PROCESS DESIGN OF NITROPHOS FERTILIZER BY UTILIZING NATIONAL PHOSPHATE RESERVES

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CERTIFICATE

This is to certify that work in this thesis has been completed by Ms. Adan Habib, Amna Amir, Mr. Hashir Anjum and Muhammad Tariq Jawed under the supervision of Dr. Sher Ahmad at the School of Chemical and Materials Engineering (SCME), National University of Science and Technology, H-12, Islamabad, Pakistan.

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DEDICATION

We would like to dedicate our work to our honorable parents and respected teachers, who supported and motivated us at every step. Their tremendous support and cooperation led us to this wonderful accomplishment.

ACKNOWLEDGEMENT

We would like to thank ALMIGHTY ALLAH, the Creator of universe, the most Beneficent the most Merciful, who gave us enough strength to accomplish this task. All respects are for His HOLY PROPHET (Peace be Upon Him), whose teachings are the true source of knowledge & guidance for the whole mankind.

We would like to thank our honorable Parents for devotion of their time. They support and encourage us whenever we need them.

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ABSTRACT

Nitro-phosphate (NP) is a complex acidic fertilizer which contains two macronutrients of plants, nitrogen (N) and phosphorous (P). NP is majorly used for rooting, blooming and fruit production of plants.

Demand of nitro-phosphate is increasing day by day with increasing demand for food and population growth. Increasing demand of NP and alkalinity of soil in Pakistan motivates us to work on this project.

Phosphate ore is an important raw material for manufacturing phosphorous chemical products. While most of the phosphate resources cannot be directly treated as feed stock due to the low grade of P_2O_5 and high content of silica and other metal impurities. For obtaining a qualified phosphate concentrate, the beneficiation of the low-grade phosphate ore is carried out to upgrade the P_2O_5 grade of phosphate ores from 20% to 28% using direct and reverse flotation techniques. This treated feed is used to make a NP fertilizer using ODDA Process.

This report includes material and energy balance, designing along with HAZOP and EIA study. The designing of each equipment (reactors, crystallizer, heat exchangers, centrifuges, evaporator and prilling tower) with designing is discussed in detail. The cost estimation of this plant was a major and compulsory part of this report. The plant capital cost and operating cost are also determined along with payback period (2.25 years). The payback period of the project is 2.25 years.

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CHAPTER 1

INTRODUCTION TO FERTILIZER

1.1 Fertilizer

The compound material that contains two or more elements required for a plant's healthy development and growth is known as fertilizer. Chemical or mineral fertilizers, plant remains, and manures are significant fertilizers. They could be organic or inorganic substances that the roots of the plants ingest to replenish vital nutrients that are lost through crop harvesting, leaching, or gaseous exchange.

We refer to these nutrients as plant nutrients. Although carbon, hydrogen, and oxygen are necessary for plants, they are not regarded as nutrients in the fertilizer industry because they are derived from water and atmospheric carbon dioxide (CO2). Up to 90–95 percent of the dry matter in all plants is made up of these three. Primary nutrients include nitrogen (N), phosphorus (P), and other key nutrients.

Table 1 Classification of elements necessary for plant growth

Major elements (Macronutrients)	Air & Water	Carbon , Hydrogen , Oxygen
	Primary Nutrients	Nitrogen , Phosphorous Potassium
	Secondary Nutrients	Calcium, Magnesium, Sulfur
Minor Elements (Micronutrients)		Chlorine, Boron, Copper, Molybdenum, Zinc, Manganese, Iron

Primary nutrients are commonly expressed as potassium oxide (K_2O), elemental nitrogen (N), and phosphorus pentoxide (P_2O_5). Although calcium and magnesium can occasionally be expressed in oxide form, secondary nutrients and micronutrients are often expressed in elemental form.

The most common way to represent fertilizers is with a series of numbers separated by dashes. This group of figures is known as the fertilizer grade. These percentages show how many nutrients are present in that fertilizer. For instance, 20-22 Nitro-phosphate (NP) contains 22% phosphorous (P2O5) and 20% nitrogen (N).

The fertilizer industry is directly or indirectly related to Pakistan's agriculture sector, which is the foundation of the country's economy. The installed capacity for Pakistan's manufacture of all phosphate fertilizers, including di-ammonium, is currently estimated at 3 million tonnes.

Table 2 Phosphate fertilizer production in capacities by industries in Pakistan

Company name	Type of fertilizer	Installed annual capacity (MTPA)
Fauji Fertilizer bin Qasim Ltd.	Di-ammonium Phosphate (DAP)	768,000
Al-Noor Fertilizers	Di-ammonium Phosphate (DAP)	390,000
Agritech Ltd.	Single Super Phosphate (SSP)	100,000
Suraj Fertilizers	Single Super Phosphate (SSP)	150,000
Allah Din Group	Single Super Phosphate (SSP)	90,000
Pak Arab Fertilizers	Nitro-phosphate (NP)	350,000
Fatima Fertilizers	Nitro-phosphate (NP)	350,000
Engro Fertilizers	Nitro-phosphate (NP)	40,000
	NPK	100,000

This sector accounts for:

1. 21% of the country’s gross domestic product (GDP).

2. 60% of employed labor in rural areas.
3. 65% of total Pakistan’s exports earnings.
4. 3% growth rate per annum
5. Provide raw materials for industries like sugar, textile, etc.

1.2 Nitro-phosphate (NP)

A complex fertilizer in the form of granules, nitrogen-phosphate (NP) primarily consists of nitrogen (N) and phosphorous (P_2O_5) with trace amounts of calcium (Ca). Phosphorus is advantageous for roots, blooming, and fruit production in plants, while nitrogen is a major nutrient required for efficient plant growth. It has a pH of 3.5 and is ideal for higher pH soils due to its acidic character. Farmers mostly employ the nitro-phosphate grades 2220, 20-20, and 18-18. Its molecular weight is 140.975 g/mol and its chemical formula is NO_6P^{-2}.

The name "nitro-phosphate" is also applied to fertilizers created by subjecting phosphate rock to nitric acid treatment.

Pakistan has a 740,000 MTPA installed capacity to produce nitrate-phosphate. Pak Arab Fertilizer is one of Pakistan's top NP fertilizer manufacturers.

1.3 History of product

Erling Johnson created the first method for producing nitro-phosphate fertilizer in 1928 by treating phosphate rock with nitric acid. Since this invention was created in the Norwegian town of ODDA, the procedure was given the name ODDA Process.

Following conception, he gave his patent to Norsk Hydro of Norway, who refined it in 1930 and released their own technology in 1938. The first business to produce multi-nutrients like NP and NPK using the Nitro-phosphate process was Norsk Hydro.

After some time, complex fertilizers (NP, NPK, etc.) were produced using a mixed acid technique that involved sulfuric acid. The mixed acid procedure was not frequently employed on a wide scale due to substantial emissions of H_2S and gypsum as a byproduct.

1.4 Physical properties & thermodynamic data

Table 3 Physical Properties

Property	Nitric acid	Ammonia	Phosphate rock	Nitro-phosphate
Molecular Formula	HNO_3	NH_3	$Ca_3(PO_4)_2$	$NH_4H_2PO_4$
Molecular Weight (g/mol)	63.02	17.03	310.18	115
Appearance	Colorless liquid	Colorless	Greyish Sedimentary rock	White/grey granules or prills
Smell	Pungent	Irritating	-	-
Boiling Point	86 ^oC	-33.35 ^oC	-	-
Melting Point	-41.59 ^oC	-77.7 ^oC	-	-
Vapor Pressure	47.8 mmHg at 20 ^oC	10.2 atm at 25 ^oC	-	-
Density (g/cc)	1.529	0.77	3.14	-

Table 4 Thermodynamic Data

Property	Nitric acid	Ammonia	Phosphate rock	Nitro-phosphate
Standard Molar Enthalpy of formation (ΔH) at 298.15 K (KJ/mol)	-174.1	-45.9	-	-
Standard Molar Gibbs energy formation (ΔG) at 298.15 K (KJ/mol)	-80.7	-16.4	-	-
Standard Molar Entropy at 298.15 K (J/mol-K)	155.6	192.8	-	-

1.5 Chemical reactions

Nitric acid is reacted with phosphate rock in first step by the following reaction,

$$Ca_3(PO_4)_2 + 6HNO_3 \rightarrow Ca(NO_3)_2 + 2H_3PO_4 + 22.25\ \text{kcal}$$

After removal of calcium nitrate crystals, nitro-phosphoric acid gets neutralized with ammonia to produce a fertilizer.

$$H_3PO_4 + 2NH_3 \rightarrow NH_4H_2PO_4 + 66.67\ \text{kcal}$$

Calcium nitrate is further utilized either as a fertilizer or in the production of calcium ammonium nitrate (CAN).

1.6 Industrial applications

All types of soil can be treated with nitro-phosphate, however it works best on soils with high pH or that are naturally alkaline. It is used during crop planting and the early stages of growth. Due to the fact that nitro-phosphate contains an equal amount of nitrogen (N) and phosphorous (P), nutrients are dispersed evenly across the area.

Because ammonium nitrate (NH_4NO_3) is present, it is occasionally also utilized in explosives. When ammonium nitrate makes up more than 70% of the product weight, it becomes unsafe to use.

1.7 Handling, storage & safety

1.7.1 Phosphate rock

Trucks and dumpers are used to move phosphate rock from the mine to the plant site. Large pieces of rock are reduced in size by passing through crushers before being stored in expansive yards. Conveyors are protected from dust using shed and bag filters. Additionally placed are magnetic separators for removing metallic contaminants.

1.7.2 Ammonia

Ammonia handling demands close attention. It reacts with body moisture to quickly dissolve in water and inflict serious damage. It should be used with the appropriate safety measures, such as safety equipment. Because ammonia corrodes metals like copper, zinc, and other metals, metal containers are not appropriate for storing ammonia. Ammonia is kept liquid by being stored in under pressure tanks, and pressurized tankers are used to carry it to the plant site.

1.7.3 Nitric acid

Because it is corrosive and produces severe skin burns, handling nitric acid calls for the use of chemical safety glasses, a face mask, and gloves. Nitric acid is stored in cool, dry, and properly ventilated containers after being carried to the plant site in cool, pressurized tankers.

1.7.4 Nitro-phosphate

Since ammonium nitrate is the fertilizer's primary source of nitrogen (N), nitro-phosphate (NP) contains a significant amount of it (> 50%). Ammonium nitrate works as an oxidizer when its weight-to-amount ratio exceeds 70%, at which point the product's explosion potential must be determined. Fertilizer's ammonium nitrate attracts moisture as well, therefore to avoid clumping or caking, it is packaged in watertight bags. Nitro-phosphate granules are transported to the farmers in 50 kg bags.

1.8 Shipping of product

If the test for ammonium nitrate is negative, the substance is safe to use and is shipped in bulk in special airtight containers.

NP fertilizer is examined for strong mechanical resilience and minimal caking tendency prior to long-distance bulk shipment. Granules in storage and transit don't break because of their high mechanical resistance. Fertilizer's low inclination to cake prevents it from forming a cake during long-term storage and transportation. To avoid caking and make fertilizer run freely, anticaking chemicals are also used.

The fertilizer collects moisture during storage and delivery because to its hygroscopic nature. Because of this, NP granules have created a hydrophobic covering to reduce moisture attraction and facilitate shipping. To stop dust development, de-dusting tools like bag filters should be employed at every stage of transit and storage.

1.9 Market assessment

There are three significant nitro-phosphate producers in Pakistan: Pak Arab Fertilizer Limited, Engro Fertilizer Limited, and Fatima Fertilizer Company Limited. The first NP manufacturer in Pakistan, Pak Arab Fertilizer, was established in 1973 and has an installed capacity of 304,000 MTPA. The need for food is rising along with the population, necessitating increased crop yields. Fertilizers are necessary for higher crop yields. Engro Fertilizer Limited (EFERT), having an installed capacity of 40,000 MTPA, began producing NP in 2005. Fatima Fertilizer Company Limited began producing NP in 2010 with an installed capacity of 360,000 MTPA in order to further meet demand.

Pakistan ceased importing nitro-phosphate (NP) in 2010 because domestic production could meet its needs. With time, as food demand rises, fertilizer with higher concentrations of macronutrients will be used to improve crop yields.

