537.3	14139.39	269332.8	56.124	9204.428

3.5 Material balance around Neutralizer

The reactions that are taking place inside the neutralizer is as follows:



Liquid ammonia from the crystallizer is fed into the neutralizer in order to dissolve the calcium nitrate solution and to convert the other products into useful by-products.

Phosphoric acid is divided into 5 equal parts for the reactions 5-9

Stoichiometric ratios are used to calculate the moles of ammonia needed for the reaction on the basis of phosphoric acid.

Phosphoric acid present for each of the 5 reaction = $25.54/5 = 5.108$ kmol/hr

For reaction 5:

1 mol of phosphoric acid need = 1 mol of ammonia to react

5.06326 kmol/hr of phosphoric acid needs = f kmol/hr of ammonia to react

$$f = \frac{1}{1} \times 5.108 = 5.108 \text{ kmol/hr}$$

1 mol of phosphoric acid produce = 1 mol of ammonium nitrate

5.06326 kmol/hr of phosphoric acid produce = g kmol/hr of ammonium nitrate

$$g = \frac{1}{1} \times 5.108 = 5.108 \text{ kmol/hr}$$

All the species are calculated using the same stoichiometric ratios and then added

However, Carbon dioxide and 90% of the water is removed from the neutralizer due to high temperature inside the neutralizer



The material balance summary is shown below

Table 12. Material balance around Neutralizer

Compounds	stream from filter drum		to evaporator		WASTE	
	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)
Ca ₃ (PO ₄) ₂ .CaF ₂	0	0	0	0		0
CaCO ₃	0	0	0	0		0
MgCO ₃	0	0	0	0		0
Fe ₂ O ₃	0	0	0	0		0
H ₃ PO ₄	25.54	2503	0	0		0
Ca(NO ₃) ₂	17.11	2806.50	12	1968.7390		0
HF	0.170276689	3.405534	0.170276689	3.405534	0	0
CO ₂	0	0	0	0	0	0
Mg(NO ₃) ₂	36.509	5403.44	31.40	4647.41		0

Fe(NO₃)₃	23	5566.278	19.59	4742.139		0
HN₃	8.51	536.37	0	0		0
H₂O	14028.5	252513.8	1402.885	25251.38	12625.69	227262.4
NH₃			54.488	926.30		0
NH₄NO₃	0	0	39.163	3133.09		0
NH₄H₂PO₄	0	0	1.7027	195.81		0
(NH₄)₂HPO₄	0	0	3.405	449.53		0
CaHPO₄	0	0	10.21	1389.457		0
Fe₂(HPO₄)₃	0	0	1.7027	681.106		0
MgHPO₄	0	0	5.108	612.996		0
Total	14139.39	269332.9	1581.8	44001.3	12625.69	227262.4

3.6 Material balance around Evaporator

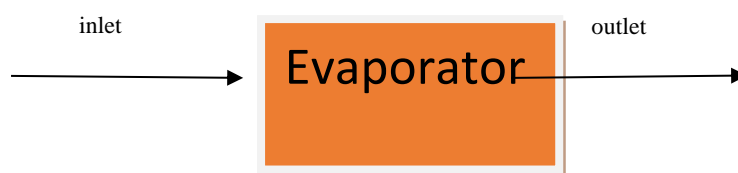
Total product contains 0.5% water, so the water evaporated

$$\text{Water in the feed} = 25251.38 \text{ kg/hr}$$

$$\text{Total feed without water} = 44001.3 - 25251.38 = 18750.1 \text{ kg/hr}$$

$$\text{Water in final product} = 0.005 \times 18750.11837 = 93.757 \text{ kg/hr}$$

$$\text{Water evaporated} = 25251.38 - 93.7507 = 25157.6 \text{ kg/hr}$$



Rest of the products are same and are represented below in the table.

Table 13. Material balance around Evaporator

Compounds	To evaporator			NP head tank		WASTE	
	Molar mass (kg/kmol)	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)	Moles (kmol/hr)	Mass (kg/hr)
Ca₃(PO₄)₂.CaF₂	388	0	0	0	0	0	0

CaCO₃	100	0	0	0	0	0	0
MgCO₃	84	0	0	0	0	0	0
Fe₂O₃	160	0	0	0	0	0	0
H₃PO₄	98	0	0	0	0	0	0
Ca(NO₃)₂	164	12	1968.7390	12	1968.7390	0	0
HF	20	0.170	3.40	0.170	3.40	0	0
CO₂	44	0	0	0	0	0	0
Mg(NO₃)₂	148	31.40	4647.41	31.40	4647.41	0	0
Fe(NO₃)₃	242	19.59	4742.139	19.59	4742.139	0	0
HN₃	63	0	0	0	0	0	0
H₂O	18	1402.85	25251.38	5.16	93	1399.1	25198.4
NH₃	17	54.488	926.30	54.488	926.30	0	0
NH₄NO₃	80	39.163	3133.09	39.163	3133.09	0	0
NH₄H₂PO₄	115	1.7027	195.81	1.7027	195.81	0	0
(NH₄)₂HPO₄	132	3.405	449.53	3.405	449.53	0	0
CaHPO₄	136	10.21	1389.457	10.21	1389.457	0	0
Fe₂(HPO₄)₃	400	1.7027	681.106	1.7027	681.106	0	0
MgHPO₄	120	5.108	612.996	5.108	612.996	0	0
Total	2529	1581.8	44001	184.11	18842.62	1399.1	25198.4

Chapter No. 4 Energy Balance

Table 14. Available data of different components

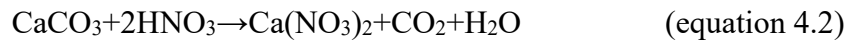
Components	Sp. Heat capacities (kj/kg K)
$\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2$	0.54
CaCO_3	0.8343
MgCO_3	1.383333333
Fe_2O_3	0.57
H_3PO_4	1.083673469
$\text{Ca}(\text{NO}_3)_2$	0.853658537
HF	2.8
CO_2	0.848863636
$\text{Mg}(\text{NO}_3)_2$	0.958783784
$\text{Fe}(\text{NO}_3)_3$	1.48
HNO_3	1.73
H_2O	4.2
NH_3	2.06
NH_4NO_3	1.74
$\text{NH}_4\text{H}_2\text{PO}_4$	0.426086957
$(\text{NH}_4)_2\text{HPO}_4$	1.423
CaHPO_4	0.653
$\text{Fe}_2(\text{HPO}_4)_3$	0.45125
MgHPO_4	1.774166667

4.1 Energy balance around Acid-dilution/Digestion

Digester is used to increase the temperature of the feed to 25 C to 70 C, so that the reactions can take place:

Following reactions are taking place in reactor.





General energy balance equation: -

$$\Delta H_{\text{in}} - \Delta H_{\text{out}} + \Delta H_{\text{generation}} - \Delta H_{\text{consumption}} = \Delta H_{\text{accumulation}}$$

Where,

$$\Delta H = m \int_{T_1}^{T_2} C_p dT$$

But if the variations in C_p at T_1 and T_2 are very small, we can take an average of C_p , hence

$$C_p = C_{p \text{ average}}$$

Therefore,

$$\Delta H = m C_{p \text{ average}} \Delta T$$

We will use the above mention equation for calculating ΔH .

Assumptions: -

- Since process is steady state the accumulation term becomes zero.
- System is reactive therefore $\Delta H_{\text{generation}}$ and $\Delta H_{\text{consumption}}$ are not zero.
- Utility used is steam to heat the process streams and is at 120.2°C and 2bar.
- reference temperature = 25 C (298K)

T_{in}	25.00	C
T_{out}	70.00	C
T_{ref}	25.00	C

Calculations: -

$$\begin{aligned} \Delta H_{\text{in}} = & (mC_p\Delta T)_{\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2} + (mC_p\Delta T)_{\text{CaCO}_3} + (mC_p\Delta T)_{\text{MgCO}_3} + \\ & (mC_p\Delta T)_{\text{Fe}_2\text{O}_3} + (mC_p\Delta T)_{\text{H}_3\text{PO}_4} + (mC_p\Delta T)_{\text{Ca}(\text{NO}_3)_2} + (mC_p\Delta T)_{\text{HF}} + (mC_p\Delta T)_{\text{CO}_2} + \\ & (mC_p\Delta T)_{\text{Mg}(\text{NO}_3)_2} + (mC_p\Delta T)_{\text{Fe}(\text{NO}_3)_3} + (mC_p\Delta T)_{\text{HNO}_3} + (mC_p\Delta T)_{\text{H}_2\text{O}} \end{aligned}$$

$$\Delta T = (25 - 25) = 0 \text{ C}$$

$$\Delta H_{\text{in}} = 0 \text{ kJ/hr}$$

$$\begin{aligned} \Delta H_{\text{out}} = & (mC_p\Delta T)_{\text{Ca}_3(\text{PO}_4)_2 \cdot \text{CaF}_2} + (mC_p\Delta T)_{\text{CaCO}_3} + (mC_p\Delta T)_{\text{MgCO}_3} + \\ & (mC_p\Delta T)_{\text{Fe}_2\text{O}_3} + (mC_p\Delta T)_{\text{H}_3\text{PO}_4} + (mC_p\Delta T)_{\text{Ca}(\text{NO}_3)_2} + (mC_p\Delta T)_{\text{HF}} + (mC_p\Delta T)_{\text{CO}_2} + \\ & (mC_p\Delta T)_{\text{Mg}(\text{NO}_3)_2} + (mC_p\Delta T)_{\text{Fe}(\text{NO}_3)_3} + (mC_p\Delta T)_{\text{HNO}_3} + (mC_p\Delta T)_{\text{H}_2\text{O}} \end{aligned}$$

$$\Delta T = (70 - 25) = 45 \text{ C}$$

$$\Delta H_{\text{out}} = 1556295.5 \text{ kJ/hr}$$

Where, ΔT_1 = inlet/outlet temperature – datum temperature

Similarly,

$$\Delta H_{\text{reaction}} = N_{\text{Reaction 1}} \times \Delta H_{\text{reaction 1}} + N_{\text{Reaction 2}} \times \Delta H_{\text{reaction 2}} + N_{\text{Reaction 3}} \times$$

$$\Delta H_{\text{reaction 3}} + N_{\text{Reaction 4}} \times \Delta H_{\text{reaction 4}}$$

Here, n1, n2, n3, n4 are the moles of species involved in the respective reactions

And

DELTA H of reaction 1=	-10724.2	kJ/mol
DELTA H of reaction 2=	-240	kJ/mol
DELTA H of reaction 3=	-274.6	kJ/mol
DELTA H of reaction 4=	-6956.6	kJ/mol

$$\Delta H_{\text{reactions}} = -143042.88 \text{ KJ/hr}$$



Table 15. Energy balance around Digester

	IN (kJ/hr)	Out (kJ/hr)
3Ca₃(PO₄)₂.CaF₂	0	0
CaCO₃	0	0
MgCO₃	0	0
Fe₂O₃	0	0
H₃PO₄	0	122062.83
Ca(NO₃)₂	0	461395.1
HF	0	21454.02
CO₂	0	112909.35
Mg(NO₃)₂	0	233132.88
Fe(NO₃)₃	0	370713.58
HN0₃	0	41756.405

H₂O	0	345914.23
Heat of reaction	-	-143042.9
Total	0	1556295.5

$$Q = \Delta H_{\text{out}} + \Delta H_{\text{reaction}} - \Delta H_{\text{in}}$$

$$Q = 1566295.504 \text{ kJ/kg}$$

4.2 Energy balance around Heat Exchanger:

The stream from the acid-dilution/digester enters into the exchanger at 70 C where it is cooled from 70 to 35 C through the cooling water.

General energy balance equation: -

$$\Delta H_{\text{in}} - \Delta H_{\text{out}} + \Delta H_{\text{generation}} - \Delta H_{\text{consumption}} = \Delta H_{\text{accumulation}}$$

Where,

$$\Delta H = m \int_{T_1}^{T_2} C_p dT$$

But if the variations in Cp at T₁ and T₂ are very small, we can take an average of Cp, hence

$$C_p = C_{p \text{ average}}$$

Therefore,

$$\Delta H = m C_{p \text{ average}} \Delta T$$

We will use the above mention equation for calculating ΔH.

Assumptions: -

- Since process is steady state the accumulation term becomes zero.
- System is non-reactive therefore ΔH_{generation} and ΔH_{consumption} becomes zero.
- Datum temperature = 298K

Conditions are

T _{in}	70	C
T _{out}	35	C
T _{reference}	25	C

Calculations: -

$$\begin{aligned} \Delta H_{\text{in}} = & (mC_p\Delta T)_{\text{phosphoric acid}} + (mC_p\Delta T)_{\text{calcium nitrate}} + (mC_p\Delta T)_{\text{hydrofluric acid}} + \\ & (mC_p\Delta T)_{\text{carbon dioxide}} + (mC_p\Delta T)_{\text{magnesium nitrate}} + (mC_p\Delta T)_{\text{nitric acid}} + \\ & (mC_p\Delta T)_{\text{Iron nitrate}} + (mC_p\Delta T)_{\text{water}} + \Delta H_{\text{reaction}} \end{aligned}$$

Where, ΔT = inlet temperature – datum temperature

$$\Delta H_{in} = 1566295.5 \text{ kJ/hr}$$

Similarly

$$\begin{aligned} \Delta H_{out} = & (mCp\Delta T)_{\text{phosphoric acid}} + (mCp\Delta T)_{\text{calcium nitrate}} + (mCp\Delta T)_{\text{hydrofluoric acid}} + \\ & (mcp\Delta T)_{\text{carbon dioxide}} + (mcp\Delta T)_{\text{magnesium nitrate}} + (mcp\Delta T)_{\text{nitric acid}} + \\ & (mcp\Delta T)_{\text{Iron nitrate}} + (mCp\Delta T)_{\text{water}} + \Delta H_{\text{reaction}} \end{aligned}$$

$$\Delta H_{out} = 379862.974 \text{ kJ/hr}$$



Table 16. Energy balance around heat exchanger

	IN (kJ/hr)	Out (kJ/hr)
Ca₃(PO₄)₂.CaF₂	0	0
CaCO₃	0	0
MgCO₃	0	0
Fe₂O₃	0	0
H₃PO₄	122062.829	27125.073
Ca(NO₃)₂	461395.098	102532.244
HF	21454.02	4767.56
CO₂	112909.34	25090.966
Mg(NO₃)₂	233132.879	51807.306
Fe(NO₃)₃	370713.58	82380.796
HN0₃	41756.40	9279.20
H₂O	345914.22	76869.828
Heat of reaction	-143042.88	10
Total	1566295.5	379862.974

$$Q = \Delta H_{out} - \Delta H_{in}$$

$$Q = -1186432.53 \text{ kJ/hr}$$

4/3 Energy balance around Crystallizer

Here the solution is cooled down using the ammonia gas from the refrigeration cycle. The solution is cooled from 30 to 10 C. due to cooling, the crystals are formed and then separated in the filtration unit

General energy balance equation: -

$$\Delta H_{in} - \Delta H_{out} + \Delta H_{generation} - \Delta H_{consumption} = \Delta H_{accumulation}$$

Where,

$$\Delta H = m \int_{T_1}^{T_2} C_p dT$$

But if the variations in C_p at T_1 and T_2 are very small, we can take an average of C_p , hence $C_p = C_{p_average}$

Therefore,

$$\Delta H = m C_{p_average} \Delta T$$

We will use the above mention equation for calculating ΔH .