1.10 Future trends

Food consumption is rising due to the population's rapid increase, which calls for higher crop yields. Demand for fertilizers is growing every day as farmers strive for higher crop yields. The two main plant nutrients nitrogen (N) and phosphorous (P), which are needed for optimal plant development, roots, blooming, and fruit production, are combined to form nitro-phosphate. Russia is the world's leading producer of NP, whilst China is the top user.

Pakistan's NP demand is met by manufacturing that began after 2010. Demand for nitro-phosphate is rising as a result of the rapid rise of the population. Nitro-phosphate consumption increased from 651,000 MT in 2016 to 688,000 MT in 2017. As was the case previously, NP demand is rising steadily.

CHAPTER 2

INTRODUCTION TO PRETREATMENT

2.1 Introduction:

Phosphate ore is a necessary and unique raw material used in the production of phosphoric industrial goods. It is widely used in industries including agriculture, chemistry, food, pharmacy, etc. Up to 70 billion tonnes of phosphate are included in the world's reserves. The largest phosphate reserve, totaling 50 billion tonnes and making up 71.43% of the global supply, is in Morocco. With a share of 79%, China, Morocco, the United States, and Russia are the top producers of phosphate. But Pakistan has limited reserves of phosphate rock located in the north-east of the country. The largest ore occur in Hazara division. The prospecting deposits are located near the villages of Kakul, Mirpur, Lagarban, Tarnawai, Barkot and Havelian.

According to mineralization, there are four main categories of phosphate resources: igneous deposits, metamorphic deposits, sedimentary deposits, and biogenic deposits (guano accumulations).

In order to obtain the intermediate products of phosphoric acid and phosphorus, which may be utilized to manufacture different phosphate fertilizers and phosphates, the high-grade phosphate ore will undergo the wet process and the pyrogenic process, respectively.

Phosphate concentrate must meet the following criteria in order to be employed in the wet phosphoric process:

(1) a P_2O_5 grade greater than 30%

(2) a CaO/P_2O_5 ratio less than 1.6

(3) a MgO content less than 1%

2.2 Beneficiation Techniques for Phosphate Ores

The high-grade phosphate ores with a minimal level of impurities are, however, running out due to a growing population and phosphate consumption. Because they have a relatively low P_2O_5 content and typically contain a variety of gangue minerals, primarily quartz, mica, feldspar, dolomite, calcite, clays, and other materials, the majority of phosphate ores are not suitable for direct use in the acidulation process. As a result, the industry of phosphate beneficiation faces a significant difficulty, namely how to utilize these low-grade phosphate ores in an affordable and effective manner.

The beneficiation methods and reagents become particularly important to achieve the selective separation of phosphate minerals because low-grade phosphate ores must be processed to produce a certified phosphate concentrate. The appropriate beneficiation methods and reagents are introduced in accordance with the various compositions and textures of the run-of-mine:

- Flotation
- Attrition Scrubbing and Desliming
- Electrostatic Separation
- Magnetic Separation
- Gravity Separation
- Calcination
- Acid Leaching

2.2.1 Flotation

For the beneficiation of phosphate ore, particularly for low-grade sedimentary phosphate rocks, froth flotation is primarily used due to the high effectiveness in the removal of silicate and carbonate gangue particles. To extract the silicate gangues from phosphate ores, direct flotation using anionic surfactants or reverse flotation using cationic surfactants are typically used.

2.2.2 Attrition Scrubbing and Desliming

This method is frequently employed to remediate weathered phosphate ores that contain a sizable number of clays. In this instance, fine-grained clays can be removed to produce a coarse phosphate concentrate. In the Dianchi district of Yunnan, China, this process has been successfully used on high-grade weathered phosphate ores. A high-quality concentrate with a grade of P_2O_5 above 32% was obtained after the gangue clays that are dominant in sesquioxide ($Al_2O_3 + Fe_2O_3$) were removed from the weathered phosphate ores after they had been crushed and sieved.

2.2.3 Electrostatic Separation

By removing a particular amount of silica, grained phosphate minerals are always pre-concentrated via electrostatic separation. This strategy can lower the cost of chemical reagents, water use, and energy used in grinding. The viability of using a rotary triboelectrostatic separator in a dry beneficiation process for Florida phosphate ores was examined by Sobhy and Tao. According to their findings, a concentrate with a P2O5 content of 30% was collected, and both the acid-insoluble rejection and recovery rates exceeded 85%.

2.2.4 Magnetic Separation

The beneficiation of igneous apatite deposits, which are rich in ferriferous minerals like magnetite or titanomagnetite, uses magnetic separation successfully. A sequential low-intensity magnetic separation, desliming, flotation, and high intensity magnetic separation method can be used to beneficiate apatite from the "Barreiro" complex carbonatite phosphate ore in Brazil. Meanwhile, it has been established that magnetic separation may be used to treat coarse-grained sedimentary phosphate ore in order to remove magnetic gangues.

2.2.5 Gravity Separation

According to the density difference between precious minerals and gangue minerals, gravity separation is performed. It is typically challenging to recover phosphate minerals that are dispersed into fine grains because of the similar density of apatite and gangue minerals. However, it was discovered that the sedimentary phosphate rock's coarse phosphate minerals with a strip structure could be beneficiated successfully by removing tailings using gravity separation. In this instance, the energy needed for grinding is significantly decreased.

2.2.6 Calcination

Worldwide, calcination produces more than 10% of the commercial phosphates. At a temperature of around 950 °C, calcareous minerals are broken down into calcium oxide and magnesium oxide, and carbon dioxide is emitted during the process. After that, NH_4Cl or NH_4NO_3 solution with a 5% concentration is used to quench the calcined phosphate rocks.

After being classified using a hydrocyclone, the fine-grained $Ca(OH)_2$ and $Mg(OH)_2$ are discarded as the slime phase, and the coarser high-grade concentrate is successfully separated. It is also a successful method for upgrading phosphate ores that contain organic materials at a temperature of 650–750 °C.

2.2.7 Acid Leaching

By using acid leaching, the calcareous phosphate ores can also be improved. While the phosphate minerals remain in the leached residues throughout this process, the carbonate gangue minerals choose to dissolve in the diluted acid solution. The separation of phosphate minerals from carbonate gangue minerals is thus accomplished. When using typical mineral acids, P_2O_5 is frequently dissolved and P_2O_5 recovery is lost, but organic acids have great leaching selectivity. As a result, the leaching process prefers the use of organic acids. Lactic acid, formic acid, and succinic acid are the most frequently utilized organic acids.

From the discussion above, beneficiation techniques and their applications for various phosphate ores are summarized in Table below:

Table 5 Beneficiation techniques and their applications for various phosphate ores

Beneficiation Techniques	Phosphate Rock Types	Applications
Flotation	Siliceous phosphate rock Calcareous phosphate rock Calcareous-siliceous phosphate rock	Widely applied in the beneficiation of various phosphate rocks on a large scale, especially for refractory sedimentary phosphate ore
Attrition Scrubbing-desliming	Weathered phosphate ores	Used for discarding clay minerals and eliminating the detrimental effect of slimes on the subsequent flotation process
Gravity separation	Sedimentary phosphate rock characterized by a strip texture	Discarding gangues to achieve the preconcentration of
Magnetic separation	Phosphate ores containing magnetic gangues	Phosphate minerals
Calcination	Calcareous phosphate rock	Acceptable for areas that have low cost energy and limited water resources
Electrostatic separation	Coarse gained siliceous phosphate rock	Mostly are experimental studies in laboratory scale

CHAPTER 3

MANUFACTURING PROCESS

3.1 Pretreatment

3.1.1 Direct and Reverse Froth Flotation

Out of all these techniques, froth flotation is selected for the beneficiation of rock phosphate depending on the mineral type and mineral granularity. It has been successfully applied to both sedimentary and igneous phosphates with siliceous gangue. But processing of sedimentary rock phosphate containing calcareous impurities such as calcite or dolomite in addition to siliceous gangue has numerous problems. Two stage direct and reverse flotation are used with depressants and reagents for the removal of carbonates and silicates. Column flotation is also used during the process to increase the efficiency.

To extract the silicate gangues from phosphate ores, direct flotation using anionic surfactants or reverse flotation using cationic surfactants are typically used. The pulp pH was brought down to roughly 9.5 during the direct flotation process by adding soda ash, and the right quantity of water glass was needed at the same time to depress the float of silicate minerals. After that, anionic surfactants were used to help the phosphate minerals float. For the reverse flotation method, cationic surfactants were used to float silica at a weakly acidic or neutral pH, and the underflow product was used to extract the phosphate minerals—mostly apatite—from it. The higher negatively charged surfaces of silica as opposed to apatite are likely the cause of the greater adsorption of cationic surfactants on silica. Without the use of a phosphate depressant, reverse anionic flotation at a pH of about 4.5 may remove carbonate gangues with great separation efficiency.

3.1.2 Properties of Flotation Reagents

The selective separation of phosphate minerals and gangues is significantly impacted by a flotation reagent, particularly depressants and collectors. Due to the hydrophilic coating that forms after the depressant adsorption, a certain mineral's surface becomes hydrophilic throughout the flotation process.

The floated mineral's surface is being directed by collector molecules, increasing its hydrophobicity. For instance, it is frequently necessary to add a depressant for gangue mineral in the straight flotation of phosphate ore. The phosphate minerals can efficiently interact with collectors following the depression of gangues and grow more gathered by adhering to the rising bubbles.

3.1.3 Depressants

3.1.3.1 Depressants of Silicate Minerals

Sodium silicate is an effective depressant in the direct flotation of apatite from siliceous phosphate ore using fatty acids. Compared to the infrared spectra of quartz in the absence and presence of sodium silicate at pH 7, the adsorption of dominant hydrolysis species $Si(OH)_4$. The quartz depression mechanism was elucidated through the ligand exchange model, where pairs of electrons were shared by sites of the surface and species of silicate. Generally, the modulus and dosage of sodium silicate have a great impact on the depression of quartz occurred in flotation process

3.1.3.2 Depressants of Phosphate Minerals

Because the floating behavior rarely happened at pH below 4.5 in an anionic flotation of apatite without the addition of any other phosphate depressant, the depression of apatite can be accomplished by altering the pH of pulp. According to the solution equilibria of dissolved apatite, it is obvious that the species of $Ca^{2+,}$ $CaH_2PO^{4+,}$ and H_2PO_4 are dominant at pH values below 4.5 and that their concentration rises as pH decreases.

3.1.4 Collectors

3.1.4.1 Anionic Collectors

In phosphate flotation, long chain fatty acids are frequently utilized as anionic collectors, particularly for sodium oleate. Using atomic force microscopy, the adsorption characteristics of potassium. On the surface of the apatite crystal, oleate and the precipitation of Ca2+ ions were seen. On the apatite surface, calcium dioleate is thought to have formed and precipitated in the form of agglomerates. Agglomerates made the surface of the apatite rougher and more heterogeneous, which affected how well apatite floated.

3.1.4.2 Cationic Collectors

In the practical flotation of phosphate ores, cationic collectors are dominantly used for the removal of silicate minerals. The cationic collector's advantage is that it can separate precious minerals from gangues at a low temperature with minimal reagent use. However, run-of-mine ore contains slimes that are typically toxic to amine collectors. Additionally, when utilising an amine as the collector during the flotation process, it is challenging to defoam due to the high foam viscosity. Because of this, cationic collectors work well for flotation of coarse-grained ores but perform poorly for separating fine-grained minerals.

3.2 Nitrophos Fertilizer

Nitro-phosphate is manufactured commercially by two processes:

1. ODDA Process

2. Mixed Acid Process

3.2.1 ODDA process

This process was invented by Erling Johnson in 1928. It is called as “Odda” because it was invented by Johnson in the town of Odda, Norway. This process was commercially used for the first time in 1938 by Norsk Hydro Company (now Yara International) for the production of NP fertilizer. Then it was further used by BASF after some modifications.

3.2.1.1 Raw materials

1. Phosphate rock

2. Nitric acid

3. Ammonia gas

3.2.1.2 Process description

<u>Dissolving reactor</u>

The process starts with the dissolution of phosphate rock in 60% nitric acid. This reaction is being carried out in a stainless-steel vessel, equipped with agitator, at a temperature of 60-70oC by the following reaction:

$Ca_3(PO_4)_2 + 6HNO_3 \rightarrow Ca(NO_3)_2 + 2H_3PO_4 + 22.25$ kcal

The rate of dissolution depends on:

1. Excess of nitric acid.

2. Surface area of phosphate rock particles.

3. Degree of agitation.

Foaming occurs in the reactor due to greater CO_2 content and is reduced by effective agitation or by adding anti-foaming agents like glycols, stearates, alcohols, etc. Urea is also used, amount

required would be 0.1-0.3% of rock feed, to suppress the emissions of NO_x and HF.

Sand removal

Sand particles are removed by washing slurry with water. Suspended solids (quartz) and inert

diluents of nutrients are removed in centrifuge.

Crystallization unit

Slurry of Calcium nitrate and phosphoric acid move towards crystallizer unit, which consists of

several batch crystallizers operated continuously. Aqueous solution of ammonia (25%) from

Higher water solubility of P_2O_5 in final product enables effective use of nutrients by the plants.

Trend of P_2O_5 water solubility with temperature inside the crystallizer is shown in Figure 3-1

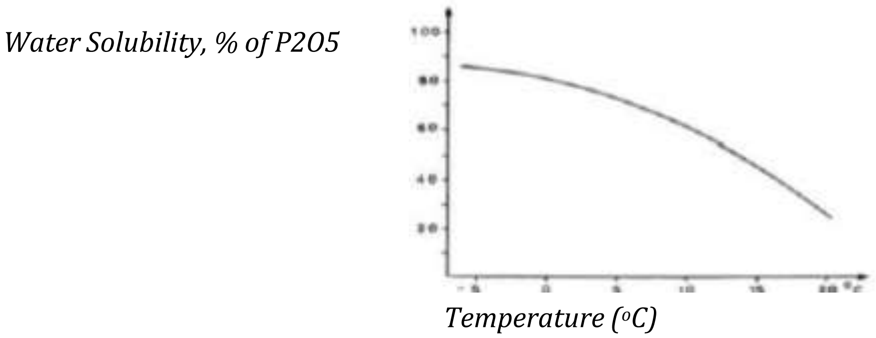

Figure 1 Effect of Crystallization temperature on P2O5 water solubility

CN crystals filter

After the formation of crystals, solution is passed through centrifuge to separate crystals from acid liquor. Calcium nitrate tetra hydrate crystals are separated and utilized either as a fertilizer or in the formation of calcium ammonium nitrate (CAN).

Neutralization reactor

Nitro-phosphoric (NP) acid is further neutralized with gaseous ammonia (NH3) in a pressurized reactor at pressure of 1.5-2.5 bar and temperature of 150-180oC. Heat of reaction released concentrates the solution to 75% by removing most of the water and ammonia.

$H_3PO_4 + 2NH_3 \rightarrow (NH_4)_2HPO_4 + 66.67\ \text{kcal}$

Evaporation & prilling

NP solution is discharged to evaporators under vacuum. Evaporator is operated at 175oC to concentrate final product to 99.9%. Pure nitro-phosphate melt is passed towards prilling tower. In prilling tower air is passed from the bottom whereas, melt enters the tower from the top.

Coating & packaging

Solid prills are removed from the bottom of the tower with scrapper at 95$_{o}$C. Prills are then cooled and conveyed to coating drum. Final coated NP prills are conveyed to packaging plant and are packaged in 50 kg bags. For shipping, air tight vessels are used to restrict moisture away from the product.

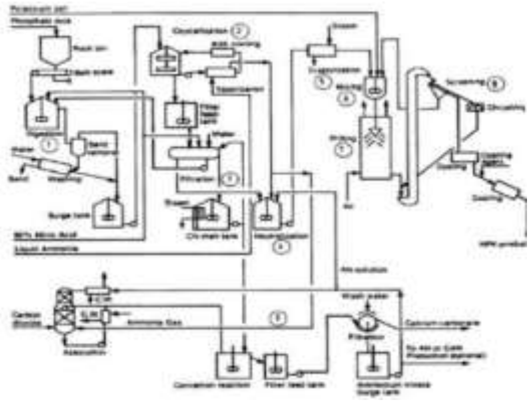

Figure 2 Process flow diagram of ODDA process

3.2.2 Mixed acid process

3.2.2.1 Raw materials

1. Phosphate rock

2. Nitric acid

3. Ammonia gas

4. Phosphoric acid

5. Sulfuric acid

3.2.2.2 Process description

<u>Digestion reactor</u>

Phosphate rock is digested by nitric acid in the first reactor at the temperature of 60-70$_{o}$C by the following reaction:

$$Ca_3(PO_4)_2 + 6HNO_3 \rightarrow 3Ca(NO_3)_2 + 2H_3PO_4 + 22.25 \text{ kcal}$$

Acid gases like nitrous oxides and compounds of fluorine are formed during the digestion reaction, which are removed by scrubber. Calcium nitrate is not removed from the reactor, it stays in the reactor to provide two nutrients, calcium and nitrogen.

Second reactor

Neutralization of acidic slurry is done in second reactor by using ammonia (NH_3). Sulfuric acid and phosphoric acid are also added to the reactor to increase P_2O_5 content in the product, increase P_2O_5 water solubility and remove calcium from the reactor as gypsum ($CaSO_4$).

$$HNO_3 + NH_3 \rightarrow NH_4NO_3 + 22.27 \text{ kcal}$$

$$H_3PO_4 + 2NH_3 \rightarrow (NH_4)_2HPO_4 + 33.52 \text{ kcal}$$

$$2H_3PO_4 + Ca(NO_3)_2 + 4NH_3 \rightarrow CaHPO_4 + (NH_4)_2HPO_4 + 2NH_4NO_3$$

$$Ca(NO)_3 + H_2SO_4 \rightarrow 2HNO_3 + CaSO_4 + 9.37 \text{ kcal}$$

Third reactor

Further neutralization of overflow of second reactor by ammonia takes place in this reactor. pH of the solution reaches 5-6 to avoid formation of HCl in the reactor and several by-products are formed like di-calcium phosphate, calcium sulfate, calcium hypophosphate, etc. Buffer tank & spherodizer Buffer tank acts as a feed tank for spherodizer. Water content in the slurry is 5-30% and temperature of 100-140$_{o}$C. Spherodizer is the combination of drying and granulating.

Evaporation and packaging

Product granules enters the evaporator, where steam enters, remove moisture and concentrate the product. Final product reach packaging plant for bulk loading or bagging.

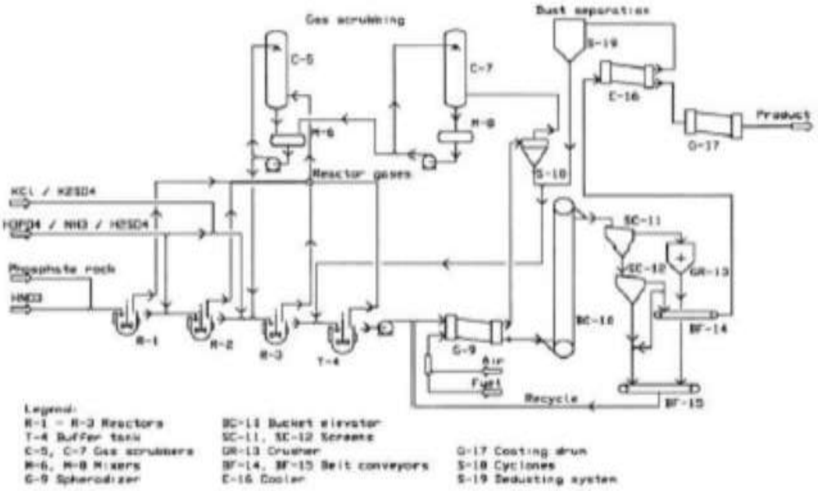

Figure 3: Process flow diagram of mixed acid process

3.3 Process comparison

Table 6 Production methods comparison

Process	ODDA Process	Mixed Acid Process
Raw materials	Phosphate rock Nitric acid Ammonia gas	Phosphate rock Nitric acid Sulfuric acid Phosphoric acid Ammonia gas
By-products	Ammonium nitrate Calcium ammonium nitrate Calcium nitrate	Calcium sulfate Calcium nitrate
Separation of CNTH crystals	This process separates the CNTH crystals, after digestion, for the production of AN & CAN.	This process does not separate CNTH crystals, to utilize micronutrient(calcium).
By-products usage/disposal	In this process, by-products are upgraded to commercial products like CAN, AN, etc.	This process does not involves utilization of by-products thus, facing difficulty in the disposal of by-products like phosphor-gypsum, etc.

Waste disposal	This process involves no wastes except NO_x, which is controlled by adding urea or scrubbing.	This process involves solid waste like phosphor-gypsum, which is difficult to dispose. It also involves reactor gases like fluorides, NO_x, SO_x, etc.
CO_2 emissions	This process utilizes CO_2 emissions from ammonia plant in the formation of ammonium carbonate for CAN plant.	No CO_2 emissions.
H_2S emissions	No H_2S emissions	Higher H_2S emissions inside the reactors due to the presence of sulfuric acid.
Simplicity of process	Oldest and simplest process	Complex process

3.4 Process selection

Selected process for the manufacture of nitro-phosphate is ODDA process because:

1. It’s the oldest, simplest and widely used process all over the world.

2. By-products are upgraded to commercial and useful products like AN and CAN.

3. Process is sulfur independent.

4. No solid waste and disposal difficulty.

5. Product quality is fine due to removal of impurities at every step

6. CO_2 emissions from ammonia plant are used in the absorption column for the formation of ammonium carbonate in CAN plant.

3.5 Process description

3.5.1 Raw materials

<u>Phosphate rock</u>

Phosphate rock is imported from Morocco, Jordan and Algeria because phosphate rock in Pakistan is not of good quality because it has P_2O_5 content of less than required (32%).

Ammonia and nitric acid

Ammonia is used in large amounts for the production of nitro-phosphate and nitric acid as the main reactant, neutralizer and cooling medium. Nitric acid is the main raw material for the production of NP.

3.5.2 Phosphate rock digestion

The process starts with the dissolution of phosphate rock in 60% nitric acid. This reaction is being carried out in two stainless steel vessels, equipped with agitator, at a temperature of 70oC by the following reaction:

Ca3(PO4)2 + 6HNO3 → Ca(NO3)2 + 2H3PO4 + 22.25 kcal

The rate of dissolution depends on:

1. Excess of nitric acid.

2. External surface of phosphate rock.

3. Degree of agitation.

Foaming occurs in the reactor due to greater CO2 content and is reduced by effective agitation or by adding anti-foaming agents like glycols, stearates, alcohols, etc. Urea is also used, amount required would be 0.1-0.3% of phosphate rock feed, to suppress the emissions of NOx and HF. Rock particle size of less than 8 mesh requires less than one hour for dissolution. Large lumps require pre-grinding for fast reaction.

3.5.3 Removal of silica

Silica particles are removed in centrifuge by washing slurry with water. Suspended solids (quartz)and inert diluents of nutrients are removed.

3.5.4 Crystallization unit

Slurry of Calcium nitrate and phosphoric acid move towards crystallizer unit, which consists of several batch crystallizers operated continuously. Refrigeration system is installed for supply of coolant. Aqueous ammonia (25%) from refrigeration system flows through each crystallizer to cool the slurry to -5oC. As the temperature decreases, water solubility of P2O5 in final product increases. Higher water solubility of P2O5 in final product enables effective use of nutrients by the plants.

3.5.5 CN crystals filter

After the formation of crystals of size 70 µm, solution is passed through centrifuge to separate crystals from acid liquor. Calcium nitrate tetra hydrate crystals are separated and move towards CAN plant.

3.5.6 Neutralization reactor

Nitro-phosphoric (NP) acid is further neutralized with gaseous ammonia (NH3) in a pressurized reactor at pressure of 1.5-2.5 bar and temperature of 120oC. Heat of reaction released concentrates the solution to 75% by removing most of the water and ammonia.

$$H_3PO_4 + 2NH_3 \rightarrow NH_4H_2PO_4 + 66.67\ \text{kcal}$$

3.5.7 Evaporation & prilling

NP solution is discharged to forced circulation evaporators under vacuum. Evaporator is operated at 175oC to concentrate final product to 99%. Pure nitro-phosphate melt is passed through prilling tower. In prilling tower air is passed from the bottom whereas, melt enters the tower from the top to form solid prills.