Assumptions: -

- Since process is steady state the accumulation term becomes zero.
- System is non-reactive therefore $\Delta H_{generation}$ and $\Delta H_{consumption}$ becomes zero.
- Datum temperature = 298K

Conditions

T_{in}	30	C
T_{out}	10	C
T_{ref}	25	C

Calculation

$$\begin{aligned} \Delta H_{in} = & (mC_p\Delta T)_{\text{phosphoric acid}} + (mC_p\Delta T)_{\text{calcium nitrate}} + (mC_p\Delta T)_{\text{hydrofluric acid}} + \\ & (mC_p\Delta T)_{\text{carbon dioxide}} + (mC_p\Delta T)_{\text{magnesium nitrate}} + (mC_p\Delta T)_{\text{nitric acid}} + \\ & (mC_p\Delta T)_{\text{Iron nitrate}} + (mC_p\Delta T)_{\text{water}} \end{aligned}$$

$$\Delta H_{in} = 379852.974 \text{ kJ/hr}$$

$$\begin{aligned} \Delta H_{out} = & (mC_p\Delta T)_{\text{phosphoric acid}} + (mC_p\Delta T)_{\text{calcium nitrate}} + (mC_p\Delta T)_{\text{hydrofluric acid}} + \\ & (mC_p\Delta T)_{\text{carbon dioxide}} + (mC_p\Delta T)_{\text{magnesium nitrate}} + (mC_p\Delta T)_{\text{nitric acid}} + \end{aligned}$$

$$(mcp\Delta T)_{\text{Iron nitrate}} + (mCp\Delta T)_{\text{water}}$$

Where, $\Delta T_1 = \text{inlet/outlet} - \text{datum temperature}$

$$\Delta H_{\text{out}} = -569779.46 \text{ kJ/hr}$$

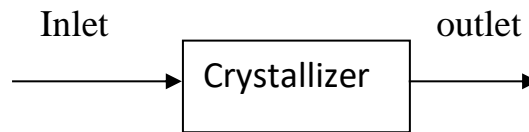


Table 17. Energy balance around crystallizer

	IN (kJ/hr)	Out (kJ/hr)
Ca₃(PO₄)₂.CaF₂	0	0
CaCO₃	0	0
MgCO₃	0	0
Fe₂O₃	0	0
H₃PO₄	27125.073	-40687.61
Ca(NO₃)₂	102532.244	-153798.37
HF	4767.56	-7151.34
CO₂	25090.966	-37636.449
Mg(NO₃)₂	51870.796	-77710.96
Fe(NO₃)₃	82380.796	-123571.19
HN0₃	9279.20	-13918.802
H₂O	76869.828	-115304.74
Heat of reaction	-	-
Total	376504.697	-569779.46

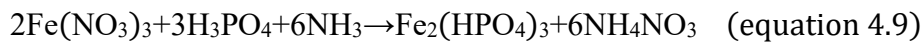
$$Q = \Delta H_{\text{out}} + \Delta H_{\text{in}}$$

$$Q = -949632.436 \text{ kJ/hr}$$

4.4 Energy balance around Neutralizer:

The reactions that are taking place inside the neutralizer is as follows:





neutralizer is used to convert the unreacted components into the useful products using the ammonia gas that is coming from the refrigeration unit. The temperature of the system is raised from 10 to 120 C and excess water is removed as vapors through condenser

General energy balance equation: -

$$\Delta H_{\text{in}} - \Delta H_{\text{out}} + \Delta H_{\text{generation}} - \Delta H_{\text{consumption}} = \Delta H_{\text{accumulation}}$$

Where,

$$\Delta H = m \int_{T_1}^{T_2} C_p dT$$

But if the variations in C_p at T_1 and T_2 are very small, we can take an average of C_p , hence $C_p = C_{p \text{ average}}$

Therefore,

$$\Delta H = m C_{p \text{ average}} \Delta T$$

We will use the above mention equation for calculating ΔH .

Assumptions: -

- Since process is steady state the accumulation term becomes zero.
- System is reactive therefore $\Delta H_{\text{generation}}$ and $\Delta H_{\text{consumption}}$ are not zero.
- Utility used is steam to heat the process streams and is at 120.2°C and 2bar.
- reference temperature = 25 C (298K)

T_{in}	10.00	C
T_{out}	120.00	C
T_{ref}	25.00	C

Calculations: -

$$\Delta H_{\text{in}} = (mC_p\Delta T)_{\text{phosphoric acid}} + (mC_p\Delta T)_{\text{calcium nitrate}} + (mC_p\Delta T)_{\text{ammonia}} +$$

$$(mC_p\Delta T)_{\text{aluminium nitrate}} + (mC_p\Delta T)_{\text{magnesium nitrate}} +$$

$$(mC_p\Delta T)_{\text{nitric acid}} + (mC_p\Delta T)_{\text{Iron nitrate}}$$

$$\Delta H_{\text{in}} = -569779.1 \text{ kJ/hr}$$

$$\Delta H_{\text{out}} = (mC_p\Delta T)_{\text{ammonium dihydrogen phosphate}} + (mC_p\Delta T)_{\text{diammonium phosphate}} +$$

$$(mC_p\Delta T)_{\text{dicalcium phosphate}} + (mC_p\Delta T)_{\text{ammonium nitrate}} +$$

$$(mC_p\Delta T)_{\text{iron hydrogen phosphate}} + (mC_p\Delta T)_{\text{dimagnesium phosphate}} + \Delta H_{\text{reaction}}$$

Where, $\Delta T_1 = \text{inlet/outlet temperature} - \text{datum temperature}$

Similarly,

$$\Delta H_{\text{reaction}} = n_1 \times \Delta H_{\text{reaction 1}} + n_2 \times \Delta H_{\text{reaction 2}} + n_3 \times \Delta H_{\text{reaction 3}} + n_4 \times \Delta H_{\text{reaction 4}}$$

Here, n1, n2, n3, n4 are the moles of species involved in the respective reactions

And

Heat of reaction 5 =	1081.1	kJ/mol
Heat of reaction 6 =	-132	kJ/mol
Heat of reaction 7 =	-2786.61	kJ/mol
Heat of reaction 8 =	-3435.7	kJ/mol
Heat of reaction 9 =	-1560	kJ/mol
Heat of reaction 10 =	-970.8	kJ/mol

$$\Delta H_{\text{reactions}} = -19626.59 \text{ kJ/hr}$$

$$\Delta H_{\text{out}} = 13413726 \text{ kJ/hr}$$

$$\Delta H_{\text{waste}} = [(mC_p \Delta T_1)_{\text{carbon dioxide}} + (mC_p \Delta T_1 + m\lambda + mC_p \Delta T_2)_{\text{water}}]$$

$$\Delta H_{\text{waste}} = 174263077.4 \text{ kJ/hr}$$

↑ *scrubber*

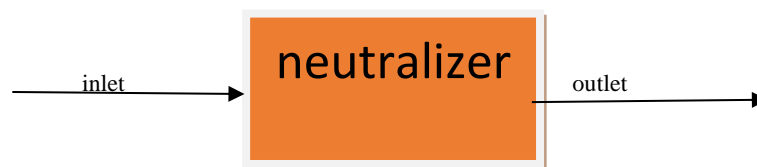


Table 18. Energy balance around neutralizer

	IN (kJ/hr)	Out (kJ/hr)	Waste (kJ/hr)
3Ca₃(PO₄)₂.CaF₂	0	0	
CaCO₃	0	0	
MgCO₃	0	0	
Fe₂O₃	0	0	
H₃PO₄	-40687.6	0	
Ca(NO₃)₂	-153798	159659.20	

HF	-7151.34	394.94	13413.536
CO₂	-37636.45	0	238364.177
Mg(NO₃)₂	-77710.96	423307.374	
Fe(NO₃)₃	-123571.2	666744.74	
HN0₃	-13918.8	0	
H₂O	-115304.7	11196662.2	174011299.7
NH₃	0	181277.908	
NH₄NO₃	0	517899.77	
NH₄H₂PO₄	0	7926.37	
(NH₄)₂HPO₄	0	60769.713	
CaHPO₄	0	86194.965	
Fe₂(HPO₄)₃	0	29198.16	
MgHPO₄	0	103316.91	
Heat of reaction	0	-19626.587	
Total	-569779.1	13413725.7	174263077.4

$$Q = \Delta H_{\text{out}} + \Delta H_{\text{waste}} - \Delta H_{\text{in}}$$

$$Q = 188246582.1 \text{ kJ/hr}$$

4.5 Energy balance around Evaporator:

Evaporator helps in removing the extra water from the stream entering into it

mass of water evaporated	18656.367	kg
latent heat of water	334	kJ/kg
total heat load =	6231226.578	kJ

$$Q = 6231226.578 \text{ kJ/hr}$$

4.6 Prilling tower and cooling exchanger:

Conditions

T _{in}	120	C
T _{out}	30	C
T _{ref}	25	C

Table 19. Energy balance around Prilling tower

	IN (kJ/hr)	Out (kJ/hr)
Ca₃(PO₄)₂.CaF₂	0	0
CaCO₃	0	0
MgCO₃	0	0
Fe₂O₃	0	0
H₃PO₄	0	0
Ca(NO₃)₂	159659.2	8403.116
HF	394.94	68.18
CO₂	0	0
Mg(NO₃)₂	423307.4	22279.34
Fe(NO₃)₃	666744.7	35091.83
HN0₃	0	0
H₂O	11196662	1968.75
NH₃	181277.9	9540.94
NH₄NO₃	517899.8	27257.88
NH₄H₂PO₄	7986.372	417.1775
(NH₄)₂HPO₄	60769.71	3198.406
CaHPO₄	86194.96	4536.577
Fe₂(HPO₄)₃	29198.16	1536.745
MgHPO₄	103316.9	5437.732
Heat of reaction	-23944.896	
Total	13409407	119736.8

$$Q = \Delta H_{\text{out}} - \Delta H_{\text{in}} = -13289670.55 \text{ kJ/hr}$$

Chapter No. 5 Design section

5.1 Introduction of Reactor:

The science and engineering are related for the designing of reactor. It highlighting how diverse knowledge and information are critical to the design and operation of chemical reactors. It is shown in the figure.

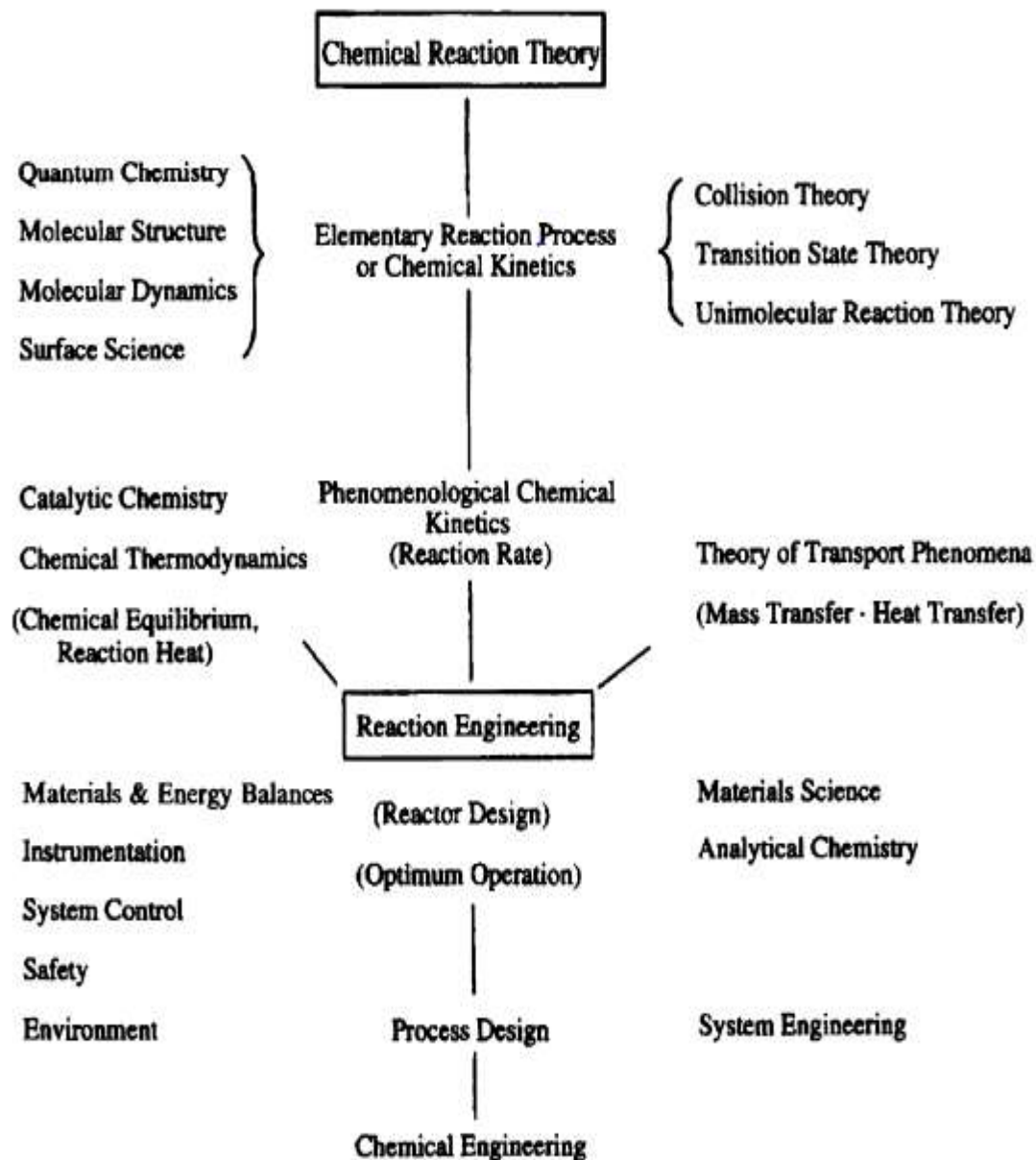


Figure 2. criteria of design reactor

Chemical kinetics is a scientific or academic subject that focuses on elucidating underlying principles on a molecular scale that influence the pace of chemical reactions.

A chemical reaction, typically one that is straightforward. The goal of kinetics in engineering, often known as chemical reaction engineering, is to provide a viable technique for reactor design and operation, not only for simple but also for complicated reaction systems, in which

mass and heat transport have significant impacts on reaction rates. The bubbling column reactor, in which a gas is blown into a liquid, and the catalytic reactor, in which a gas travels through a porous granular catalyst bed, are two examples of reactors where mass transfer of reactants in heterogeneous phases is crucial. The mass transfer on the gas/liquid interface, and the diffusion rate of gaseous molecules in the pores of the catalyst, respectively, regulate the reaction rate and product distribution in the former case and the latter case, respectively.

High-temperature thermal cracking and partial oxidation of hydrocarbons are examples of heat transfer-controlled reaction systems. Because the former case is accompanied by a large amount of heat absorption, the heat transfer rate through the reactor tube wall substantially governs the reaction rate, whereas in the latter case, minimizing the side reaction, namely complete oxidation of hydrocarbons, requires elimination of the large amount of heat generated and control of the reaction temperature.

The notion of reactor design is to first identify the preferred reaction conditions in order to efficiently carry out the planned chemical reaction, and then to select the reactor type and size. More specifically, the reactor operating parameters should be adjusted from both an economic and technical standpoint to accomplish the given targets established for both production size and product quality. The process scheme is configured, the reactor type is chosen, and the reactor's size and structure are determined to reduce both operational and investment costs.

Chemical reactions are characterized from a variety of perspectives, each with its own set of properties. Reactors can also be classed based on their kinetic characteristics, as seen below.

1. Batch Reactor

2. Continuous flow Reactor

Batch reactors are most commonly employed for liquid-phase systems with a sluggish reaction rate. This type of reactor is useful for small-scale operations manufacturing colors and medicines in commercial applications. Furthermore, the reactor has the benefit of being multi-purpose; a range of products may be created using only one reactor in a batch process.

The continuous flow type reactor, in which raw materials are continually fed and the result is extracted, is well suited to large-scale chemical processes requiring a high reaction rate under generally constant working conditions.