3.5.8 Coating & packaging

Solid prills are removed from the bottom of the tower with scrapper at 95oC. Prills are then cooled and conveyed to coating drum. Final coated NP and CAN prills are conveyed to packaging plant and are packaged in 50 kg bags. For shipping, air tight vessels are used to restrict moisture away from the product.

3.6 Capacity selection

Demand of nitro-phosphate fertilizer is increasing day by day due to increasing demand of food with population growth. After analyzing the consumption data of last 10 years, we concluded that the consumption is increasing with growth rate of 9.6%. Current consumption of NP in Pakistan is 688,000 MTPA and after five years it will reach 992,040 MTPA. The difference would be then 304,040 MTPA. So we selected the capacity of 300,000 MTPA to fulfill our country's demand.

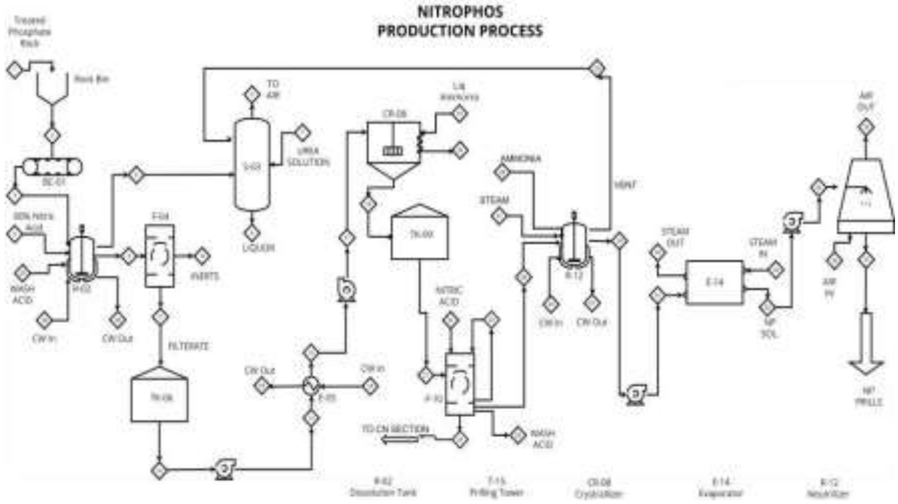

Figure 4 Flowsheet of ODDA process

CHAPTER 4

MATERIAL AND ENERGY BALANCE

4.1 Material balance

A balance on conserved quantity (mass, energy, momentum, etc.) in a system (single unit, multiple units or entire process) may be written in the following general way,

Input + Generation − Output − Consumption = Accumulation.

Steady state process is assumed hence, accumulation becomes zero.

4.1.1 Plant capacity

1,500 MTPD

4.1.2 Material Balance over Pre-Treatment Plant:

(Basis: 1 hour operation)

(Unit: kg)

4.1.2.1 WET GRINDER

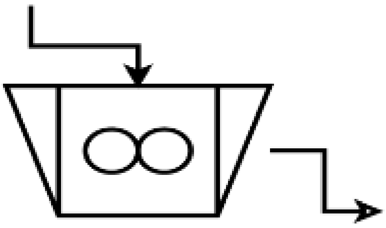

Figure 5 Wet Grinder

Table 7 Mass balance of Wet Grinder

MASS IN		MASS OUT	
BALL MILL IN		BALL MILL OUT	
RP	64942	RP	64942
WATER	25000	WATER	25000
	89942		89942

4.1.2.2 CONE CLASSIFIER

Figure 6 Cone Classifier

Table 8 Mass Balance of Cone Classifier

MASS IN		MASS OUT			
CONE CLASSIFIER IN		ROUGHER STEREAM		FINES STREAM	
RP	64942	RP	58447.8	RP	6494.2
WATER	25000	WATER	25000	WATER	0
	89942		83447.8		6494.2

4.1.2.3 ROUGHER CONDITIONING

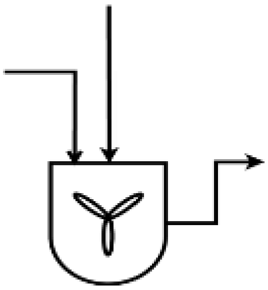

Figure 7 Rougher Conditioning

Table 9 Mass balance of Rougher Conditioning

MASS IN				MASS OUT	
ROUGHER STREAM		REAGENTS DOSING 1		ROUGHER PULP	
RP	58447.8	OLEIC ACID	116.89	RP	58447.8
WATER	25000	POLYPROPYLENE GLYCOL	3.50	WATER	25000
		SODIUM SILICATE	17.53	OLEIC ACID	116.89
				POLYPROPYLENE GLYCOL	3.50
				SODIUM SILICATE	17.53
	83447.8		137.93		83585.73

4.1.2.4 Rougher Floatation

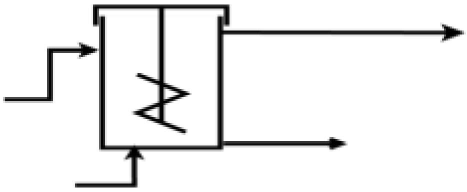

Figure 8 Rougher Floatation

Table 10 Mass balance of Rougher Floatation

MASS IN		MASS OUT			
ROUGHER PULP		ROUGHER CONCENTRATE		ROUGHER TAILINGS	
RP	58447.8	RP	43270.85	RP	15176.94
WATER	25000	WATER	26301.51	WATER	61370.19
OLEIC ACID	116.8956	OLEIC ACID	116.89		76547.13
POLYPROPYLENE GLYCOL	3.506868	POLYPROPYLENE GLYCOL	3.50		
SODIUM SILICATE	17.53434	SODIUM SILICATE	17.53		
	83585.73		69710.30		
ÀDDITIONAL WATER					
WATER	62671.7				

4.1.2.5 WET REGRINDING

Table 11 Mass balance of Wet Regrinder

MASS IN		MASS OUT	
BALL MILL IN		BALL MILL OUT	
RP	43270.8546	RP	43270.8546
WATER	26301.51	WATER	26301.51
OLEIC ACID	116.8956	OLEIC ACID	116.8956
POLYPROPYLENE GLYCOL	3.506868	POLYPROPYLENE GLYCOL	3.506868
SODIUM SILICATE	17.53434	SODIUM SILICATE	17.53434
	69710.30141		69710.30141

4.1.2.6 DIRECT CLEANER CONDITIONING

Table 12 Mass balance of Cleaner Conditioning Tank

MASS IN				MASS OUT	
ROUGHER CONCENTRATE		REAGENTS DOSING 1		ROUGHER PULP	
RP	43270.85	OLEIC ACID	43.27	RP	43270.85
WATER	26301.51	POLYPROPYLENE GLYCOL	1.29	WATER	43270.854
OLEIC ACID	116.89	SODIUM SILICATE	6.49	OLEIC ACID	160.16
POLYPROPYLENE GLYCOL	3.50			POLYPROPYLENE GLYCOL	4.804
SODIUM SILICATE	17.53			SODIUM SILICATE	24.02
	69710.30		51.05		86730.70
ADDITIONAL WATER					
WATER	16969.34				

4.1.2.7 REVERSE FLOATATION

Table 13 Mass balance of Reverse Floatation

MASS IN		MASS OUT			
REVERSE PULP		REVERSE CONCENTRATE		REVERSE TAILINGS	
RP	42485.05	RP	38419.69	RP	4065.37
WATER	18207.88	WATER	12745.52	WATER	5462.36
FATTY AMINES	4.24			FATTY AMINES	4.25
FUEL OIL	2.12			FUEL OIL	2.12
ACETIC ACID	2.97			ACETIC ACID	2.97
	60702.28		51165.20		9537.08

4.1.2.8 CENTRIFUGATION

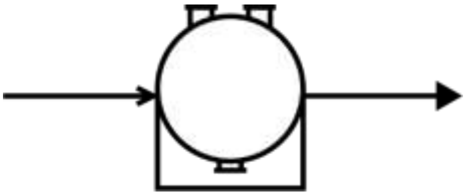

Figure 9 Centrifugation

Table 14 Mass balance of Centrifugation

MASS IN		MASS OUT			
REVERSE CONCENTRATE		TREATED RP TO NP		REMOVED WATER	
RP	38419.68	RP	38419.69	WATER	12007.52
WATER	12745.51	WATER	738.00		
	51165.20		39157.69		12007.52

4.1.2.9 FINES CONDITIONING

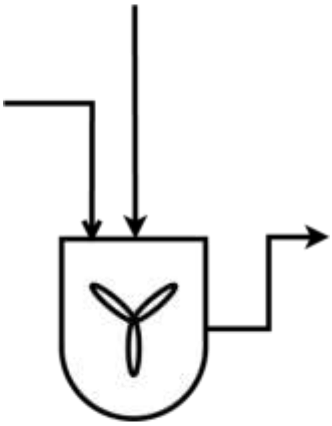

Figure 10 Fines Conditioning Tank

Table 15 Mass balance of Fines Conditioning Tank

MASS IN				MASS OUT	
FINES STREAM		REAGENTS DOSING 2		FINES PULP	
RP	6494.2	OLEIC ACID	12.98	RP	6494.2
WATER	0	POLYPROPYLENE GLYCOL	0.38	WATER	2783.22
		SODIUM SILICATE	1.94	OLEIC ACID	12.98
				POLYPROPYLENE GLYCOL	0.38
				SODIUM SILICATE	1.94
	6494.2		15.32		9292.75
ADDITIONAL WATER					
WATER	2783.22				

4.1.2.10 DIRECT CLEANER FLOATATION

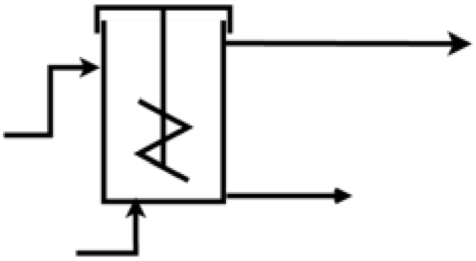

Figure 11 Direct Cleaner Floatation

Table 16 Mass balance of Direct Cleaner Floatation

MASS IN		MASS OUT			
FINES PULP		FINES CONCENTRATE		FINES TAILINGS	
RP	6494.20	RP	4130.31	RP	2363.89
WATER	2783.23	WATER	834.97	WATER	1948.26
OLEIC ACID	12.99	OLEIC ACID	13.69		
POLYPROPYLENE GLYCOL	0.39	POLYPROPYLENE GLYCOL	0.69		
SODIUM SILICATE	1.95	SODIUM SILICATE	1.95		
	9292.75		4981.61		4312.15

4.1.3 Material Balance On Nitrophos Plant:

4.1.3.1 Main reactions

$$Ca_3(PO_4)_2 + 6HNO_3 \rightarrow 3Ca(NO_3)_2 + 2H_3PO_4$$

$$H_3PO_4 + 2NH_3 \rightarrow NH_4H_2PO_4$$

Conversions = 99%

4.1.3.2 Material balance on digester

$$Ca3\ (PO4\ )2 + 6HNO3 \rightarrow 3Ca(NO3\ )2 + 2H3PO4$$

$$CaCO3 + 2HNO3 \rightarrow Ca(NO3\ )2 + H2O + CO2$$

$$CaF2 + 2HNO3 \rightarrow Ca(NO3\ )2 + 2HF$$

$$Ca(NO3\ )2 + SiO2 + 6HF \rightarrow CaSiF6 + 2HNO3 + 2H2O$$

<u>Theoretical nitric acid required</u>

$6 * 63 \text{ x } (23757)/310 + 2 * 63 \text{ x } (4930)/100 + 2 * 63\ x\ (2968)/78 - 2 * 63\ x\ (1822)/60$

Theoretical HNO3 = 38689 kg/hr

Table 17 Overall balance of Digester

Components	Material in			Material out	
	Rock Phosphate 3S	Nitric Acid 4S	Wash Acid 42S	Solution to decanter 5S	Off gases (to vent) 6S
	kg/hr.				
$Ca_3(PO_4)_2$	23757				
$CaCO_3$	4930				
$CaSO_4$	983			1007	
CaF_2	2968				
HNO_3		23404	19583.8		383
Inerts+SiO2	1070				
Reactive SiO2	1822				
Fe_2O_3	360.89				
$A_{l2}O_3$	226.78				
K_2O_3	20.03				
Na_2O_3	323.8				
MgO	202.7				
H_2O	738	17656	9028	27750	815
$CaSiO_3$	1244				
Me O	512			1007	
$Ca(NO_3)_2$				52899	
$Mg(NO_3)$				746	
Fe (NO3)2				1094	
Al (NO3)2				667	
KNO3				43	
NaNO3				888	
H_3PO_4				16637	133
CO_2					2159

CHAPTER 5

EQUIPMENT DESIGN

5.1 Floatation Tanks

5.1.1 Rougher Floatation Tank

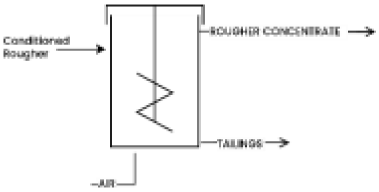

Figure 12 Rougher Floatation Tank

Residence time = t = 1 hr = 60 min

Mass flow rate of feed = 83585 kg/hr

weighted density = 0.224 kg/cm^3

Volume of feed = V_0 = 371 m^3

with 10% air holdup = 408 m^3

Diameter of Tank = D = 4.46 m

<u>Impeller Design</u>

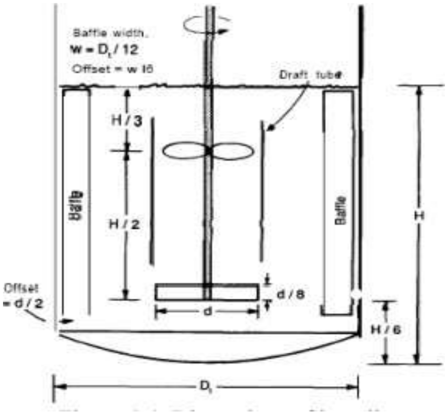

Figure 13 Impeller Diagram

$D_a / D_t = 1/3$

$H / D_t = 1.22$

$J / D_t = 1/12$

$E / D_t = 1/3$

$W / D_a = 1/5$

$L / D_a = ¼$

Tip Speed = $T_S = \pi D_a$ * rpm of impeller

Where:

D_a = Diameter of impeller

D_t = Tank diameter

H = Depth of liquid in tank

J = Width of baffles

E = Hight of impeller above vessel flow

W = Impeller width

L = Length of impeller blade

Table 18 Specification Sheet Rougher Floatation Tank

Specification Sheet Tank	
Length	6.55 m
Diameter	4.46 m
Volume per tank	103 m^3
No. Of Tanks	4
Residence time	60 min

Table 19 Specification Sheet Rougher Floatation Tank Impeller

Specification Sheet Impeller	
Impeller Speed(rpm)	1100
Agitator Diameter Da (m)	1.48
Depth of liquid in tank H (m)	3.65
Width of baffles J (m)	0.37
Height of impeller above vessel E (m)	1.49
Impeller width W (m)	0.29
Length of impeller blade L (m)	0.37
Tip Speed Ts (m/min)	5137

5.1.2 Cleaner Floatation Tank

Table 20 Specification Sheet Cleaner Floatation Tank

Specification Sheet Tank	
Length	6.55 m
Diameter	4.82 m
Volume per tank	120 m^3
No. Of Tanks	4
Residence time	60 min

Table 21 Specification Sheet Cleaner Floatation Tank Impeller

Specification Sheet Impeller	
Impeller Speed(rpm)	1000
Agitator Diameter Da (m)	1.61
Depth of liquid in tank H (m)	3.95
Width of baffles J (m)	0.40
Height of impeller above vessel E(m)	1.61
Impeller width W (m)	0.32
Length of impeller blade L (m)	0.40
Tip Speed Ts (m/min)	5047

5.1.3 Reverse Floatation Tank

Table 22 Specification Reverse Floatation Tank

Specification Sheet Tank	
Length	6.55 m
Diameter	4.43 m
Volume per tank	100 m^3
No. Of Tanks	3
Residence time	60 min

5.1.4 Column Floatation Tank

Table 23 Specification Sheet Column Floatation Tank

Specification Sheet Tank	
Length	6.55 m
Diameter	3.23 m
Volume of tank	54 m^3
No. Of Tanks	1
Residence time	60 min

5.2 Reactor

5.2.1 Selection of reactor

Selected reactor for dissolution of phosphate rock is “Continuous Stirred Tank Reactor (CSTR)”

The main reasons supporting the logical selection of CSTR are,

1. Large scale application
2. Economical (reduced operation cost)
3. Control of product quality

5.2.2 Design of reactor (R-02)

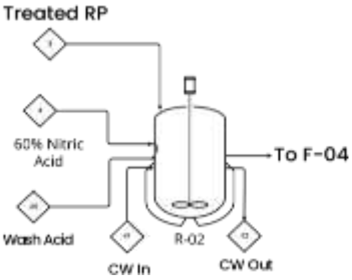

Figure 14 Digestion Reactor

Design equation of CSTR

$$V = \frac{F_{A0}X}{-r_A}$$

Main reaction

$$Ca_3(PO_4)_2 + 6HNO_3 \rightarrow 3Ca(NO_3)_2 + 2H_3PO_4$$

Conversion = 99%

Let A = Calcium phosphate

B = Nitric acid

F_{A0} = 76.6 kmol/hr.

F_{B0} = 682.3 kmol/hr.

V_{A0}= 7.57m^3/hr

V_{B0}= 31.38m^3/hr

$V_0 = V_{A0} + V_{B0} = 38.94\ m^3/hr$

$C_{A0} = \frac{FA0}{V0} = 1.97 kmol/m^3$

$C_{B0} = \frac{FB0}{V0} = 17.52 kmol/m^3$

For irreversible reactions,

$-r_A = k_A C_A^{\alpha} C_B^{\beta}$

As HNO_3 is in excess so $C_B = C_{B0}$

$-r_A = k_A C_A^{\alpha} C_{B0}^{\beta}$

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

Where $K' = k_A C_{B0}^{\beta}$

<u>Developing rate law</u>

Mole balance equation: $\frac{dN_A}{dt} = -r_A V$

Rate law: $-r_A = k' C_A^{\alpha}$

$$C_A = \frac{N_A}{V_0}$$

Combining the equations

$$\frac{dN_A}{dt} = -k' C_A^{\alpha} V_0$$

$$\frac{d(\frac{N_A}{V_0})}{dt} = -k' C_A^{\alpha}$$

$$-\frac{dC_A}{dt} = k' C_A^{\alpha}$$

Taking ln on both sides,

$$\ln(-\frac{dC_A}{dt}) = \ln(k') + \alpha \ln(C_A)$$

$$\left(\frac{dCA}{dt}\right)t0 \quad = \frac{-3C_{A0}+4C_{A1}+C_{A2}}{2\Delta t} = -491.15 \text{ x } 10^{-3}$$

$$\left(\frac{dCA}{dt}\right)t1 = \frac{C_{A2}-C_{A0}}{2\Delta t} = -343.25 \text{ x } 10^{-3}$$

$$\left(\frac{dCA}{dt}\right)t2 = \frac{C_{A3}-C_{A1}}{2\Delta t} = -219.30 \text{ x } 10^{-3}$$

$$\left(\frac{dCA}{dt}\right)t3 = \frac{C_{A4}-C_{A2}}{2\Delta t} = -136.70\text{x } 10^{-3}$$

$$\left(\frac{dCA}{dt}\right)t4 = \frac{C_{A5}-C_{A3}}{2\Delta t} = -88.80 \text{ x } 10^{-3}$$

$$\left(\frac{dCA}{dt}\right)t5 = \frac{C_{A3}-4C_{A4}+3C_{A5}}{2\Delta t} = -58.20 \text{ x } 10^{-3}$$

Order of reaction = slope of graph = α = 1.04 = 1 Ln(k’) = y-intercept of graph = -1.495

K′ = 0.22min^{-1}

C_{B0} =17.52kmol/m

K = $\frac{k'}{CB0}$ = 0.013dm^3/mol.min

Rate of reaction = $-r_A$ = 0.013 C_AC_B

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

$C_B = C_{A0}(\Theta_B - 6X)$

Θ_B = $^{C_{B0}}/_{C_{A0}}$ = $^{21.74}/_{1.97}$ = 8.91

Rate law can be written as:

$-r_A = C_{A0}(1\text{-}X) \times C_{A0}(\Theta_B - 6X)$

$-r_A$ = 1.97 x (1-0.99) x 1.97 x (8.91 – (6 x 0.99)) = 0.12 mol-dm^{-3}-min^{-1}

Volume of reactor = $^{F_{A0}X}/_{-r_A}$ = 11.0 m^3

Space time = $^{V}/_{V_0}$ = $^{658}/_{38.94}$ = 0.28 hr.

Optimum L/D ratio = 1.4, L = 1.4D

Volume of reactor = V =$\pi D^2L/4$= $\pi D^2(1.4D)/4$

Diameter of reactor = D = 2.15 m

Length of reactor = L = 1.4D = 3.0 m

5.2.3 Design of cooling jacket

Following are the cooling arrangements for continuous stirred tank reactors:

1. Jackets
2. Internal coils

5. External coils

Selection of Jacket

In terms of control, effectiveness, and product quality, a jacket offers the best way to heat and cool a process vessel.

There are now three primary categories of jackets.

1. Spiral baffle Jacket
2. Half pipe coil Jacket
3. Dimple jacket

Jacket selection

Factor which need to be considered when selecting the type of jacket use are listed below: [13]

1. Cost in the terms of cost the design can be ranked, form cheapest to cost expensive

    - Simple no baffles

    - Agitation nozzles

    - Dimple Jacket

    - Half-pipe jacket

2. Heat transfer rate required; select a spirally baffled or half-pipe jacket if high rate are required.

3. Pressure as a rough guide, the pressure rating of the designs can be taken as:

- Jackets, up to 10bar.
- Half-pipe up to 40bar.
- Dimple jacket up to 70bar.

So, dimple jackets would be used for high pressure.

Dimple jacket is less expensive and give low pressure drop than spiral and half-pipe coil jacket. So the selected jacket is Dimple baffle jacket.

Cooling Jacket Calculations

$Q = UA\Delta t$

$U_D = 350\ W/m^2.{}^0C$

Assuming 80% area of CSTR is covered with jacket

$A_j = \pi D^2/4 + \pi(D)(L)(0.8)$

$A_j = 20m^2$

$Q = 1.12x10^7\ kJ/hr$

For jacket

$h_o\ D_t / k = 0.85(\rho x D^2/\mu)^{0.33}(C\mu/k)^{0.66}(\mu/\mu s)^{0.14}$

Assume,

$(\mu/\mu s) = 1$

$h_o = 425\ W/m^2\ {}^oC$

Tank side

$hD/\lambda = 0.73(\rho D^2/\mu)^{0.33}(C\mu/k)^{0.66}(\mu/\mu s)^{0.1}$

$h = 1309\ W/m^2.{}^0C$

Corrected Heat transfer Coefficient

$U_c = (h_o \times h)/(h_o + h) = 320 W/m^{2o}C$

Table 15 Specification Sheet of Reactor

Function: Digestion of phosphate rock by 60% nitric acid.	
$Ca_3(PO_4)_2 + 6HNO_3 \rightarrow 3Ca(NO_3)_2 + 2H_3PO_4$	
Operating Temperature	70°C
Operation Pressure	1 bar
Residence Time	0.28 hr.
Volume	11 m^3
Diameter	2.15 m
Length	3.0 m

5.2.4 Thermal design

Hot fluid (Product slurry) inlet temperature = T1 = 70C = 158F

Cold fluid (cold water) inlet temperature = t1 = 25C = 77F

Hot fluid (product slurry) outlet temperature = T2 = 45C = 113F

Cold fluid (cold water) outlet temperature = t2 = 40C = 104F

Mass flow rate of hot fluid (slurry) = W1 = 107365 kg/hour

Mass flow rate of cold fluid (water) = W2 = 114814 kg/hr.

Heat balance Heat inside the exchanger = Q = 7.20E+07 kJ/hr.