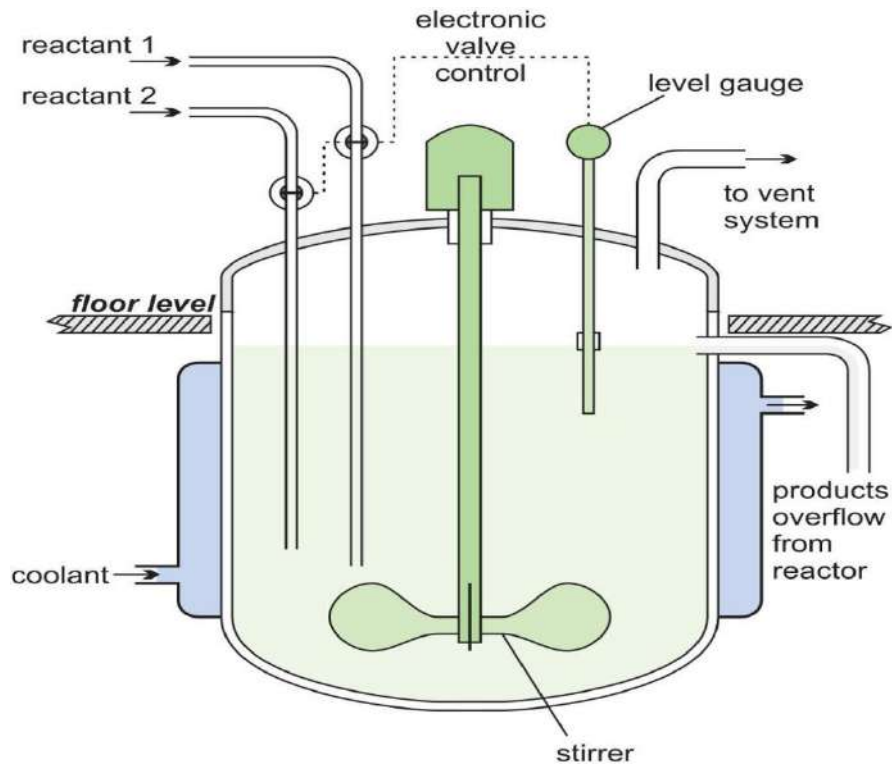


Figure 3: Continuous Stirrer Tank Reactor (CSTR).

5.2 Continuous Stirrer Tank Reactor (CSTR):

The Continuous Stirred Tank Reactor, or CSTR, is a continuous reactor that is considered to be at steady-state, with a net accumulation of zero. The mass balance equation below explains the core idea of this reactor. Where F_{j0} denotes the flow of intake, F_j denotes the flow of output, and G_j is the mass generated during the reaction. Because it follows a fundamental template of an input flow of reactants and an output flow of products having gone through a stirring activity, this ideal reactor type is a template of many industrial key unit activities employing agitated stirring reactor tanks.

$$F_{j0} + G_j - F_j = \frac{dN_j}{dt} \quad \text{equation 5.1}$$

Where;

F_{j0} = Denotes the flow of intake

F_j = Denotes the flow of output

G_j = Is the mass generated during the reaction.

Because it follows a fundamental template of an input flow of reactants and an output flow of products having gone through a stirring activity, this ideal reactor type is a template of many industrial key unit activities employing agitated stirring reactor tanks.

5.3 Continuous Stirred Tank Reactor Design:

Continuous Stirred Tank Reactors are proffered over Batch systems for Industrial production because

- Compatible Product Quality
- Low capital expense
- Decreased operating cost per unit of product
- Since CSTR involves continuous mixing so there would be no accumulation or buildup of soaps and mixture would be perfectly homogenized.

5.4 Material of Construction:

Material of Construction chosen is Stainless Steel 304 Type because

- Excellent Corrosion resistance to caustic media
- Coupled with good strength, ductility, toughness and ease of fabrication
- No Corrosion products
- Resists the formation of deposits, resulting in no contamination issues
- Low cleaning costs
- The temperature limitation for stainless 304 Type is about 1037 Centigrade whereas system temperature is 60 Centigrade so can be used safely.

5.5 Design of Dissolving Reactor:

Continuous stirred tank reactor

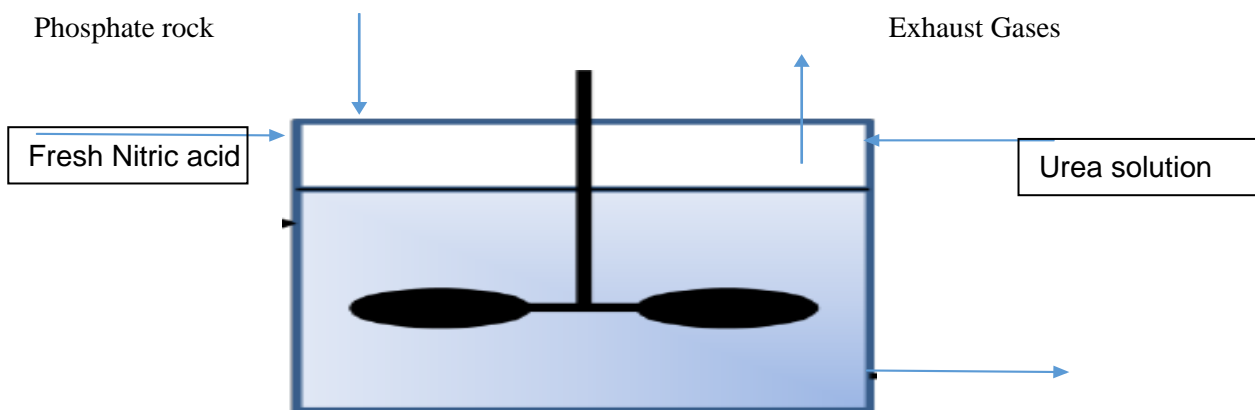
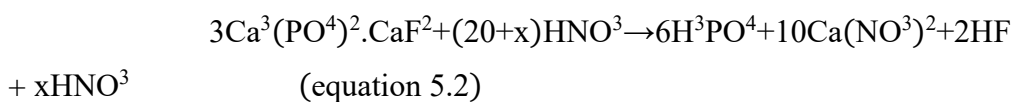


Figure 4. Continuous stirrer tank reactor



5.51 Design equation of C.S.T.R:

$$V = F_{AO} \cdot \frac{X}{-r_A}$$

$$F_{AO} = V_o C_{AO}$$

F_{AO} = Initial molar flow rate of specie A, $3\text{Ca}(\text{PO}^4)^2 \cdot \text{CaF}^2$

$$F_{AO} = 4.25 \text{ Kmol/hr}$$

Total volumetric flow rate

$$V_O = V_{AO} + V_{BO}$$

$$V_{AO} = \frac{\text{mass flow rate of } \frac{\text{kg}}{\text{m}^3}}{\text{density of stream 01}}$$

$$\begin{aligned} V_{AO} &= (\text{mass flow rate of kg/m}^3) / \text{density of stream 01 kg/m}^3 \\ &= 4293.5 / 3140 \\ &= 1.367 \text{ m}^3/\text{hr} \end{aligned}$$

$$\text{Density of HNO}_3 \text{ (60\% conc) at } 25^\circ\text{C} = 1360 \text{ kg/m}^3$$

$$\begin{aligned} V_{BO} &= 18711.7 / 1360 \\ &= 13.758 \text{ m}^3 / \text{hr} \end{aligned}$$

$$\begin{aligned} \text{Total } V_o &= V_{Ao} + V_{Bo} \\ &= 1.367 + 13.758 \\ &= 15.125 \text{ m}^3/\text{hr} \\ C_{Ao} &= F_{Ao} / V_o \\ &= 4.25 / 14.125 \\ &= 0.281 \text{ kmol /m}^3 \end{aligned}$$

For irreversible reaction

$$-r_A = K A C_A^\alpha \cdot C_A^\beta$$

In this case

$$-r_A = K A C_A^\alpha \cdot C_A^\beta$$

As HNO₃ is in excess than 3Ca₃(PO₄)₂.CaF₂. so

$$C_B \approx C_{BO}$$

$$-r_A = K A C_A^\alpha \cdot C_{BO}^\beta$$

$$K' = k_A C_{BO}^\beta$$

$$-r_A = K' C_A^\alpha$$

5.52 Determining of rate law:

1- Postulate a rate law

$$-r_A = K C_A^\alpha \cdot C_A^\beta$$

2- Look for simplifications

As HNO₃ is used in excess $C_B \approx C_{BO}$

$$-r_A = K' C_A^\alpha \text{ where } K' = K_A C_{B0}^\beta$$

3- Apply C.R.E algorithm r_A

$$\text{Mole balance } dN_A / dT = R_{av}$$

$$\text{Rate law } -r_A = K' C_A^\alpha$$

$$C_A = N_A / V_0$$

Combining

$$dN_A / dT = -K' C_A^\alpha$$

$$-dC_A / dt = k' C_A^\alpha$$

Takin log on both sides

$$\ln [-dC_A / dt] = \ln k' + \alpha \ln C_A$$

$$\ln [-dC_A / dt] = \alpha \ln C_A + \ln k'$$

$$y = mx + c$$

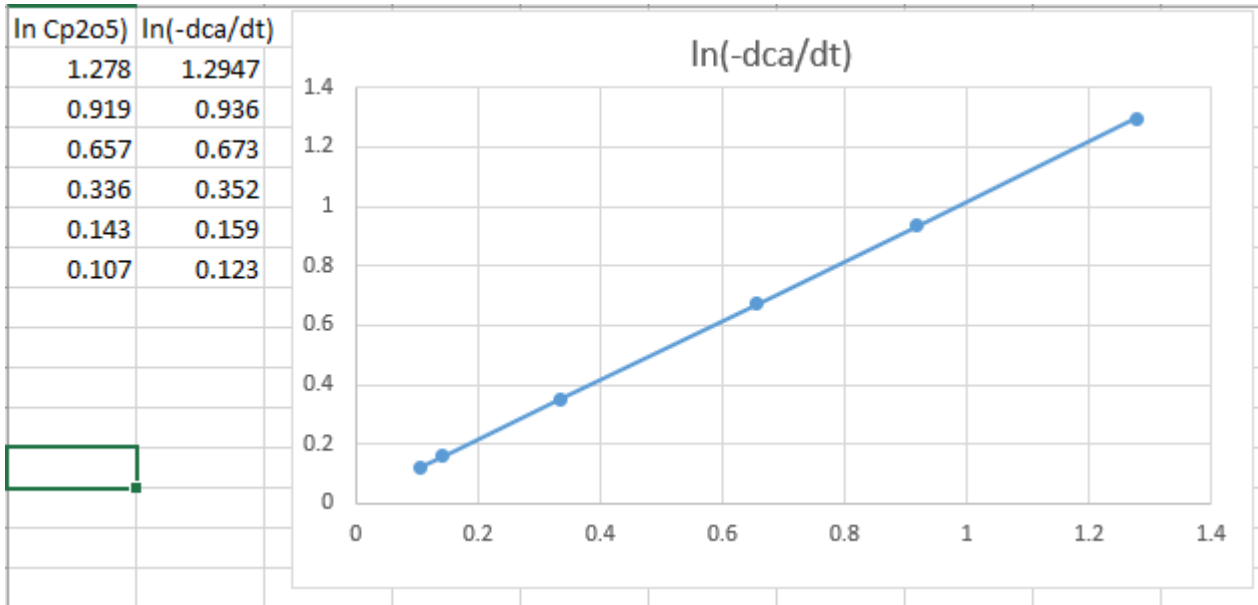
Slope of graph between $\ln [-dC_A / dt]$ and $\ln C_A$ will yield the reaction order w.r.t $3Ca_3(P_0_4)_2 \cdot CaF_2$ and y intercept will yield value of $\ln k'$.

5.53 Reaction kinetics data:

Time(min)	Conc P_2O_5 (mol/dm ³) $\times 10^3$	$(-dca/dt) 10^{-3}$ mol/dm ³ .min
0	9.103	3.64
1	6.003	2.54
2	4.002	1.96
3	2.744	1.42
4	1.970	1.172
5	1.424	1.130

$\ln (p^2 o^5)$	$\ln \left(\frac{-dCA}{dt} \right) (10^{-3} \text{ mol/dm}^3 \cdot \text{min})$
1.278	1.2947
0.919	0.936

0.657	0.673
0.336	0.352
0.143	0.159
0.107	0.123



From graph α slope is 1.

$$\alpha = 1.0$$

$$\ln [-dCa / dt] = \alpha \ln CA + \ln k'$$

for $k'=?$

for $\ln [-dCa / dt]= 1.2947$ and $\ln CA= 1.278$

$$k' = 1.0159/\text{min}$$

for $\ln [-dCa / dt]= 0.673$ and $\ln CA= 0.657$

$$k' = 1.0160/\text{min}$$

for $\ln [-dCa / dt]= 0.352$ and $\ln CA= 0.336$

$$k' = 1.0159/\text{min}$$

So k' will be 1.0159/min.

$$\begin{aligned}
C_{Bo} &= F_{Bo} / V_o \\
&= 297 / 15.125 \\
&= 19.65 \text{ kmol/m}^3
\end{aligned}$$

The reaction is first order. So $\beta = 1$

$$\begin{aligned}
K &= k' / C_{Bo} \\
&= 1.0159(\text{min}^{-1}) / 19.65(\text{kmol/m}^3) \\
&= 3.102 \text{ m}^3/\text{kmol.hr}
\end{aligned}$$

5.54 Rate law:

$$\begin{aligned}
-r_A &= K \times C_A \cdot C_B \\
C_A &= C_{P_2O_5} \\
C_B &= C_{HNO_3}
\end{aligned}$$

Using stoichiometry

$$X = 0.99$$

$$\begin{aligned}
\Theta_B &= C_{A0} / C_{B0} \\
&= 0.281 / 19.65 \\
&= 69.928
\end{aligned}$$

$$C_A = C_{A0}(1 - X)$$

$$C_B = C_{A0}(\Theta_B - 20X)$$

$$C_A = C_{A0}(1 - X)$$

$$C_A = 0.281(1 - 0.99)$$

$$C_A = 2.81 \times 10^{-3} \text{ kmol/m}^3$$

$$C_B = C_{A0}(\Theta_B - 20X)$$

$$C_B = 0.281[69.928 - 20(1 - 0.99)]$$

$$C_B = 14.085 \text{ kmol/m}^3$$

$$-r_A = K \times C_A \cdot C_B$$

$$= 3.102 \times (2.81 \times 10^{-3}) \cdot (14.085 \text{ kmol/m}^3)$$

$$= 0.1228 \text{ kmol/m}^3 \cdot \text{hr}$$

$$V = F_{A0} \cdot X / -r_A$$

$$= 4.219 \times 0.99 / 0.1228$$

$$= 34.01 \text{ m}^3$$

Optimum L/D ratio = 1.4 (James.M. Douglas)

$$\text{Volume of reactor} = 3.14D^2 \cdot L / 4$$

$$34.01 \times 4 = 3.14D^2 \times (1.4D)$$

$$D = 3.14 \text{ m}$$

$$L = 1.4D$$

$$L = 1.4(3.14)$$

$$L = 4.396 \text{ m}$$

5.6 Mechanical design of reactor:

5.61 Material of construction:

Stainless steel will be used as a material of construction as it shows good corrosion resistance to nitric acid up to temperature as high as 100 centigrade.

(Appendix B corrosion chart Gavin Tower page 1103)(Tower and Sinnott 2013)

5.62 Design pressure:

Taking as 10% above operating gage pressure.

$$= 1 \text{ atm} \times 1.1$$

$$= 1.1 \text{ atm as } 1 \text{ atm} = 101325 \text{ N/m}^2 \text{ and } \text{m}^2 = (1000)^2 \text{ mm}^2$$

$$= 0.1115 \text{ N/mm}^2$$

Adding hydrostatic pressure

$$= (3140 + 1360/2) (9.81) (L)$$

$$= 97030.71 \text{ kg/ m.s}^{-2}$$

$$= 0.097 \text{ N/mm}^2$$

Pi= hydrostatic pressure + operating pressure

$$= 0.1115 + 0.097$$

$$= 0.2085 \text{ N/mm}^2$$

5.63 Design temperature:

Maximum working temperature= 70 °C= 158 °F

From table 13.2 (gavin towler)(Tower and Sinnott 2013)

At 158 maximum allowable stress can be determined through interpolation

$$= 20 + \frac{158-100}{300-100} (15-20)$$

$$= 18.55 \text{ Ksi}$$

$$1 \text{ Ksi} = 1000 \text{ Psi and } 1000 \text{ Psi} = 6.8948 \text{ N/mm}^2$$

$$\text{Maximum allowable stress} = 127.554 \text{ N/mm}^2$$

Cylindrical section:

The equation specified by ASME BPV code (sec V111 D1 part U.G 27) is

$$t = \frac{P_i \cdot D_i}{2SE - 1.2P_i}$$

P_i = internal pressure = 0.2085 N/mm²

D_i = internal diameter = 3140 mm

S = maximum allowable stress = 127.554 N/mm²

E = welded joint efficiency from table 13.3 Gavin Tower

Because Phosphoric acid is toxic substance. So welds will be fully radiographically examined. So $E = 1.0$

$$t = \frac{(0.2085)(3140)}{2(127.554)(1) - 1.2(0.2085)}$$

$$= \frac{654.69}{254.8578}$$

$$= 2.57 \text{ mm}$$

Adding corrosion allowance of 2mm

$$= 4.57 \text{ mm}$$

5.64 Design of ends:

Trying Hemispherical Heads

$$t = (P_i \cdot D_i) / (4SE - 0.4P_i) \quad (\text{Tower and Sinnott 2013})$$

$$t = ((0.2085)(3140)) / (4(127.554)(1) - (0.4)(0.2085))$$

$$= 654.69 / 510.1326$$

$$= 1.283 \text{ mm}$$

Adding corrosion allowance of 2mm

$$= 3.283 \text{ mm}$$

Trying Ellipsoidal

$$T = (P_i \cdot D_i) / (4SE - 0.4P_i)$$

$$t = (0.2085)(3140) / (2 * 127.554 - (0.2) * (0.2085))$$

$$= 654.69 / 255.06$$

$$= 2.57 \text{ mm}$$

Adding corrosion allowance of 2mm

$$= 4.57 \text{ mm}$$

So hemispherical head will be more economical

5.65 Design of Agitator of reactor

As a starting point for design in ordinary agitator problems a turbine agitator is commonly used.

Typical proportions are

$$\frac{D_a}{D_t} = \frac{1}{3}$$

$$\frac{H}{D_t} = 1$$

$$\frac{J}{D_t} = \frac{1}{12}$$

$$\frac{E}{D_t} = \frac{1}{3}$$

$$\frac{W}{D_a} = \frac{1}{5}$$

$$\frac{L}{D_a} = \frac{1}{4}$$

(unit operations of McCabe & smith page 243)(McCabe 2018)

$$\text{As } D_t = 3140 \text{ mm}$$

$$D_a = \text{impeller dia} = D_t/3 = 1.05 \text{ m}$$

For design of baffled agitator vessel, the following values are recommended (by McCabe & smith)

Using 4-blade 45° standard turbine

$$\frac{W}{D_a} = \frac{1}{5}$$

$$W = 1.05/5$$

$$= 0.21 \text{ m}$$

$$NQ = \text{flow number} = q/n D_a^3$$

Where N = turbine speed

Q = volumetric flow rate through impellers

$$\text{Given } Q = V_r' A_p$$

V_r' = radial velocity of liquid leaving the blades.

As fluid leaves the impellers blades the radial component of fluid velocity V_r at centerline of impeller is about 0.6 times of tip speed of impeller(x2)

$$U_2 = \pi D_a n$$

$$A_p = \pi D_a W$$

$$= (3.14) (1.05) (0.21)$$

$$=0.692 \text{ m}^2$$

$$\text{Assuming value of } n = 30 \frac{\text{rev}}{\text{min}}$$

$$U_2 = \pi D a n$$

$$= (3.14) (1.05) (30)$$

$$= 98.91 \text{ m/min} \quad 1 \text{ minute} = 60 \text{ seconds}$$

$$= 1.64 \text{ m/s}$$

$$Q = \pi a^2 U_2 = 0.6 U_2 A_p$$

$$= (0.6) (1.64) (0.692)$$

$$= 0.680 \text{ m}^3/\text{s}$$

$$NQ = Q / (n D a^3)$$

$$NQ = 0.680 / ((0.5)(1.05)^3)$$

where n is taken in seconds.

$$= 1.174$$

5.66 Calculation of power consumption

$$D a = 1.05 \text{ m} = 3.44 \text{ ft}$$

$$N = 0.5 \text{ rev/s}$$

Density of phosphate solution = ?

$$\begin{aligned} \text{Density of } \text{H}^3\text{PO}^4 &= \left(\frac{2503}{30976.43} \right) \times 1.88 \frac{\text{g}}{\text{cm}^3} \\ &= 0.151 \text{ g/cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Density of } \text{Ca}(\text{NO}^3)^2 &= \left(\frac{12010.92}{30976.43} \right) \times 2.5 \frac{\text{g}}{\text{cm}^3} \\ &= 0.969 \text{ g/cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Density of HF} &= \left(\frac{170.27}{30976.43} \right) \times 1.015 \frac{\text{g}}{\text{cm}^3} \\ &= 5.58 \times 10^{-3} \text{ g/cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Density of } \text{Co}^2 &= \left(\frac{2955.83}{30976.43} \right) \times 1.562 \frac{\text{g}}{\text{cm}^3} \\ &= 0.149 \text{ g/cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Density of } \text{Mg}(\text{NO}^3)^2 &= \left(\frac{5403.44}{30976.43} \right) \times 2.3 \frac{\text{g}}{\text{cm}^3} \\ &= 0.401 \text{ g/cm}^3 \end{aligned}$$

$$\begin{aligned} \text{Density of } \text{Fe}(\text{NO}^3)^3 &= \left(\frac{5566.27}{30976.43} \right) \times 1.68 \frac{\text{g}}{\text{cm}^3} \\ &= 0.301 \text{ g/cm}^3 \end{aligned}$$

$$\text{Density of HNO}_3 = \left(\frac{536.37}{30976.43} \right) \times 1.51 \frac{\text{g}}{\text{cm}^3}$$

$$= 0.0261 \text{ g/cm}^3$$

$$\text{Density of H}_2\text{O} = \left(\frac{1830}{30976.43} \right) \times 1 \frac{\text{g}}{\text{cm}^3}$$

$$= 0.059 \text{ g/cm}^3 \quad \text{Density of phosphate solution} = 2.06 \text{ g/cm}^3$$

$$= 2060 \text{ kg/m}^3 \quad \text{and } 128.6 \text{ lb/ft}^3$$

$$\text{Viscosity of phosphate solution} = 10 \text{ cp} = 24.19 \text{ lb/ft.h}$$

$$= 6.719 \times 10^{-3} \text{ lb/ft.s}$$

$$\text{NRe} = \text{Da}^2 \times n \times \frac{\rho}{\mu}$$

$$= \frac{(3.44)^2 (0.5)(128.6)}{6.719 \times 10^{-3}}$$

$$= 113246$$

$N_p = 5$ from unit operations from McCabe & Smith figure 9.12 (McCabe 2018)

$$P = N_p \times n^3 \times \text{Da}^5 \times \frac{\rho}{g_c}$$

$$P = \frac{(5)(0.5)^3(3.44)^5(128.6)}{32.17}$$

$$P = 1203.5 \text{ ft.lbf/s} \quad 1 \text{ ft.lbf/s} = 0.00181818 \text{ hp}$$

$$P = 2.188 \text{ hp} \quad 1 \text{ hp} = 0.7457 \text{ KW}$$

$$P = 1.63 \text{ KW}$$

5.8 Design of Heat Exchanger:



5.8a Physical properties:

Phosphate Solution (hot Solution):

Table. Physical properties of streams

Mass Flow rate (kg/hr)	30976.43		
Properties	Inlet	mean	outlet

Temperature °C	70	52.5	35
Heat Capacity (KJ/kg °C)		2.7	
Thermal Conductivity (W/m. K)		0.64	
Density (kg/m ³)		2060	
Viscosity (lb/ft.s)		6.719x10 ⁻³	
Mass Flow rate (kg/hr)	14124.2		
Properties	Inlet	mean	Outlet
Temperature °C	25	35	45
Heat Capacity (KJ/kg °C)		4.2	
Thermal Conductivity (W/m. K)		0.59	
Density (kg/m ³)		997	
Viscosity (Ns/m ²)		0.8x10 ⁻³	

5.8b Specification:

Hot fluid: Phosphate solution

Cold fluid: Cooling Water

Fluid Allocation

Phosphate solution is at high temperature and causes corrosion so it is taken on the tube side

Design Steps:

Following steps are done during designing of heat exchanger.

- mass flowrates
- log mean temperature, temperature correction factor
- true mean temperature
- required area
- number of tubes, tube passes
- tube and shell side heat transfer coefficient
- Overall Heat Transfer Coefficient U
- pressure drop of both shell and tube side

Q of water:

$$Q=1186432.53 \text{ Kj}$$

$$m=?$$

$$C_p= 4.2 \text{ KJ/kg. } ^\circ\text{C}$$

$$\Delta T = 45 - 25 \text{ }^\circ\text{C} = 20^\circ\text{C}$$

$$Q = m \cdot C_p \cdot \Delta T$$

$$m = \frac{Q}{C_p \cdot \Delta T}$$

$$m = 14124.2 \frac{\text{Kg}}{\text{hr}}$$

$$m = 3.92 \frac{\text{Kg}}{\text{s}}$$

5.8c Log Mean Temperature Difference:

$$\text{LMTD} = \frac{\Delta T_1 - \Delta T_2}{\ln\left(\frac{\Delta T_1}{\Delta T_2}\right)}$$

$$\Delta T_1 = 70 - 45 = 25^\circ\text{C}$$

$$\Delta T_2 = 35 - 25 = 10^\circ\text{C}$$

$$\text{LMTD} = \frac{25 - 10}{\ln\left(\frac{25}{10}\right)}$$

$$= 16.38 \text{ }^\circ\text{C}$$

To achieve greater equipment compactness and also to make the equipment economical, one shell pass and two tube passes layout of heat exchanger is selected.

$$R = \Delta T_{\text{hot}} / \Delta T_{\text{cold}}$$

$$R = (70 - 35) / (45 - 25)$$

$$R = 1.75$$

$$S = \Delta T_{\text{cold}} / \Delta T_{\text{hot}}$$

$$S = (45 - 25) / (70 - 35)$$

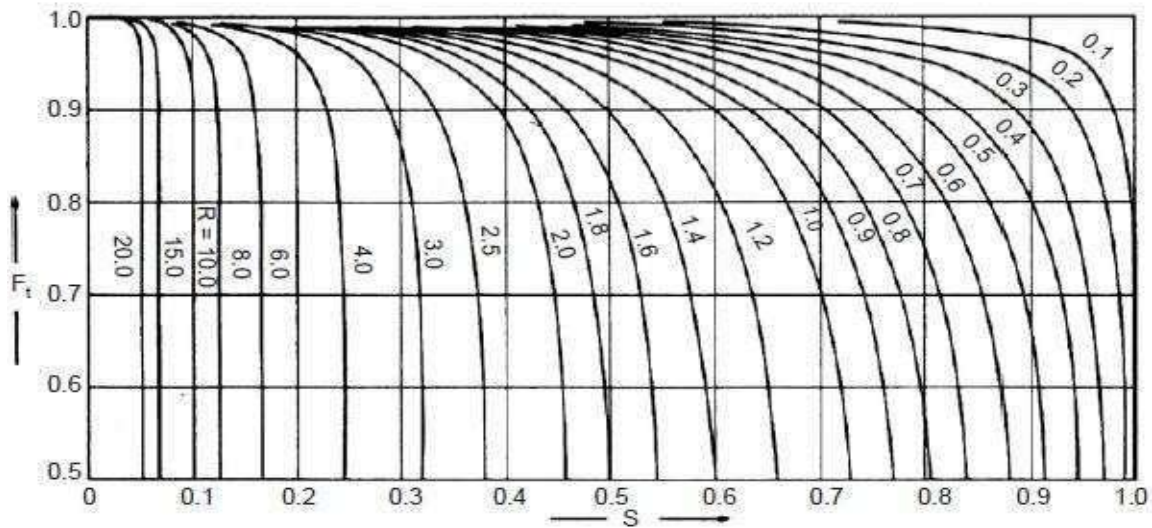
$$S = 0.51$$

5.8d Temperature correction factor, Ft:

From Figure (Gavin Tower) value of F_T can be noted against $S=0.51$ and $R= 1.75$

T_1

Figure 12.19. Temperature correction factor: one shell pass; two or more even tube passes.



By intercepting the lines of “S” and “R” lines it is found that

$$F_t = 0.8$$

$$\Delta T_m = LMTD \times F_t$$

$$\Delta T_m = 16.38 \times 0.8 = 13.1 \text{ } ^\circ\text{C}$$

Fluid allocation:

Because phosphate solution has a greater tendency to cause corrosion and fouling compared to water, so tube side will be allocated to phosphate solution and shell side will be allocated to cooling water. From Table 15-6 (Peter’s) estimating overall heat transfer coefficient from range of 250-500 (BTU/hr-ft². °F) for aqueous solution as hot fluid and water as cold fluid.

$$\text{Taking } U = 350 \text{ (BTU/hr-ft}^2 \cdot \text{ } ^\circ\text{F)}$$

$$U = 613.04 \text{ W/m}^2 \cdot \text{K}$$

Heat transfer area:

$$Q = 329.56 \text{ KJ/s}$$

$$A = Q / (U \times \Delta T_m)$$

$$A = 329.56 / (613.04 \times 13.1)$$

$$A = 0.04 \text{ KJ.m}^2/\text{W.s}$$

$$A = 41 \text{ m}^2$$

5.8f Layout and tube side dimension:

Tube diameters in range 5/8 in (16mm) or 2in (50mm) are used. The smaller diameter 5/8 in to 1in (16-25mm) are preferred for most duties, as they will give more compact and therefore cheaper exchanger. So from table 12.3 (Gavin Tower)

Tube specification:

Choose stainless steel tube, having

Table 12.3. Standard Dimensions for Steel Tubes

Outside Diameter (mm)	Wall Thickness (mm)				
	1.2	1.6	2.0	—	—
16	—	1.6	2.0	2.6	—
20	—	1.6	2.0	2.6	3.2
25	—	1.6	2.0	2.6	3.2
30	—	—	2.0	2.6	3.2
38	—	—	2.0	2.6	3.2
50	—	—	2.0	2.6	3.2

Tube outer diameter= 25mm

Tube thickness at 25 mm diameter = 2.0mm

Tube inner diameter = 23mm

Tube length selected = 3.66m

Area of single tube:

$$A = 3.14 \times 25 \times 10^{-3} \times 3.66 = 0.2873 \text{m}^2$$

Number of tubes:

$$N_t = 41 / 0.2873$$

$$= 142 \text{ tubes}$$

Equilateral triangular pitch will be used because shell side fluid is water and it will not cause fouling problems. Tube pitch selected is 1.25 times the outer diameter of tubes.