Log mean temperature difference

LMTD = $(T1 - t1) - (T2 - t2)\ /\ ln\ (T1 - t1\ /T2 - t2)$

LMTD = 89°F

Corrected LMTD (true temperature)

R = $T1 - T2\ /t2 - t1$ = 1.67

S = $t2 - t1/\ T1 - t1$ = 0.33

Temperature correction factor (FT) should be greater than 0.75.

From appendix, LMTD correction factor = FT = 0.94

A 1-2 heat exchanger will be satisfactory for the specific conditions.

Corrected LMTD = Δt = 89 x 0.94 = 83.66°F.

Caloric temperature

There is negligible change in viscosity on the cold end of heat exchanger therefore, calculations for design can be done on true temperature.

Surface area

Value of UD should be in the range of 250-500 Btu/h-ft2 - 0F. It is always better to assume value of UD too high than too low, so that the final exchanger will just fulfil the requirements.

Place the viscous stream (product slurry) on the shell side and less viscous fluid (water) on the tube side.

Assume overall heat transfer coefficient = UD = 300 Btu/h-ft2 -F.

Area = A =Q / UΔt

= 7.20E + 07/ 1080 x 83.66

= 797.4 m2

Tube Specifications

Assume

Outside diameter of tube = OD = 3/4 = 0.75ft.

Length of tube = L = 21ft.

BWG = 16

Surface/lin. ft. = a’ = 0.502m.

Number of tubes = A/La′

= 797.4/21 x 0.502

= 1587 tubes

Assume 1 in. triangular pitch,

With 2 tube passes, 3/4 in (0.0195m) OD and 1 in. triangular pitch,

Corrected number of tubes (nearest tube count) = NT = 1590 (from appendix)

Inside diameter of shell = Ds = 1.09m.

Shell side (Hot fluid, product slurry)

Baffle spacing

Assume baffle spacing = 5 in.

(Baffle spacing (0.4-0.6 od shell dia) = 0.545m

Clearance = c' = P_T-OD = 1-0.75 = 0.25 ft=0.00635m

Area of shell = As = $^{IDxc'b}/_{144n}$

Area of shell = As = $^{0.545x0.00635x1.09}/_{0.0254}$

= $0.148m^2$

<u>Mass Velocity</u>

$G_s = W_1/A_s$ = 107365/0.148

= $725440^{kg}/m^2hr.$

<u>Reynolds number</u>

Re = $^{D_eG_s}/_{\mu}$

Equivalent diameter of shell = De = 0.73 in.

= 0.061 ft.

Fluid viscosity = μ = 3.13 lb/ft.hr.

Re = $0.061x725440/3.13$

= 1.6E+05

<u>Prandtl number</u>

k = 0.37 $Btu/hr.ft^2.^0F/ft.$

Cp = 0.646 $Btu/lb.^0F$

$(Cp\mu/k)_{1/3} = (0.646x3.13/0.37)_{1/3}$

$(Cp\mu/k)^{1/3}$ = 1.76 Btu/hr.ft^2.^{0}F/ft.

J_H factor

J_H = 250

Heat transfer coefficient (h)

ho = $jH\ k\ /De$ $(Cp\mu/k)^{1/3}$

= 2607.0 Btu/hr.ft2.

Clean overall heat transfer coefficient = Uc = $^{h_{io}h_0}/_{h_{io}} + h_0$ = 1163 Btu/hr.ft^2.^{0}F

Dirt factor = R_d = $^{U_C - U_D}/_{U_C U_D}$ = 6.60E-05 h.ft^2.°F/Btu

Pressure Drop calculations

Re =1.6E+05

Friction factor = f = 0.0012

Number of crosses = N+1 = $12L/B$ = 38.5

De = 0.73in

Specific gravity = s = 1.55

$\Delta Ps = fGs^2 Ds\ (N + 1)/5.22x1010Des$

ΔPs = 3 psi

Table 24 Specification Sheet of Heat Exchanger

Specification Sheet	
Identification	
Item	Heat Exchanger
Type	Shell and Tube Heat Exchanger
Function: Cool the process liquid from 70^0C to 45^0C.	
Tube side	Shell side
Fluid: Cold (Water)	Fluid: Hot (Process liquid)
Flow rate: 114814kg/hr	Flow rate: 107365 kg /hr
Temperature: 77°F to 104°F	Temperature:158°F to 113°F
Pressure drop:1.4 psi	Pressure drop: 3 psi
Tubes: 3/4 in OD. 16 BWG	Shell ID:43 inch
1587 number of tubes each 21 ft long	Baffles spacing: 0.545m
Passes: 02	Passes: 01
Pitch: 1 in Triangular	U_D calculated: 1163 kg/m^2h.F
U_D assumed: 1080 kg/h.m^2 °F	R_d= 6.60E-05

5.3 Centrifuge

5.3.2 Selection

The best technique to heat and cool a process vessel is with a jacket in terms of control, efficiency, and product quality. Today, these fall into three main groups.

Physical properties of a material

The characteristics of the solids and liquid handled in a process will influence centrifuge selection

Specific gravities of solids and liquids

A decanting centrifuge is not an option if the solids are lighter than the liquid. A decanting centrifuge may be considered if the specific gravities are extremely near but the solids are slightly higher, but only if either the particle size or centrifugal force improve the settling of solids.

Particle size

The optimum coarse solids for a filtering type centrifuge typically have particle sizes greater than 100 m. Sedimentation centrifuges are the most effective way to manage the smaller solids, less than 10 m.

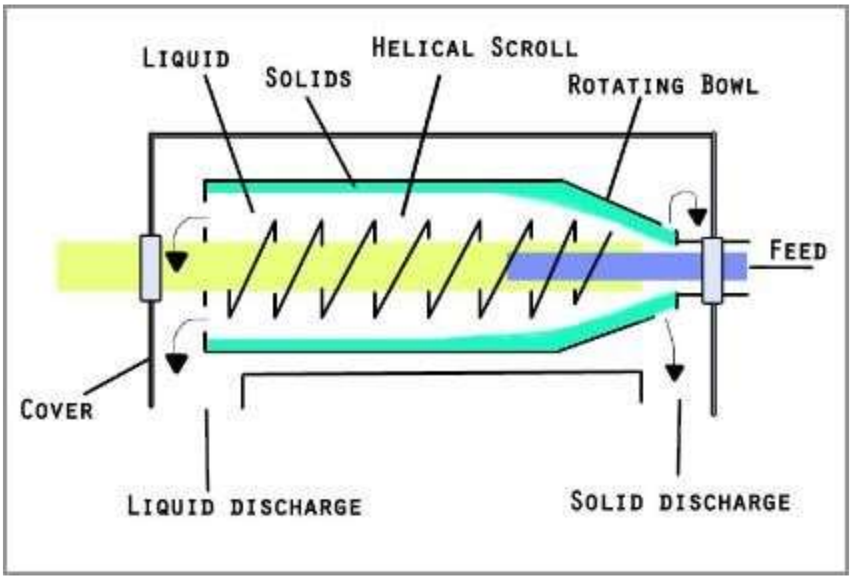

Figure 16 Solid Bowl Centrifuge

5.3.3 Design

Figure 17 Design Data for Centrifuge

Density of solids	1610 kg/m^3
Average Density of mother liquor leaving	820 kg/m^3
Mass Flow rate of feed (kg/hr)	108830
DOA leaving the centrifuge (kg/hr)	1465
Mother liquor leaving (kg/hr)	107365
Diameter of bowl	0.635 m
RPM of centrifuge	50
Average viscosity of liquid	$1.29*10^{-3}$ Pas
Minimum Particle diameter dp	$5*10^{-6}$ m

Mass flow rate of slurry = 81080 kg/hr.

Weighted density of slurry = 2450.51 kg/m^3

Volumetric flow rate of slurry = v_1 = 81080/2450.51 = 33.08 m^3/hr.

Mass flow rate of water = 27750 kg/hr.

Density of water = 1000 kg/m^3

Volumetric flow rate of water = v_2 = 27.75 m^3/hr.

Total volumetric flow rate of feed = v_0 = 60.84 m^3/hr. = 0.017 m^3/s.

Length of centrifuge

Length of centrifuge can be calculated as:

$$L = v_0\mu/3.5D^2(\rho_S - \rho_L)d^2N^2$$

Where,

L = length of centrifuge,
m

μ = average viscosity of
liquid, Pas

v_0 = volumetric flow rate
of feed, m3/s D =
diameter of bowl, m

d = diameter of solid
particle, m

N = rotor speed, rpm

ρ_s = density of mother
liquor, kg/m^3

ρ_L = density of solid
particles, kg/m^3

Length of centrifuge = L = 3.14 m = 10.3 ft.

$L/D = 1.6$

$L = 1.6D$

$D = 1.96m = 6.4$ ft.

Volume of bowl

Volume of bowl = $V = \pi D^2 L/4 = 0.99 m^3 = 34.96 ft^3$

Residence time

Residence time for slurry = $\tau = {}^{\text{Volume of liquid}}/\text{Volumetric flow rate}$

Volume of liquid in bowl = $\pi L(r_2^2 - r_1^2)$

$r_2 = D/2 = 0.635/2 = 0.318$ m

$r_1 = r_2/2 = 0.318/2 = 0.159$ m

Volume of liquid in bowl = $0.74 m^3 = 26.13 ft^3$.

Residence time = 0.012 hr. = 0.73 min.

Relative centrifugal force

$RCF = {}^{F_c}/F_g = \omega^2 r_2/g$

$\omega = 2\pi N = 2x3.1415x50 = 314$ rad/s

RCF = 3199

Settling velocity

Settling velocity can be calculated as:

$$u_t = {d^2(\rho_s - \rho_L)\omega^2 r_2}\Big/_{18\mu}$$

u_t= 0.027 m/s^2

Table 25 Specification Sheet of Centrifuge

Specification sheet	
Item	Centrifuge
Type	Solid bowl Centrifuge
Function	Continuous
Length	3.14m
Diameter	1.96 m
Residence time	0.73 min
Settling velocity	0.027 m/sec^2
Relative Centrifugal Force (RCF)	3199

5.4 Crystallizer

5.4.2 Selection

Cooling scraped surface swenson walker crystallizer is selected. The selection of cooling scraped

surface swenson walker crystallizer includes many aspects but primarily they may be summarized

under the following points.

1) Crystals are produced by lowering the temperature from 45$_o$C to -5$_o$C

2) Process is continuous

3) Minimum fouling problems

4) Minimum capital cost

5) Maintain uniform temperature

6) It is best for viscous material

5.4.3 Design

Volume of Crystallizer

Residence time = t = 8 hr. = 480 min

Volumetric flow rate of feed = V_0 = 79566.32/1658 = 74.571 m$_3$/hr.

Volume of crystallizer = V = V_0xt = 47.99x8 = 596.69 m^3.

Volume for one continuous crystallizer is too large. It is convenient to install 8 batch crystallizers

in series.

Hence Volume for one batch crystallizer = V/8 = 74.587 m^3

Length and diameter

For cylindrical crystallizer:

V = πD2L/4 = 74.5871 m^3

Length of crystallizer = L = 8.5 m

Diameter of crystallizer = D = 3.342 m

Heat transfer area

It can be calculated by using general heat transfer equation:

Q = UAΔT

LMTD = ΔT = 33.73$_0$F = 0.96^{0}C = 273.98 K

Q = 1.79E+07 kJ/hr.