Bundle diameter:

$$D_b = d_o \left(\frac{N_t}{K_1} \right)^{1/n_1}$$

D_o = tube outside diameter (mm)

N_t = number of tubes

K_1 and n_1 are constants

From table (Gavin Tower) for triangular pitch $p_t=1.25d_o$ and 2 number of passes

Table 12.4. Constants for Use in Equation 12.3

Triangular Pitch, $p_t = 1.25d_o$					
No. passes	1	2	4	6	8
K_1	0.319	0.249	0.175	0.0743	0.0365
$> n_1$	2.142	2.207	2.285	2.499	2.675
Square Pitch, $p_t = 1.25d_o$					
No. passes	1	2	4	6	8
K_1	0.215	0.156	0.158	0.0402	0.0331
n_1	2.207	2.291	2.263	2.617	2.643

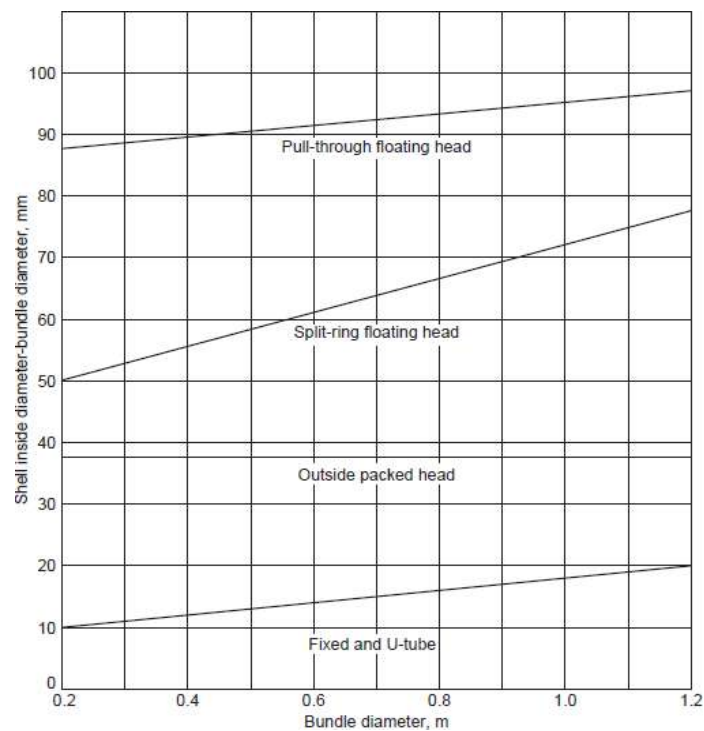
$$K_1 = 0.249$$

$$n_1 = 2.207$$

$$D_b = 25 \left(\frac{142}{0.249} \right)^{1/2.207}$$

$$D_b = 443 \text{ mm}$$

From figure 12.10 (Gavin Tower)



$$\text{Bundle diameter clearance} = 12 \text{ mm}$$

$$\text{Shell diameter } D_s = 12 + 443 = 455 \text{ mm}$$

Tube side coefficient:

$$\text{Mean temperature} = (70+35)/2 = 52.5^\circ\text{C}$$

Tube side cross sectional area:

$$(\pi/4) \times (23)^2 = 415.2 \text{ mm}^2$$

$$\begin{aligned} \text{Tubes per pass, } N_p &= \text{Number of tubes/number of passes} \\ &= 142/2 = 71 \end{aligned}$$

Total flow area:

$$= 71 \times 415.2 \times 10^{-6} = 0.029 \text{ m}^2$$

Mass velocity of phosphate solution:

$$= 8.604 / 0.029$$

$$= 296.7 \text{ kg/m}^2 \cdot \text{s}$$

Linear velocity of phosphate solution:

$$u_t = \text{mass velocity of phosphate solution} / \text{density of phosphate solution}$$

$$u_t = 296.7 / 2060$$

$$u_t = 0.15 \text{ m/s}$$

Reynolds number:

$$\text{Re} = (d_i \times u_t \times \rho) / \mu$$

$$\text{Density} = 2060 \text{ kg/m}^3$$

$$u_t = 0.15 \text{ m/s}$$

$$d_i = 23 \times 10^{-3} \text{ m}$$

$$\mu = 6.179 \times 10^{-3} \text{ lb/ft} \cdot \text{s}$$

$$\text{Re} = 773$$

Prandtl number:

$$pr = \frac{(C_p \mu)}{(k)}$$

$$C_p = 2.7 \text{ kJ/kg} \cdot ^\circ\text{C}$$

$$K = 0.64 \text{ W/m} \cdot \text{K}$$

$$\text{Pr} = 38.8$$

$$L/D_i = 3.66 / 23 \times 10^{-3}$$

$$= 160$$

JH factor:

From figure 12.23 (Gavin Tower)

$$JH = 5 \times 10^{-3}$$

By neglecting viscosity correction factor

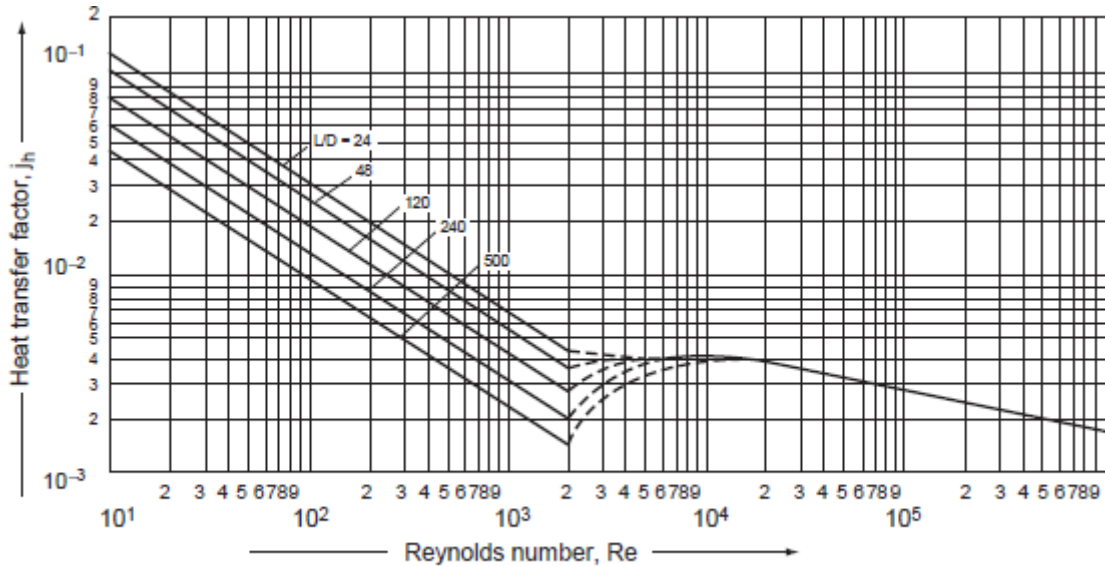


Figure 12.23. Tube-side heat transfer factor.

$$h_i = JH \times Re \times Pr^{0.33} \times \frac{k}{d_i}$$

$$h_i = 3597 \text{ W/m}^2 \cdot \text{°C}$$

shell side heat exchanger:

$$\begin{aligned} \text{baffle spacing} &= D_s/5 = 455/5 \\ &= 91 \text{ mm} \end{aligned}$$

Cross area of shell area:

$$A_s = \frac{(P_t - d_o) d_s \times L_B}{P_t}$$

$$P_t = 1.25 d_o$$

$$\text{where } d_o = 25 \text{ mm}$$

$$P_t = 1.25 \times 25 \text{ mm}$$

$$= 31.25 \text{ mm}$$

$$d_s = 455 \text{ mm}$$

$$L_B = \text{baffle spacing} = 91 \text{ mm}$$

$$A_s = \frac{(31.25 - 25) 455 \times 91}{31.25}$$

$$= 0.0082\text{m}^2$$

Shell side equivalent diameter:

$$\text{For equilateral triangular pitch } d_e = \frac{1.10}{d_o} (p^2 - 0.917d_o^2)$$

$$d_e = 17.75\text{mm}$$

Reynold's number:

$$Re = \left(\frac{D_e G_s}{\mu} \right)$$

$$\text{Mass flow rate of water} = 3.92\text{kg/s}$$

$$\text{Mass velocity} = 3.92/0.0082\text{m}^2$$

$$G_s = 478 \text{ kg/m}^2.\text{s}$$

$$U_s = \frac{478}{997}$$

$$\text{where } 997 \frac{\text{kg}}{\text{m}^3} \text{ is water density}$$

$$U_s = 0.48 \frac{\text{m}}{\text{s}}$$

$$Re = \left(\frac{(17.75 \times 10^{-3} \text{m})(478 \frac{\text{kg}}{\text{m}^2}.\text{s})}{0.8 \times \frac{10^{-3} \text{Ns}}{\text{m}^2}} \right)$$

$$Re = 10606$$

Prandtl number:

$$Pr = \frac{C_p \mu}{k}$$

$$C_p = 4.2 \text{ kJ/kg.}^\circ\text{C}$$

$$K = 0.59 \text{ W/m.k}$$

$$Pr = 5.694 \times 10^{-3} \text{ Kj/W.s}$$

$$Pr = 5.7$$

Taking 25% baffle cut.so From figure 12.29 (Gavin Tower)

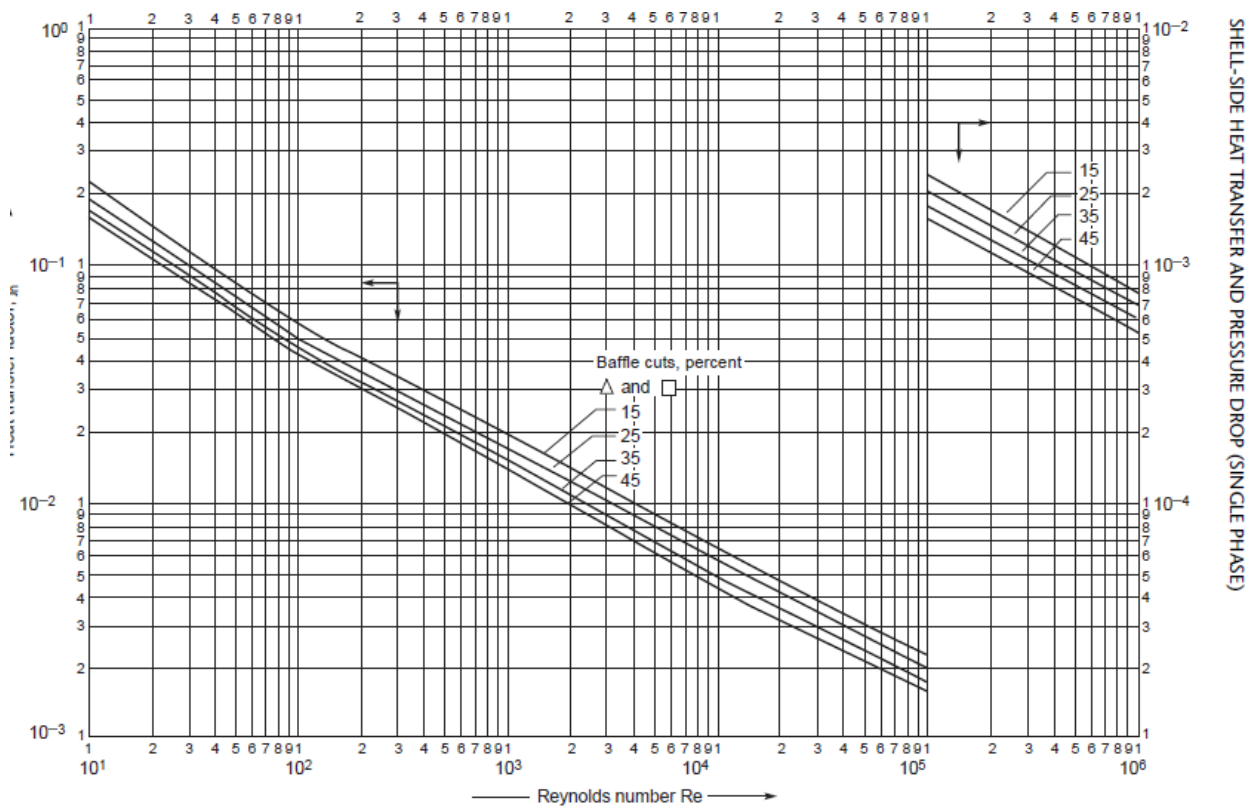


Figure 12.29. Shell-side heat transfer factors, segmental baffles.

JH factor:

$$jH = 5.8 \times 10^{-3}$$

$$h_s = \left(\frac{K_f}{d_e} \right) \times jH \times Re \times Pr^{0.33}$$

$$h_s = \left(\frac{0.59}{17.75 \times 10^{-3}} \right) \times 5.8 \times 10^{-3} \times 10606 \times 5.7^{0.33}$$

$$h_s = 3631 \frac{W}{m^2 \cdot ^\circ C}$$

fouling factors are taken from table 12.2 Gavin Tower for cooling water.

$$H_{od} = 6000 \frac{W}{m^2 \cdot ^\circ C}$$

For phosphate solution

$$H_{id} = 5000 \frac{W}{m^2 \cdot ^\circ C}$$

Overall heat transfer coefficient:

$$\frac{1}{U} = \frac{1}{h_o} + \frac{1}{h_{od}} + \frac{d_o \ln \left(\frac{d_o}{d_i} \right)}{2kw} + \frac{d_o}{d_i} \times \frac{1}{h_{id}} + \frac{d_o}{d_i} \times \frac{1}{h_i}$$

Material	Thermal Conductivity
	(W/m °C) (Btu/ft h F)
Admiralty (71 Cu - 28 Zn - 1 Sn)	111
Aluminum	205 - 250
Aluminum brass (76 Cu - 22 Zn - 2 Al)	100
Brass (70 Cu - 30 Zn)	99
Carbon Steel	45
Carbon-moly (0.5 Mo)	43
Chrome-moly steel (1 Cr - 0.5 Mo)	42
Chrome-moly steel (2 1/4 Cr - 0.5 Mo)	38
Chrome-moly steel (5 Cr - 0.5 Mo)	35
Chrome-moly steel (12 Cr - 1 Mo)	28
Copper	386
Cupro-nickel (90 Cu - 10 Ni)	71
Cupro-nickel (70 Cu - 30 Ni)	29
Inconel	19
Lead	35
Monel (67 Ni - 30 Cu - 1.4 Fe)	26
Nickel	62
Polypropylene	0.1 - 0.22
Red Brass (85 Cu - 15 Zn)	159
Stainless Steel, type 316 (17 Cr - 12 Ni - 2 Mo)	16
Stainless Steel, type 304 (18 Cr - 8 Ni)	16
Titanium	19

Figure. Thermal conductivity of different materials

$$K_w = 16 \frac{W}{m \cdot ^\circ C}$$

$$h_i = 3597 \frac{W}{m^2 \cdot ^\circ C}$$

$$d_o = 25 \text{ mm}$$

$$d_i = 23 \text{ mm}$$

$$\frac{1}{U} = \frac{1}{3631} + \frac{1}{6000} + \frac{25 \times 10^{-3} \ln\left(\frac{25}{23}\right)}{2 \times 16} + \frac{25}{23} \times \frac{1}{5000} + \frac{25}{23} \times \frac{1}{3597}$$

$$\frac{1}{U} = 1.0267 \times 10^{-3} \frac{W}{m^2 \cdot ^\circ C}$$

$$U = 974 \frac{W}{m^2 \cdot ^\circ C}$$

Below assumes value

$$Re = 773$$

Tube side pressure drop:

From figure 12.24

$$Jf = 9 \times 10^{-3}$$

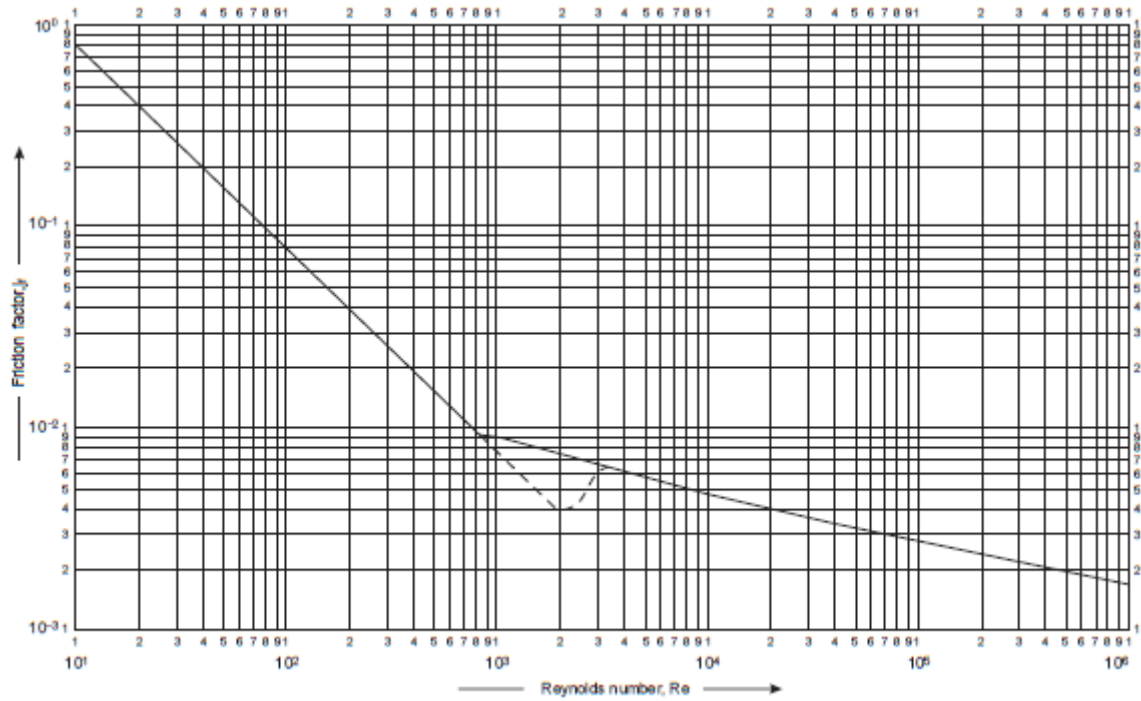


Figure 12.24. Tube-side friction factors.

$$\Delta P_t = (N_p [8j_f(L/d_i) (\mu/\mu_w)^{-m} + 2.5] \rho u_t^2) / 2$$

Number of tubes per pass, $N_p = 71$

$$\mu = 6.719 \times 10^{-3} \text{ lb/ft.s}$$

$$j_f = 9 \times 10^{-3} \quad \mu_w = 5.37 \times 10^{-4} \text{ lb/ft.s}$$

$$(\mu/\mu_w)^{-m} = 0.531 \quad \text{where } m = 0.25 \text{ for laminar flow } Re < 2100$$

$$\text{Length, } L = 3.66 \text{ m}$$

$$\text{Inner diameter, } d_i = 23 \times 10^{-3} \text{ m}$$

$$u_t = 0.15 \text{ m/s}$$

$$\rho = 2060 \text{ kg/m}^3$$

$$\Delta P = 14124 \text{ kg/m.s}^2$$

$$\Delta P = 14.124 \text{ kPa}$$

Shell side pressure drop:

$$Re = 10606$$

From figure 12.30

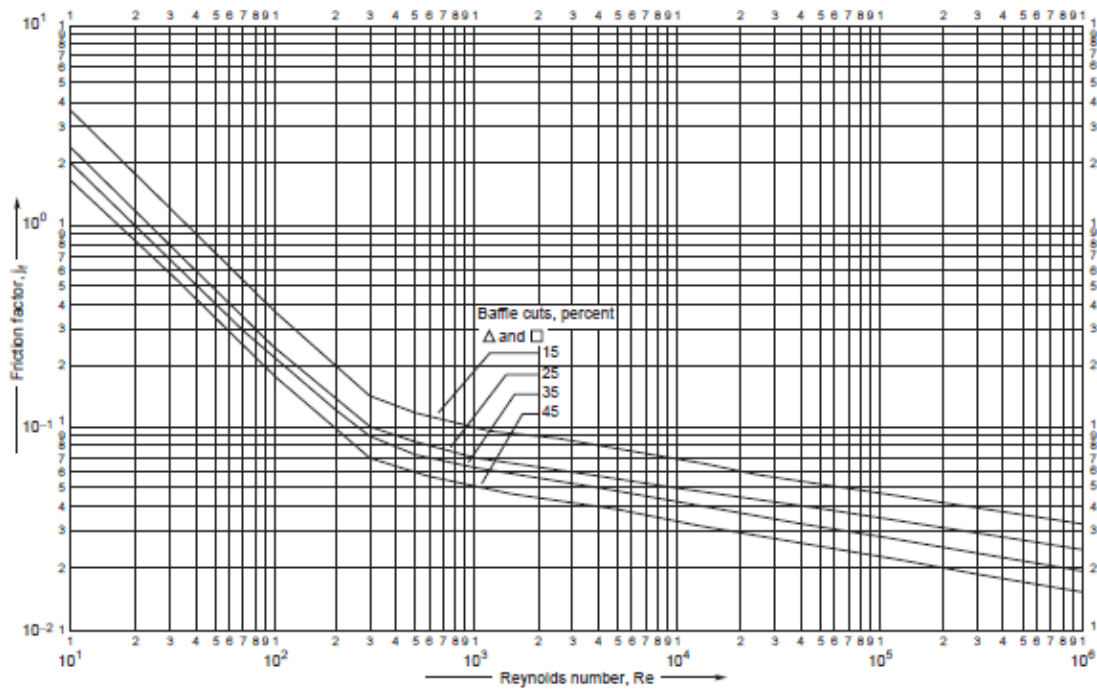


Figure 12.30. Shell-side friction factors, segmental baffles.

$$Jf = 5.8 \times \left[\frac{10}{Re} \right]^{-2} \Delta P_s = (8jf(D_s/de)(L/l_b)(\rho u_s^2)) \left(\frac{\mu}{\mu_w} \right)^{-m} / 2$$

Length of tube, $m = 3.66$

Baffle spacing, $l_b = 91 \text{ mm}$

Shell diameter, $D_s = 455 \text{ mm}$

Shell side equivalent diameter, $d_e = 17.75 \text{ mm}$

$$U_s = 0.48 \frac{\text{m}}{\text{s}}$$

$m = -0.14$ for turbulent flow

$$\Delta P_s = 38574 \frac{\text{kg}}{\text{m}} \cdot \text{s}^2$$

$$\Delta P_s = 38574 \text{ Pa}$$

$$\Delta P_s = 38.57 \text{ kPa}$$

Pressure drop could be reduced by increasing baffle spacing pitch.

Doubling the pitch halves the shell side velocity, which reduces the pressure drop by a factor

$$\text{of approximately } \left(\frac{1}{2} \right)^2$$

$$\Delta P_s = 38.57/4$$

$$\Delta P_s = 9.64 \text{ kPa}$$

Design of Evaporator:

Feed Conditions:

Temperature of feed = 91.5 C = 364.5 K

Temperature of bottom = 95 C = 368 K

Feed composition:

Feed flow rate = F = 46813.3 kg/hr

Flow rate of vapors = V = 18657.72 kg/hr

Flow rate of liquid = L = 28155.58 kg/hr

Wt% of water in feed = 59.94 %

Wt% of water in liquid = 0.33 %

Steam conditions

Steam Pressure = 1123.5 kPa

Temperature of steam at this pressure = 185 C = 458 K

Latent heat of steam = 1996 kJ/kg

Evaporator Conditions

Pressure inside evaporator = 110 kPa

Latent heat of vapor (water) at this pressure = 2250 kJ/kg

Temperature of vapor (water) at this pressure = 102.5 C = 375.5 K

Overall heat transfer coefficient of evaporator = 1500 W/ m² K

Boiling Point Elevation = T_{BPE} = 38.9 °C

Since vapors are superheated due to boiling point elevation. So,

$$h_v = \lambda_s + C_p \times T_{BPE}$$

$$h_v = 2250 + 1.884 \times 38.9 = 2323.3 \text{ kJ/kg}$$

Energy balance around the evaporator

$$S\lambda_s = Lh_L + Vh_v - Fh_F$$

Steam flowrate is calculated using:

$$S = \frac{Lh_L + Vh_v - Fh_F}{\lambda_s}$$

$$S = \frac{(28155.58 \times 3347) + (18657.72 \times 2323.3) - (46813.3 \times 2023)}{1996} = 21483.3 \text{ kg/hr}$$

Duty of evaporator is calculated using:

$$q = S\lambda_s = 21483.3 \times 1996/3600 = 11911.3 \text{ kW}$$

Area of evaporator can be calculated using:

$$A = \frac{q}{U(T_s - T_1)}$$

$$A = \frac{11911.3 \times 1000}{1500(185 - 91.5)} = 84.93 \text{ m}^2$$

Steam economy is calculated using:

$$\text{Steam Economy} = \frac{V}{S} = \frac{18657.72}{21483.3} = 0.868 \frac{\text{kg vaporised}}{\text{kg steam used}}$$

Dimensions of Evaporator:

Using 25 mm OD and 5 m length tube

Area of one tube

$$A_o = \pi DL = \pi \times 0.025 \times 5 = 0.3927 \text{ m}^2$$

Number of tubes

$$N_T = \frac{A}{A_o} = \frac{84.93}{0.3927} = 216 \text{ tubes}$$

Bundle diameter

$$D_b = d_o \left(\frac{N_T}{K_1} \right)^{1/n_1} = 0.025 \left(\frac{216}{0.319} \right)^{\frac{1}{2.142}} = 0.524 \text{ m}$$

Taking 20% clearance on both sides;

$$D = 2 \times 1.2 \times D_b = 0.734 \text{ m}$$

Height of evaporator is calculated (taking 50% disengaging space);

$$H = 1.5 \times L = 7.5 \text{ m}$$

Chapter No. 6 Instrumentation and Control

The key to the effective and efficient production is the instrumentation and control over the process. Instrumentation plays a vital role in the process control. Instrumentation gives the indication of various process parameters such as flow, pressure, temperature and level. If these parameters are known and they start to deviate, the control loops can be useful in controlling these parameters. By this, not only the time, but production losses can also be saved.

Without instrumentation and control, a plant cannot be operated because all the steps and decisions are taken based on value of any physical or chemical parameter. Instrumentation not only provides the information and values of the parameters but also help us to control the quality of the product. Process greatly depends on the process parameters and these parameters greatly influence the yield and quality of the product. controlled, it can lead to some very severe accidents.

6.1 Instrumentation:

Instrumentation is an art of science of measurement and control. The basic aim of instrumentation used in measurement systems is to assign some numerical value to the measured variables for the process. It is an applied scientific technique that deals with the examination and design of measurement systems such as determining the magnitude of measured variables, performing some sort of operations on the variables by using various instruments and quantifying a variable. Variables are classified as either automatic or manual adjustable variables. Instruments checking the key process variables are fixed with automatic alarms to prepare the operators for any hazardous states. A wide-ranging computing device, ranging from set in microprocessors to out-and-out computers, is commonly engaged throughout the industry.

6.2 Control:

Process Control is the automatic control of an output variable. This is carried out by pick up of amplitude of output parameter from process and then comparing it to desired value or set point for the process. This control is then activated by sending an error signal back to control an input variable.

6.3 Purpose of Chemical Process Control:

Chemical Process Control serves the following purposes

- The Safe operation of a chemical process is the primary factor towards the wellbeing of plant personnel as well as the economic development. Therefore, various process parameters like operating pressure, temperature and concentration of chemicals should also be within the safe limits
- To ensure compliance with the desired production level and quality of final products. For example: To produce Nitro phosphate fertilizer with a capacity of 135,000 tons per year and 92% purity, a control loop is almost important to meet the desired targets
- To meet the Environmental Harmony. In this regard, various federal and state laws have specified limits for operating temperature, operating pressure and concentrations of chemicals as well as effluent flowrates

- The process of a plant must adapt to the market surroundings means the accessibility to raw materials and the ultimatum of the desired products. It must be as cost-effective and efficient as possible in the exploitation of energy, raw materials and capacity and labor.

6.4 Fundamentals of Control System

Following hardware elements are found in almost every control structure

- Chemical Process
- Measuring elements or sensors
- Transducers
- Transmission line controllers
- Final Control Element

6.5 Approaches to Control:

There exist several manners by which a process is monitored and then controlled. These methods depend upon the controller kinds and the action done to handle any process parameter. This controller action is influenced by the output signal from transmitter which is then related with the set (desired) point to the controller and on the deviation or error in between the two parameters.

6.6 Combinations of Control Actions

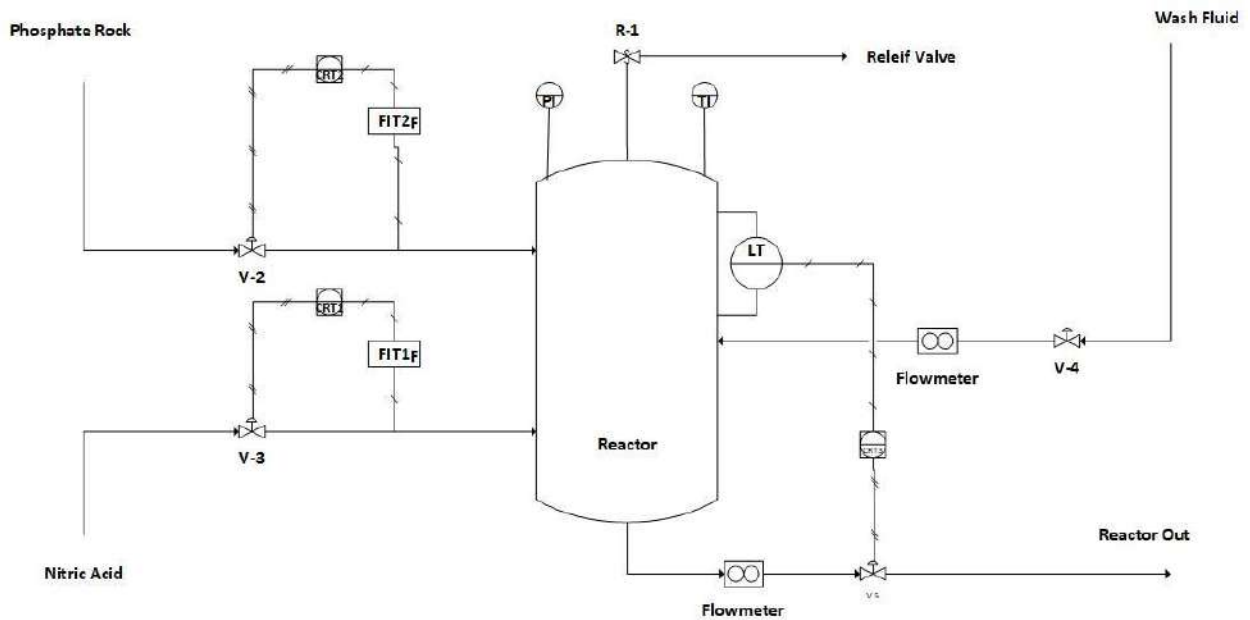
- Proportional Controller
- On-Off Controller
- Integral Controller
- Derivative or Rate Controller
- Composite Controller

6.7 Control Loops:

- Feed Backward Control Loop
 - Feed Forward Control Loop
 - Cascade Control Loop
 - Ratio Control Loop
 - Split Range Control Loop

6.8 Reactor:

The instrumentation and control diagram for the reactor provides the necessary information related to the process parameters. The instrumentation and control diagram for the reactor is shown below.



1. Figure 1: Control loop for the Reactor

Pressure and temperature indicators are installed on the reactor for monitoring pressure and temperature values. The pressure relief valve is also installed at the top of the reactor in order to release the excess pressure in case of any abnormality. The inlet material is controlled with the flow control loop. FIT1 and FIT2 are two flow indicators and transmitters for nitric acid and phosphate rock. V-2 and V-3 are the control valves operated pneumatically on the signal provided by the controller CRT1 and CRT2.

V-4 is the inlet valve placed at the inlet line of wash fluid. The flow meter along with totalizer is connected with the same line in order to record the flow of the was fluid in to the reactor.

Level transmitter Is also installed on the reactor. The purpose of the level indicator is to measure the level of the reactor and maintain the level by manipulating the outlet valve V-5. Controller compare the level signal with the set value and then transmit the signal to the control valve and thus the level of the reactor is maintained.

6.9 Heat Exchanger:

Heat exchanger is used to heat the process fluid using any utility. The heat transfer depends on the temperature of the process fluid and the utility. Therefore, it is necessary to maintain the outlet temperature of the feed so that the product quality can be maintained properly.

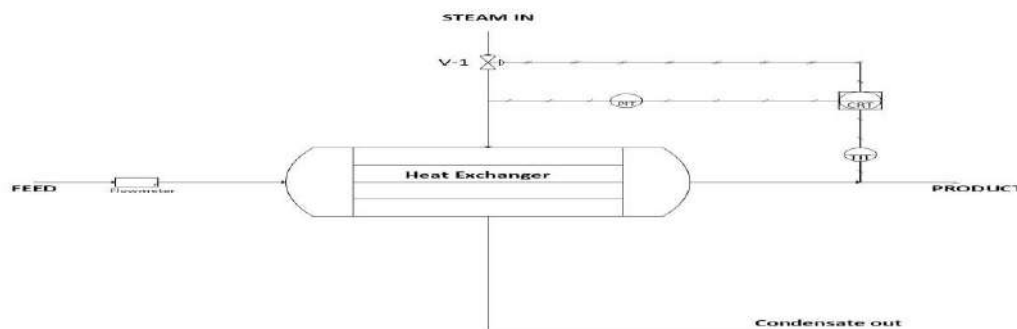


Figure 2: Instrumentation and control diagram for heat exchanger

Starting from the feed, the feed line has the flowmeter which measures the feed flow rate. There is a pressure indicator and transmitter installed on the steam inlet which gives the signal to the controller. The outlet temperature of the product is also monitored and the signal is transmitted to the controller. The controller is same for both of the parameters (pressure, temperature). The input signal is compared with the set point fed to the controller. After comparing in the comparator, the controller generates the signal and control the flow of the steam by manipulating valve V-1.

6.10 Pump:

Pump is used to transfer fluids from one point to another point. Pump 1 is being used to transfer the phosphate solution drawn from the phosphate solution tank and then transferred to the heat exchanger.

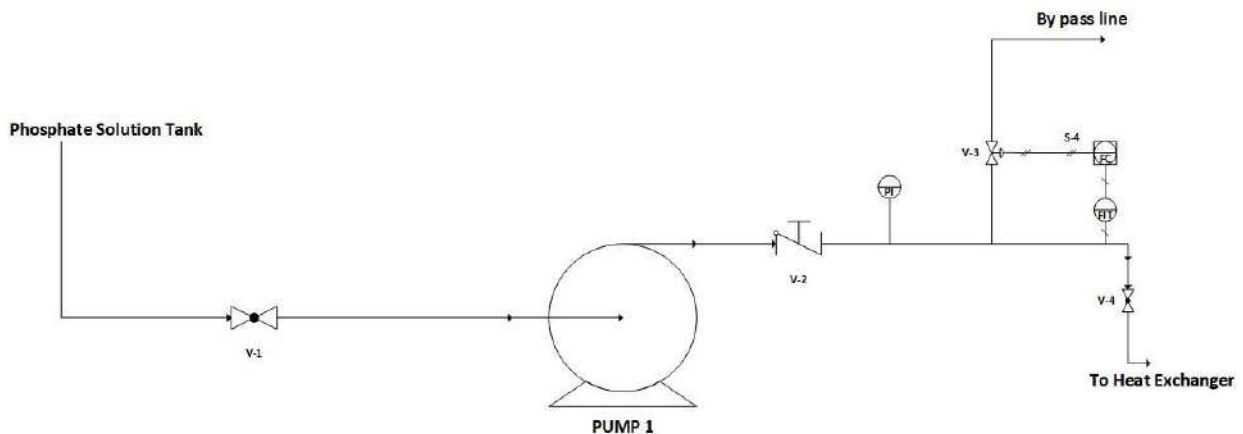


Figure 3: Instrumentation and control diagram for pump

the suction line has the globe valve at the inlet. This is due to maintain the net positive suction head for the pump suction. At the outlet of the pump, there is a valve V-2 which is a non return valve. This NRV helps to stop the back flow of fluid into the discharge line. A pressure indicator is also installed right after NRV because it will give the indication of the flow of fluid. Discharge line is divided into two lines. One line is used as a bypass line to control the fluid transfer to heat exchanger. FIT is flow indicator and transmitter that reads the flow and transmit the signal to the flow controller (FC). This FC compares the signal with the set value and then generates a signal through which the control valve V-3 is operated. V-4 is a simple globe valve which is used to connect or disconnect the discharge line of the pump. Heat exchanger can also be isolated if the valve V-4 is closed.

6.11 Scrubber:

The purpose of scrubber is to remove the carbon dioxide being produced as a result of fermentation. The instrumentation and control diagram of scrubber is as follows

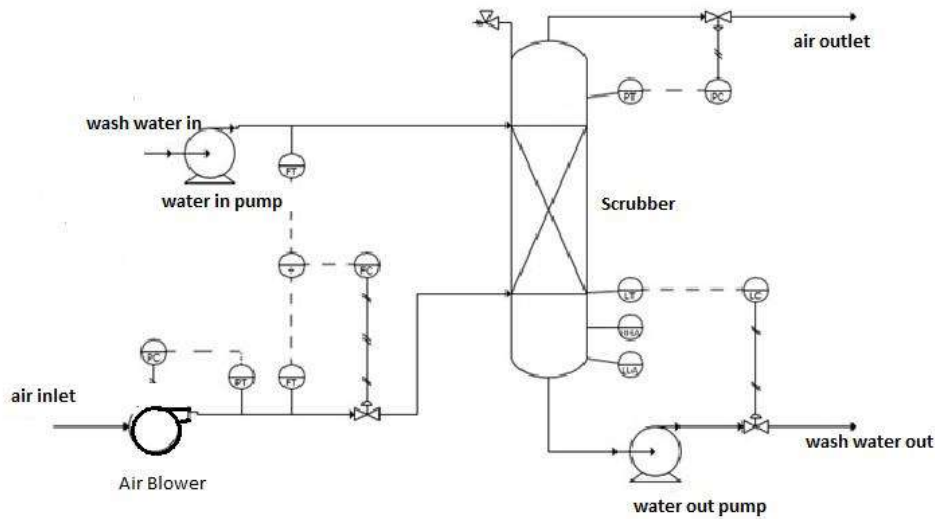


Figure 4: Scrubber instrumentation and control diagram.

As we can see from the above figure that the flow of air and water are fed to the scrubber. The flow of each of the component is measured using the flow transmitters and ratio controller is used which controls the flow rates of each of the component according to the changes. PSV is installed at the top of scrubber which helps in releasing the excess pressure for safe and smooth operations.

However, the exit air flow is regulated by monitoring the pressure of the column. If there is a rise in pressure in the column, the control valve opens and more air exits the column. Thus, releasing the excessive pressure. The flow of the rich solution is controlled by the liquid level inside the scrubber. If the liquid holdup starts to decrease, controller sends the signal to the downstream valve of water pump out and the valve is closed.

Moreover, the column also has the high-level alarm and low level alarm. This control strategy helps in regulating the parameters inside the scrubber to achieve maximum absorption.

Chapter No. 7 Cost Estimation

7.1 Economics Analysis:

This section deals with the overall economics of the suggested plant. The plant installation cost, operational cost, raw material costs and all the taxes have been incorporated into the economic study. All the costs are taken in USD because it is the standard currency, and all the economic evaluations are dealt in USD.

7.2 Purchase Equipment Cost:

PE is the purchased price of equipment from a vendor (someone selling the equipment). It is one of the major factors in the TCI direct costs. It includes the cost to build the equipment but does not include the cost associated with transportation of that equipment to the site, and installation, etc. The cost of each equipment must be calculated to find the purchase equipment cost. Bare module cost method is used for each equipment.

7.21 Storage Tanks:

The graph to determine purchased cost of storage tank is

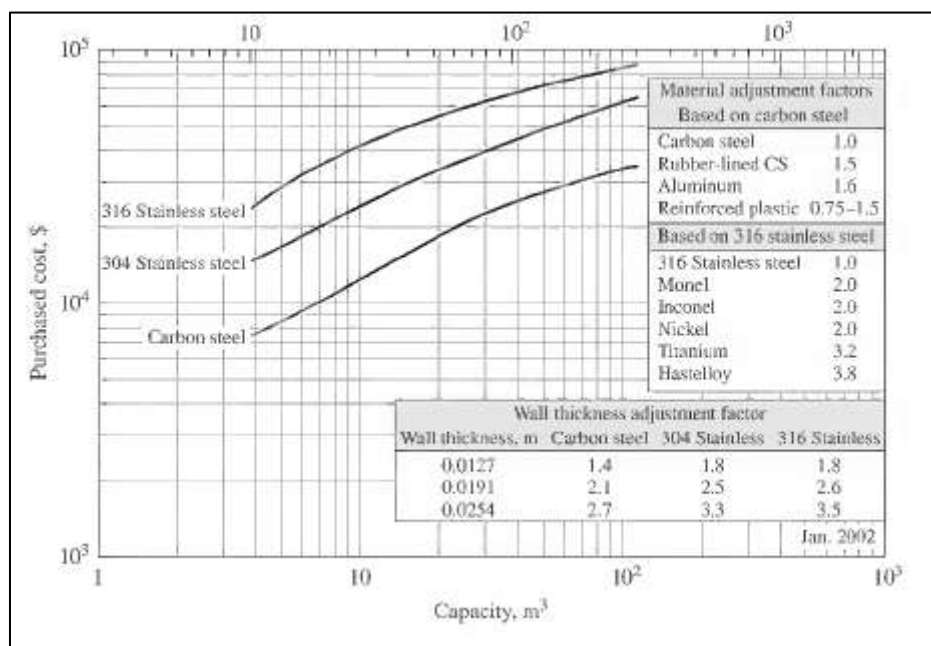


Figure 5: Graph for the Purchased Cost of Storage Tank

Material = Stainless Steel 304 Type

Bare cost = \$3000

Material factor = 2

Pressure factor = 1

Purchased cost = Bare Cost × Material Factor × Pressure Factor

$$= 3000 \times 2 \times 1 = \$6000$$

Cost index 1990 = 500.1

Cost index 2021 = 700.1

Cost in year 1990 = \$6000

Cost in year 2021 = Cost in year 1990 × (Cost index in 2021/Cost index in 1990)

Cost in year 2021 = 6000 × (700.1/500.1) = \$8400

We are using five storage tanks in our process so the total cost is

5x8400=\$42,000

Total Cost of Storage Tanks = \$42,000

7.22 Continuous Stirred Tank Reactor

The graph to determine purchased cost of Reactor is

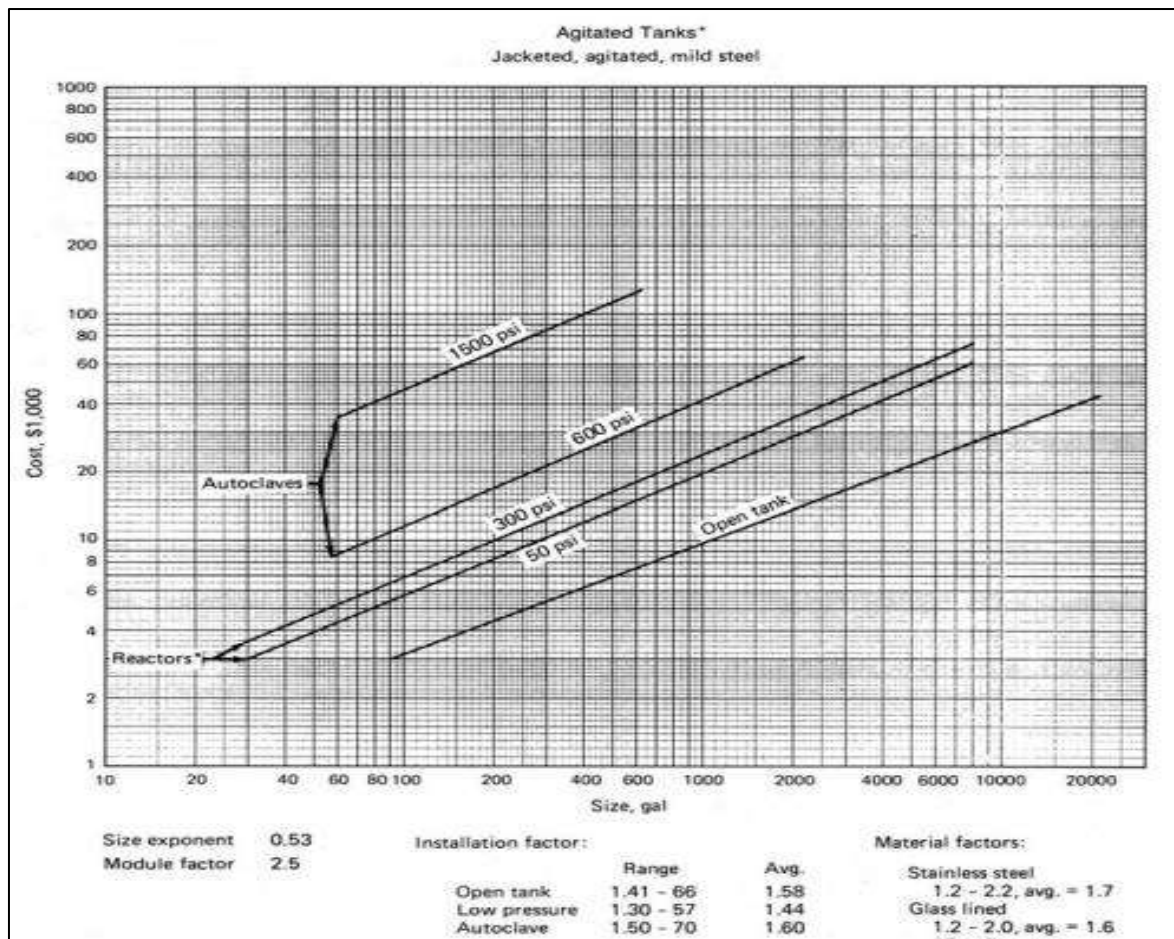


Figure 6: Graph for the Purchased Cost of Agitated Reactor

Volume of CSTR = 34 m³

Purchase Cost = \$ 25000

CEPCI (1987) = 350

CEPCI (2021) = 776.9

Cost of equipment = (cost index of 2021\CE of 1987) *purchased cost

Cost of equipment = \$ 55492

7.23 Shell and Tube Heat Exchanger

The graph to determine purchased cost of shell and tube heat exchanger is

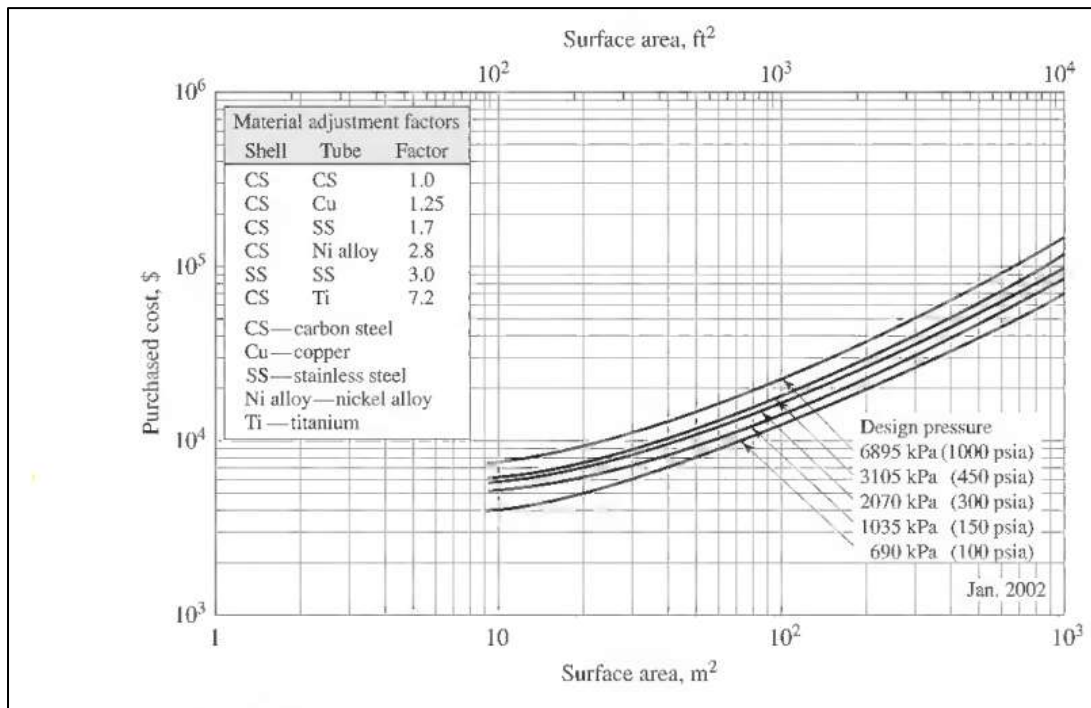


Figure 7: Graph for the Purchased Cost of Heat Exchanger

Surface area of Shell and Tube Heat Exchanger = 74m^2

Pressure = 43.51 psia

Purchased cost = \$6000 (Data published in year 2002)