For typical Swenson walker Crystallizer U = 378 kJ/m$_2$.hr.K

Area = 21.6 m^2

Table 26 Specification Sheet of Crystallizer

Specification Sheet	
Item	Crystallizer
Type	Scraped surface Swenson walker
Operation	Batch
Function	Formation of calcium nitrate crystal
Heat transfer area required	21.6 m^2
Length	8.5 m
Diameter	3.34 m
Volume	74.587 m^3
Residence time	480 min

5.5 Evaporator

5.5.2 Selection

Falling film evaporator is selected due to following reasons

1. Best product quality
2. Simple process control and operation
3. High energy efficient

Total feed to the evaporator = 136000 lb/hr.
Temperature of feed = 244.5^{0}F
Specific heat of feed = 0.675 Btu/lb^{0}F
Temperature of steam = T_s = 398^{0}F
Pressure of steam = 16 bar
Water need to be evaporated = 33170 lb/hr.
In double effect evaporator:

$m_1 + m_2 = 33170$ lb/hr

Pressure inside the evaporator = 0.3 kgf/cm^2

Equations for the heat balance are:

For 1st effect:

$m_s\lambda_s + (t_f - t_1) = m_1\lambda_1$

For 2nd effect:

$m_1\lambda_1 + (m_f - m_1)(t_1 - t_2) = m_2\lambda_2$

Here:

m_s = mass flow rate of steam

m_1 & m_2 = water removed from effect

c_1 = specific heat capacity of liquor in effect 1

λ_1 = Latent heat of vapors

Applying heat balance on first effect:

$m_s\lambda_s + (t_f - t_1) = m_1\lambda_1$

Latent heat of steam = 828 lb/hr. [7]

Latent heat in second effect = 918 lb/hr. [7]

Temperature of vapors in effect 2 = t_1 = 289^{0}F By putting the values:

$828m_s - 3541151 = 918m_1$

Heat balance on second effect:

$m_1\lambda_1 + (m_f - m_1)(t_1 - t_2) = m_2\lambda_2$

$m_1 839.24 + 9285684.6 = m_2 1004.6$

The total vaporization rate required is:

$m_1 + m_2 = 33170$

$m_2 = 33170 - m_1$

No by solving the equations simultaneously:

Number of Tubes =

Length of tube = L = 16ft.

Select number of tubes, shell ID and passes at 0.75 inch, OD tubes on 1-inch triangular pitch

$N_t = 37$

Corrected area = N_t*at*L

Area per linear foot =0.1963ft^2

Corrected area = 116.2 ft^2

Corrected U_D = $U_1 = 534.3$ Btu/hr-ft^2-^{0}F

Similarly, for U_2:

$U_2 = 300$ Btu/hr-ft^2-^{0}F

$A_1 = 296.27$ ft^2

$A_2 = 298.7$ ft^2

So A_1 is approximately equal toA_2 <u>Outer Diameter</u>

Let us select ¾ inch nominal pipe size schedule 40 and length of tube is 16ft.

Length of tube = L = 16ft. = 4.87m

Outer Diameter = d_o =
1.05 inch=26.7mm Inner
diameter = 21mm

Surface area for each tube =
$\pi * d_o * L$ = 0.40m^2 Number of
tubes

Area = 30 m^2 (with 10% over design)

N_t = Area/Surface
area of each tube
No of tubes = 75

Select 1-inch
triangular
pitch P_t =
1.25*d_o

P_t = 33.3 mm

Total area occupied by tubes
n =
number of passes = 2

Area occupied by tubes = 0.04 m^2

This area is generally divided by a factor which varies from 0.8 to 1 to find out the actual area. This allows for position adjustment of peripheral tubes as those can't be too close to tube sheet edge.

m^2

Down comer Area

The central down comer area is generally taken as 40 to 70% of the total cross sectional area of tubes.

$A = 0.0208\ m^2$

Down comer diameter

$D = 0.1335\ m$

Area of tube sheet

Total area of tube sheet in evaporator = Down comer area + area occupied by tube $A = 0.0208 + 0.04$

$A = 0.0608\ m^2$

Tube sheet Diameter

$D = 0.227\ m$

4.6.2 Design

Q = 7.2E+07 kJ/hr. = 20060 kW = 20060000 W

Heat transfer is by convection, apply Newton's law of cooling. $Q = hA\Delta T$

Temperature difference = $\Delta T = 80^0C$

Convection heat transfer coefficient for air = h = $10\ W/m^2\text{-}^0C$

Area = $Q/h\Delta T = 2507\ m^2$.

CHAPTER 6

COST ESTIMATION

6.1 Introduction

Cost estimation is a specialized subject and a profession in its own rights. The design engineer needs to be able to make quick, rough, cost estimates. Chemical plants are built to make a profit.

6.1.1 Equipment Purchase Cost of Digester

Stainless steel 304 CSTR

Volume of reactor = 11 m^3

Diameter = 2.15 m

Length = 3.0 m

Equipment exponent = n = 0.7 (from appendix)

Cost constants, a = 14,000 & b = 15,400 (from appendix)

Material factor = 1.3

Cost of reactor in 2006 = (a + b S^n) x Material factor

Cost of reactor in 2023 = $Cost\ in\ 2006\ x\ Index\ in\ 2023/\ Index\ in\ 2006$

Cost of Digester (reactor) = 480,011

6.1.1.1 Equipment Purchase Cost of Centrifuge:

Material of Construction = Stainless steel

Cost of reactor in 2023 = $Cost\ in\ 2006\ x\ Index\ in\ 2023/\ Index\ in\ 2006$

Bowl diameter = 0.635 m

Cost of centrifuge = $ 124930

6.1.1.2 Equipment Purchase Cost of Heat Exchanger

Hot fluid = Process fluid (shell side)

Cold fluid = Coldwater (tube side)

Heat transfer area = 797 m2

Pressure factor = 1.0 (1-10 bar)

Type head = 0.85 (U tube sheet)

Material factor = 1 (carbon steel)

Bare cost of shell and tube heat exchanger in 2004 = $ 130000

Purchased cost = bare cost x pressure factor x type factor

Purchased cost in 2004 = $ 110,500

Cost of exchanger in 2023 = $Cost\ in\ 2004\ x\ Index\ in\ 20123/\ Index\ in\ 2004$

Cost of exchanger in 2023 = $ 150,000

6.1.1.3 Equipment Purchase Cost of Crystallizers

Cooling type scraped surface crystallizer

Length of crystallizer = 8.5 m

Diameter of crystallizer = 3.34 m

Volume of crystallizer = 74 m3

Capacity = 31.72 tons/hr.

8 Crystallizers in Series

Cost for the year 2012 = $ 85,228

Cost index for year 2012 = 584.6

Cost index for year 2018 = 711.11

Cost for year 2023 = $Cost\ in\ 2012\ x\ Index\ in\ 2023/\ Index\ in\ 2012$

Cost for year 2023 = $ 103,672

Cost of 8 crystallizers in series = 8 x 103,672

= $ 819,642

6.1.1.4 Equipment Purchase Cost of Centrifuge

Bowl diameter = 0.72 m

Material of Construction = Stainless steel

Cost of centrifuge in 2002 = $ 80,000

Cost index in 2002 = 395.6

Cost index in 2023 = 711.11

Cost of centrifuge in 2023 = Cost in 2002 x Index in 2023/ Index in 2002

Cost of centrifuge = $ 143,804

6.1.1.5 Equipment Purchase Cost of Neutralizer

Volume of reactor = 60.19 m3

Diameter = 3.80 m

Length = 5.32 m

Equipment exponent = n = 0.7

Cost constants, a = 14,000 & b = 15,400

Material factor = 1.3

Cost of reactor in 2006 = (a + b S^n) x Material factor

Cost of reactor in 2006 = $ 411,029

Cost index in 2006 = 478.6

Cost index in 2023 = 711.11

Cost of reactor in 2023 = $Cost\ in\ 2006\ x\ Index\ in\ 2023/\ Index\ in\ 2006$

Cost of reactor = $ 610,712

6.1.1.6 Equipment Purchase Cost of Evaporators

Dual effect falling film evaporator

75 Tubes, 2-Passes, 28m2 Area

Area of 1st effect = 296 ft2 = 27.4 m2

Area of 2nd effect = 298 ft2 = 27.6 m2

Hot fluid = Steam at 205^{0}C & 16 bar

Cold fluid = NP solution

Use the following formula to calculate purchased cost

Ce = CS^n

Where Ce = Purchased cost, $

S = Characteristic size parameter = 27.4

C = Cost constant, $ = 10,000

n = Index for that type of equipment = 0.52

material = carbon steel

Cost of 1st effect for year 2004 = $ 55,928

Cost index for year 2004 = 444.2

Cost index for year 2023 = 711.1

Cost of 1st effect for year 2023 = $Cost\ in\ 2004\ x\ Index\ in\ 2023/\ Index\ in\ 2004$

Cost of 1st effect for year 2023 = $ 89,532

Area of 1st effect is approximately equal to the 2nd effect.

Therefore, Cost of 2nd effect for year 2023 = $ 89,532

Total cost of evaporators = $ 179,064

6.1.1.7 Equipment Purchase Cost of Prilling Tower

Length of tower = 53 m

Diameter of tower = 15 m

Area of tower = A = 2507 m3

Cost of tower for year 1987 = $ 260,000

Cost index for the year 1987 = 326

Cost index for the year 2023 = 711.11

Cost of tower for the year 2023 = Cost in 1987 x Index in 2023/ Index in 1987

Cost of tower for the year 2023= $ 567,143

6.1.1.8 Equipment Purchase Cost of Pump 1

High speed centrifugal pump

Inlet pressure = 1 bar

Discharge pressure = 2 bar

Pump work = -176.20 J/kg

Capacity = 10.52 L/s

Cost constants, a = 3300 & b = 48

Equipment exponent = n = 1.2

Cost of pump in 2006 = a + b S^n

Cost of pump in 2006 = $ 5,562

Cost index in 2006 = 478.6

Cost index in 2023 = 1032.4

Cost of pump in 2023 = Cost in 2006 x Index in 2023/ Index in 2006

Cost of pump in 2023 = $ 11,998

6.1.1.9 Equipment Purchase Cost of pump 2

Centrifugal pump

Inlet pressure = 0.30 bar

Discharge pressure = 1 bar

Pump work = -165.18 J/kg

Capacity = 7.32 L/s

Cost constants, a = 3300 & b = 48

Equipment exponent = n = 1.2

Cost of pump in 2006 = a + b S^n

Cost of pump in 2006 = $ 4,103

Cost index in 2006 = 478.6

Cost index in 2023 = 1032.4

Cost of pump in 2023 = Cost in 2006 x Index in 2023/ Index in 2006

Cost of pump in 2023 = $ 8,851

6.1.2 NP Plant Costing

Table 27 NP Plant Purchase Cost

Digester	480011
Neutralizer	610712
Centrifuge 1	124930
Centrifuge 2	143804
Heat exchanger 1	150000
Crystallizers	819642
Evaporators	179064
Prilling tower	567143
Pump 1	11998
Pump 2	8851
Pump 3	9824
Pump 4	10948
Total Purchase Cost	3116927

6.1.3 Pre-Treatment Plant Costing

Table 28 Pre-Treatment Plant Purchase Cost

Wet grinder	6500
Wet re-grinder	11000
Jaw crusher	42000
Conditioning tank 1	185400
Conditioning tank 2	160785
Conditioning tank 3	14100
Conditioning tank 4	11900
Column floatation tank	200500
Rougher floatation tank	175667
Cleaner floatation tank	150572
Reverse floatation tank	125476
4 Floatation tank impellers	26832
Blower 1	136268
Blower 2	862798
Blower 3	580963
Blower 4	662445
4 Conditioning tank agitators	24148
Total Purchase cost	3377354

Total Purchase cost of Plant = 6494281

6.1.3.1 Raw Material Cost of NP-Plant

Table 29 Rock Phosphate Cost

Flowrate of Phosphate rock	65000kg/hour	
Operating days in a year	300	
Flowrate per year	468000	ton/hour
Cost of Rock Phosphate	20	dollar/ton
Cost of rock phosphate per year		9360000

Table 30 Nitric Acid Cost

Flowrate of nitric acid	23404kg/hour
Flowrate of nitric acid per year	168508 ton
Cost of nitric acid	500$/ton
Cost of nitric acid per year	84254000

Table 31 Ammonia Cost

Flowrate of ammonia	7157kg/hour	
Flowrate of ammonia per year	51530400	
	515304	ton/year
Cost of ammonia	200	$/ton
Cost of ammonia per year	103060800	

Total raw material cost = 196674800

6.1.3.2 Utilities Cost of NP-Plant

Table 32 Cooling Water NP Plant Cost

Flow rate of cooling water	460000	kg/hr
Cost of cooling water	0.00001	$/kg
Flowrate of cooling water per year		3312000000
Cost of cooling water per year		33120

Miscellaneous Materials Cost of NP-Plant

Total Variable cost of NP-Plant = 200,072,730

6.1.4 Variable Cost of Pre-Treatment Plant

6.1.4.1 Raw Material Cost of Pre-Treatment Plant

Total raw material cost= 3304245

Total Variable cost of Pre-Treatment Plant = 4,114,164

Total Variable cost of Both Plants = 204186894

6.1.5 Fixed Cost of NP-Plant

Table 33 Fixed Cost NP Plant

Maintenance	676373.159
Operating Labor	0.01(TPC)
Laboratory	0.015(TPC)
Supervision	0.02(TPC)
plant overhead	0.05(TPC)
Capital charges	966247.37
Insurance	96624.737
Local taxes	193249.474
Royalties	96624.737

6.1.6 Fixed Cost of Pre-Treatment Plant

Table 34 Fixed Cost Pre-Treatment Plant

Maintenance	732885.8
Operating Labor	0.01(TPC)
Laboratory	0.015(TPC)
Supervision	0.02(TPC)
Plant overhead	0.05(TPC)

Capital charges	1046980
Insurance	104698
Local taxes	209395.9
Royalties	104698

Direct production cost = variable cost + fixed cost

Total production cost of Plant = 208,726,717

TPC = total production cost = $ 208,726,717/yr.