Cost index for 2022 = 785

Cost index for 2002 = 395.6

Cost of equipment = (cost index of 2022 / CE of 2002) * purchased cost

Cost of Shell and Tube Heat Exchanger = \$11880

7.24 Evaporator

Material = Stainless Steel 304

Bare cost = \$5,000

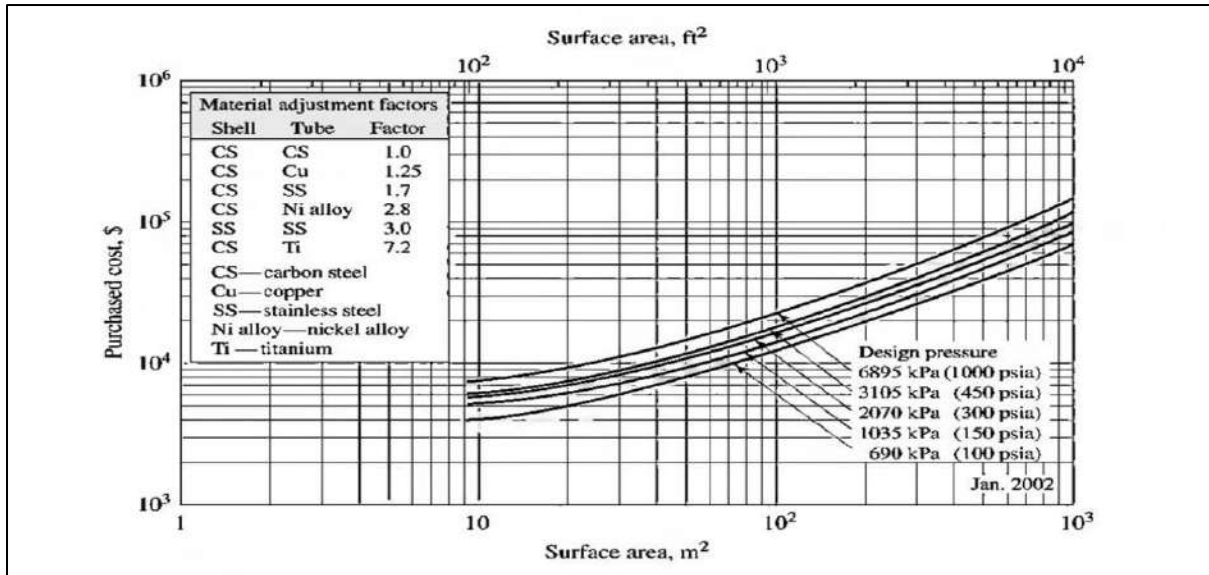


Figure 8: Graph to determine Purchased Cost of Evaporator

Material factor = 3

Pressure factor = 1

Purchased cost = Bare Cost × Material Factor × Pressure Factor

Purchased cost = \$15,000

Cost index 2002 = 364.6

Cost index 2021 = 787.4

Cost in year 2002 = \$15,000

Cost in year 2021 = Cost in year 2002 × (Cost index in 2021/Cost index in 2002)

Cost in year 2021 = 15,000 × (787.4/364.6)

Cost of Evaporator = \$ 32394.404

7.25 Centrifugal Pump:

Graph to determine purchased cost of Centrifugal Pump is

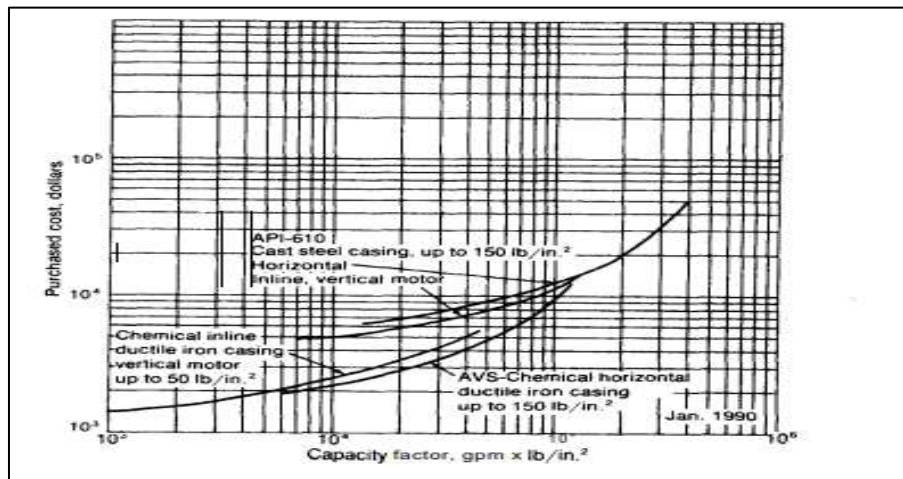


Figure 9: Graph to determine Purchased Cost of Centrifugal Pump

Mass flow rate of phosphate solution = 30703.36 kg/hr = 511.72 kg/min

Density of phosphate solution = 1480 kg/m³ = 5.60 kg/gal

Pressure = 300kPa = 43.51 psi

Capacity factor = 511.72*43.51/5.60

Capacity factor = 3975.8 gpm lb/in²

The graph for purchased cost of Centrifugal Pump is

Choosing the Inline Pump

Purchased Cost = \$1000 (Data is reported for Jan 1990)

CEPCI (1990) = 500

CEPCI (2022) = 785.9

Index Ratio = 785.9/500

Index Ratio = 1.572

Cost in 2021 = Purchased cost * Index ratio

Cost of CP-01 = \$1000 * 1.572

Cost of CP-01 = \$1572

We are using four pumps. So \$ 1572*4=\$6288

7.26 Crystallizer cost estimation:

Purchased cost of CSTR in 2004 = Bare cost from table 6.2 Coulson & Richardson Volume 6th in (2004) × material factor

Pressure Factor PF = 1

Material Factor = 1.56

Bare Cost of Crystallizer = 22231 \$

Cost Index in 2004 = 444

Purchase Cost in 2004 = Bare Cost × Pressure Factor × Material factor

Purchase Cost in 2004 = 22231 \$ × 1 × 1.56

Purchase Cost in 2004 = 34680 \$

According to CEPCI; Cost index of 2021 = 771.9

Cost Index is 23% higher in 2022.

Cost index in 2022 = Cost index in 2021 + (0.23×771.9)

Cost index in 2022 = 949

Purchase cost of Crystallizer in 2022 = purchase cost in 2004 × $\left(\frac{\text{cost index in 2022}}{\text{cost index in 2004}}\right)$

Purchase cost of Crystallizer in 2022 = 34680 \$ × $\left(\frac{949}{444}\right)$

Purchase cost of Crystallizer in 2022 = 74124 \$

7.27 Filter Drum cost:

Purchased cost of filter drum in 2004 = Bare cost from table 6.2 Coulson & Richardson Volume 6th in (2004) × material factor × pressure factor

$$\text{Pressure Factor PF} = 1$$

$$\text{Material Factor} = 1.56$$

$$\text{Bare Cost of filter drum} = 8800 \$$$

$$\text{Cost index in 2004} = 444$$

$$\text{Purchase Cost in 2004} = \text{Bare Cost} \times \text{Pressure Factor} \times \text{Material factor}$$

$$\text{Purchase Cost in 2004} = 8800 \$ \times 1 \times 1.56$$

$$\text{Purchase Cost in 2004} = 13728 \$$$

$$\text{According to CEPCI; Cost index of 2021} = 949$$

$$\text{Cost Index is 23\% higher in 2021.}$$

$$\text{Cost index in 2021} = \text{Cost index in 2021} + (0.23 \times 949)$$

$$\text{Cost index in 2022} = 949$$

$$\text{Purchase cost of filter drum in 2022} = \text{purchase cost in 2004} \times \left(\frac{\text{cost index in 2022}}{\text{cost index in 2004}} \right)$$

$$\text{Purchase cost of filter drum in 2022} = 13728 \$ \times \left(\frac{949}{444} \right)$$

$$\text{Purchase cost of filter drum in 2022} = 29337 \$$$

Now find; Bare Module Cost of filter drum:

$$C_{BM} = C_p(F_{BM})$$

$$C_{BM} = 45948 \$ \times 1.6$$

$$C_{BM} = 46938 \$$$

7.28 Cost estimation of pilling tower:

$$\text{Cost of pilling tower in 2017} = \$ 250,000$$

Cost index increase 23% every year. so

$$\text{Cost of pilling tower in 2021} = (0.23 \times 4 \times 250000) + 250000 = \$ 470000$$

7.29 Cost estimation of fluidized cooler bed:

Purchase cost of fluidized cooler bed in 2004 from Coulson and Richardson Volume 6th is

$$\text{Purchase cost of fluidized cooler bed in 2004} = 8670 \$$$

$$\text{According to CEPCI; Cost index of 2021} = 772$$

$$\text{Cost Index is 23\% higher in 2022.}$$

$$\text{Cost index in 2021} = \text{Cost index in 2021} + (0.23 \times 772)$$

$$\text{Cost index in 2022} = 949$$

$$\text{Purchase cost of fluidized cooler bed in 2022} = \text{purchase cost in 2004} \times \left(\frac{\text{cost index in 2022}}{\text{cost index in 2004}} \right)$$

$$\text{Purchase cost of fluidized cooler bed in 2022} = 8670 \$ \times \left(\frac{949}{772}\right)$$

$$\text{Purchase cost of fluidized cooler bed in 2022} = 10658 \$$$

7.20 Cost estimation of neutilizer:

Purchased cost of neutilizer in 2004 = Bare cost from table 6.2 Coulson & Richardson Volume 6th in (2004) × material factor × pressure factor

$$\text{Pressure Factor PF} = 1$$

$$\text{Material Factor} = 1.50$$

$$\text{Bare Cost of neutilizer} = 20218 \$$$

$$\text{Cost Index in 2004} = 444$$

$$\text{Purchase Cost in 2004} = \text{Bare Cost} \times \text{Pressure Factor} \times \text{Material factor}$$

$$\text{Purchase Cost in 2004} = 20218 \$ \times 1 \times 1.5$$

$$\text{Purchase Cost in 2004} = 30327 \$$$

$$\text{According to CEPCI; Cost index of 2021} = 772$$

$$\text{Cost Index is 23\% higher in 2021.}$$

$$\text{Cost index in 2021} = \text{Cost index in 2021} + (0.23 \times 772)$$

$$\text{Cost index in 2022} = 949$$

$$\text{Purchase cost of neutilizer in 2022} = \text{purchase cost in 2004} \times \left(\frac{\text{cost index in 2022}}{\text{cost index in 2004}}\right)$$

$$\text{Purchase cost of neutilizer in 2022} = 30327 \$ \times \left(\frac{949}{444}\right)$$

$$\text{Purchase cost of Crystallizer in 2022} = 64820 \$$$

7.3 Total Purchased Equipment Cost:

$$\text{Cost of storage tanks} = \$ 42000$$

$$\text{Cost of crystallizer} = \$ 74122$$

$$\text{Cost of filter drum} = \$ 46938$$

$$\text{Cost of CSTR reactor} = \$ 55592$$

$$\text{Cost of neutilizer} = \$ 64820$$

$$\text{Cost of heat exchanger} = \$ 11880$$

$$\text{Cost of pilling tower} = \$ 480000$$

$$\text{Cost of fluidized cooler bed} = \$ 10658$$

$$\text{Cost of evaporator} = \$ 32394$$

$$\text{Cost of centrifugal pumps} = \$ 6288$$

$$\text{Total cost} = \$ 824692$$

7.4 Direct Cost:

Installation Cost (40% of equipment cost) = \$329877

Instrumentation and Control Cost (13% of equipment cost) = \$107210

Piping Cost (32% of equipment cost) = \$263901

Electric Cost (10% of equipment cost) = \$82469

Building Cost (29% of equipment cost) = \$239161

Land Cost (6% of equipment cost) = \$49482

Total Direct Cost = \$1072100

7.5 Indirect Cost:

Engineering and Supervision Cost (32% of equipment cost) = \$263901

Construction Expenses (30% of equipment cost) = \$247408

Contractor fee (18% of equipment cost) = \$148445

Contingency fee (30% of equipment cost) = \$247408

Total Indirect Cost = \$907162

7.6 Total Capital Investment:

Fixed Capital Investment = Direct cost + Indirect Cost

Fixed Capital Investment = \$1979262

Working Capital (18% of Fixed Capital Investment) = \$356267

Total Capital Investment = Working Capital + Fixed Capital Investment

Total Capital Investment = \$2335529

7.7 Plant Cost:

Assume that the fixed capital investment devaluates by straight line method for 10 years.
Assuming 5% salvage value at the end of plant life.

Depreciation = $D = (V - V_s) / N$

$V =$ Fixed capital investment

$V_s = 0.05 * \text{Fixed Capital Investment}$

$V_s = \$116776$

$N = \text{No. of years} = 10$

$D = \$186248$

Total Plant Cost = Total Capital Investment + Depreciation

Total Plant Cost = \$2521777

7.8 Raw Material Cost:

Total Product Cost=?

Total Product Cost = Total Capital Investment – Depreciation

$$\text{Total Product Cost} = \$2335529 - 186248$$

$$\text{Total Product Cost} = \$2149281$$

$$\text{Fixed Charges} = 15\% \text{ of Product Cost} = \$322392$$

$$\text{Plant Overhead} = 50\% \text{ of Product Cost} = \$1076640$$

$$\text{Total Manufacturing Cost} = \text{Fixed Charges} + \text{Plant Overhead}$$

$$\text{Total Manufacturing Cost} = \$1397033$$

$$\text{General Expenses are } 20\% \text{ of Product Cost;}$$

$$\text{General Expenses} = \$429856$$

$$\text{Total Product Cost} = \text{Total Manufacturing Cost} + \text{General Expenses}$$

$$\text{Total Product Cost} = \$1826889$$

$$\text{Gross Income} = \text{Total Investment} - \text{Total Product Cost}$$

$$\text{Gross Income} = \$508640$$

$$\text{Let the tax rate is } 30\%$$

$$\text{Taxes} = 0.30(\text{gross income})$$

$$\text{Taxes} = \$152592$$

$$\text{Net Profit} = \text{Gross Income} - \text{Taxes}$$

$$\text{Net Profit} = \$356048$$

7.9 Rate of Return:

$$\text{Rate of Return} = (\text{net profit}/\text{total investment}) \times 100$$

$$\text{Rate of Return} = 15.24\%$$

7.9a Pay Back Period:

$$\text{Payback period} = \frac{1}{\text{Rate of Return}}$$

$$\text{Pay Back Period} = 6.5 \text{ years}$$