Production rate = 15000 MTPD

Total production rate = 450,000 MTPA (ton/yr.)

Production cost ($/ton) = total production cost ($/yr.)/total production rate (ton/yr.)

Production cost ($/ton) = $ 463.83/ton

Below this, we cannot sale our product.

6.1.7 Selling Price

Selling price = $ 540.5/ton

6.1.8 Profitability Analysis

Production cost = $ 0.463/kg

Selling price = $ 0.540/kg

Profit = Selling price – production cost

= 0.463 – 0.540

= $ 0.0767/kg

= $ 76/ton

Total production per year = 450,000 ton/yr.

Profit per year = 76 x 450,000

= $ 34516525.6/y

6.1.9 Total Income

Selling price of NP = $ 540.5/ton

Total production per year = 450,000 ton/yr.

Total income = 450,000 x 540.5

= $ 243,243,243/yr.

6.1.10 Gross Profit

Gross profit = Total income – Total production cost

= 243,243,243– 208726717.7

= $ 34516525.6/yr.

6.1.11 Net Profit

Assume that the fixed capital investment depreciated by straight line method for 20 years.

Assuming 5% selvage value at the end of plant life.

Depreciation = D = $V - V_s$ /N

V = FCI = $ 20132271.1

Salvage value = V_S = 0.05(FCI) = $ 1,006,613.56

Number of years = N = 20 years.

Depreciation = D = $ 20,081,940.4

Net profit before taxation = Gross profit – Depreciation

= $ 14434585.2/yr.

Taxes = 29% of net profit before taxation

Taxes = 0.29(14,434,585.2)

= $ 4186029.7/yr.

Net profit after taxation = Net profit before taxation – Taxes

= $ 10,248,555.5/yr

6.1.12 Rate of Return

Rate of return = (Net Profit /Total Capital Investment) x 100

Rate of return = 44.26%

6.1.13 Payback Period

Payback period = 1/Rate of return

Payback period = 2.25 years

CHAPTER 7

INSTUMENTATION AND CONTROL

7.1 Objective

The primary objectives of the instrumentation and control are:

1. To help the process variables within safe operating limits.

2. To detect dangerous situations as they develop and to provide alarms and automatic shutdown systems.

3. To provide inter locks and alarms to prevent dangerous operating procedures.

7.2 Cost

To operate at the lowest production cost, commensurate with the other objective. These are not separate objectives and must be considered together. Measurement is a fundamental requisite of process control either the control will be affected automatically, semi-automatically or manually.

The quality of the control obtainable is a function of the accuracy, repeatable and reliability of the measuring devices employed. The objective of an automatic process control is to use the manipulated variable to maintain the controlled variable at its set point in spite of disturbances. Instruments are provided to monitor the key process variables during plant operations. Instruments monitoring critical process variables will be fitted with automatic alarms to alert, the operations to critical and hazardous situations. Pneumatic instruments are used in this plant. The main process parameters are all indicated in the control room where automatic or remote control is carried out centrally. The process parameters e.g. temperatures, pressure, flow, liquid level etc. are converted to signals with transducers and then indicated, recorded and controlled with secondary instruments.

7.3 Process Instrumentation

No chemical plant can be operated unless it is adequately instrumented. The monitoring of flows, pressures, temperatures and levels is necessary in almost every process in order that plant operator can see that all part of plant functioning as required. Additionally, it may be necessary to record and display many other quantities which are more specific to the particular process in question. In many instances sensors forms an essential part of control system or strategy for a process. Process instrumentation may be quite complex and rely upon the performance and characteristics of a substantial number of different sensors.

7.3.1 Temperature

Measurement and control of temperature are possibly the most common operations in process control. The basic principles of temperature measurement are:

1. Expansion of substance with temperature, which produces a change in length, volume, pressure in its simplest form this is the common mercury in glass thermometer.

2. Change in electric resistance with temperature used in resistance thermometers (RTD) and thermostats.

3. Change in contact potential between dissimilar metals with temperature thermocouples.

4. Change in radiant energy with temperature, optical and radiation pyrometers.

7.3.2 Pressure

For measurement of pressure, all pressure transducers are concerned with measurement of static pressure e.g. pressure of the fluid at rest. The most common of pressure transducer is DP cell which is used in conjunction with a sensing device such as an orifice meter, any mechanism of outputted.

Pneumatic, electrical or mechanical can be coupled with it for signal transmission.

7.3.3 Flow

The measurement of flow is an essential part of almost every industrial process, and many techniques have been evolved for it. Measurement of flow usually employs the same principal as the measurement of pressure i.e. sensing device coupled with a DP cell. For special applications other flow meters may be employed e.g. for process no external disturbance in the fluid stream is required for magnetic flow meters.

7.3.4 Concentration

Knowledge of composition of a process stream is often of major importance. Such information may be necessary to determine whether the particular product has the required specification or whether composition of a particular stream is changing. Frequently, variation in either will require some kind of control action to maintain plant. Property of material is usually employed for the measurement of concentration e.g. absorption of electromagnetic waves, refractive index, pH, density of components as in chromatography.

7.4 Control Loops

For instrumentation and control of different sections and equipment’s of plants, following control loops are most often used.

1. Feedback control loop
2. Feed forward control loop
3. Cascade control loop

7.4.1 Feedback Control Loop

A method of control in which a measured value of a process variable is compared with the desired value of the process variable and any necessary action is taken. Feedback control is considered as the basic control loops system. Its disadvantage lies in its operational procedure. For example, if a certain quantity is entering in a process, then a monitor will be there at the process to note its value. Any changes from the set point will be sent to the final control element through the controller so that to adjust the incoming quantity according to desired value (set point). But in fact, changes have already occurred and only corrective action can be taken while using feedback control system.

7.4.2 Feed forward Control Loop

A method of control in which the value of disturbance is measured than action is taken to prevent the disturbance by changing the value of a process variable. This is a control method designed to prevent errors occurring in a process variable. This control system is better than feedback control because it anticipates the change in the process variable before it enters the process and takes the preventive action. While in feedback control system action is taken after the change has occurred.

7.4.3 Cascade Control Loop

This is a control in which two or more control loops are arranged so that the output of one controlling element adjusts the set point of another controlling element. This control loop is used where proper and quick control is difficult by simple feed forward or feed backward control. Normally first loop is a feedback control loop.

7.5 Cascade Control Loop on Crystallizer

Control Objective Let us discuss instrumentation and control on crystallizer. Calcium nitrate tetra-hydrate crystals are formed inside the crystallizer. Conditions should be maintained inside the crystallizer to form specific calcium nitrate tetra-hydrate crystal size

Where,

TT = Temperature transmitter

FT = Flow transmitter

LT = Level transmitter

FC = Flow controller

LC = Level controller

TC = Temperature controller

T = Temperature inside the crystallizer, ^{0}C

T_c = Temperature of coolant, ^{0}C

CHAPTER 8

HAZOP STUDY

8.1 Introduction

The HAZOP study is a formal procedure used to identify potential dangers in a chemical process facility. It is widely accepted and effective within the chemical industry. The main concept behind HAZOP is to encourage creative thinking while maintaining control, allowing for the consideration of all possible ways that process and operational failures could occur. Before starting the HAZOP study, it is crucial to have detailed information about the process, such as up-to-date process flow diagrams (PFDs), process and instrumentation diagrams (P&IDs), and specific equipment specifications, construction materials, and mass and energy balances

A complete HAZOP study involves a committee comprising various professionals with experience in plant operations, laboratories, technical fields, and safety. The committee is led by a trained HAZOP leader who acts as the chairperson, guiding the discussions. This person should have expertise in the HAZOP procedure and the specific chemical process being reviewed. Another individual is assigned the responsibility of recording the results, although there are software options available from vendors to assist with this task using a personal computer.

The committee convenes regularly for a few hours at a time, ensuring that the meeting duration is kept short to maintain interest and active participation from all committee members. For a complex process, the HAZOP study may require several months of biweekly meetings to reach completion. Although conducting a thorough HAZOP study demands significant time and effort, the resulting value makes it a worthwhile endeavor.

8.2 Background

The main purpose of a HAZOP study is to identify potential hazards and operational issues. The study involves investigating how the plant may deviate from its intended design. During the HAZOP study, if a solution to a problem becomes apparent, it is documented as part of the HAZOP results. However, it's important to be cautious about trying to find solutions that may not be readily apparent, as the primary focus of the HAZOP study is problem identification. While the HAZOP study was initially developed to complement experience-based practices for new designs or technologies, its application has expanded to encompass almost all stages of a plant's lifespan.

The HAZOP methodology is based on the principle that when several experts with diverse backgrounds collaborate, they can collectively identify more problems than they would individually, and their results can be combined. The most well-known form of HAZOP is the "Guide-Word" HAZOP, but various specialized versions of this basic method have also been developed.

8.3 Success or Failure

Several factors contribute to the success or failure of a HAZOP study:

1. The completeness and accuracy of the drawings and other data used as the foundation for the study play a crucial role. Having comprehensive and precise information ensures that the study is based on reliable and relevant data.

2. The technical skills and insights of the team members are vital. Their expertise and knowledge enable them to analyze the process effectively, identify potential hazards, and propose appropriate solutions.

3. The team's ability to utilize the HAZOP approach as a tool for stimulating their imagination is important. By visualizing deviations, causes, and consequences, they can uncover potential issues that may arise during the operation of the plant.

4. It is essential for the team to prioritize and concentrate on the more severe hazards that are identified during the study. By focusing on these critical risks,

they can develop suitable control measures and preventive actions to mitigate their impact.

These factors collectively contribute to the overall effectiveness and outcomes of the HAZOP study, ensuring that it serves its purpose of identifying hazards and improving the safety of the chemical process facility.

8.4 HAZOP Characteristics

HAZOP is a highly suitable method for evaluating hazards in facilities, equipment, and processes, as it offers the capability to assess systems from multiple perspectives. Here are some key aspects of HAZOP:

8.4.1 Design

HAZOP allows for the assessment of the system's design capabilities, ensuring they meet user specifications and safety standards. It helps identify any weaknesses or flaws in the system design.

8.4.2 Physical and operational environments

HAZOP assesses the environment in which the system operates to ensure it is appropriately situated, supported, serviced, and contained, among other factors.

8.4.3 Operational and procedural controls

The methodology evaluates engineered controls, such as automation, as well as sequences of operations and procedural controls. It examines different operational modes, including start-up, standby, normal operation, steady and unsteady states, normal shutdown, and emergency shutdown.

CHAPTER 9

ENVIRONMENTAL IMPACT AND ASSESMENT

Environmental Impact Assessment (EIA) involves evaluating the potential environmental effects of a project or plan and finding ways to minimize harm to the environment. The primary goal of EIA is to provide decision makers with information about the expected impacts of a proposal before they make a final decision. By conducting an EIA, we can identify important concerns and involve relevant stakeholders early on in the process, allowing us to address any potential negative impacts before making final approval decisions. The EIA report also includes a detailed description of the actions taken to prevent, lessen, or rectify these effects.

9.1 Introduction

The US Environmental Protection Agency (EPA) has played a significant role in developing pathway analysis as a means to assess the potential impact of environmental factors on human health. This scientific approach, commonly known as environmental science, enables us to analyze and understand how various factors in the environment can affect human well-being.

The primary areas of focus in pathway analysis include:

1. Effects of noise on human health

2. Impacts of water pollution

3. Assessment of ecological impacts, including the evaluation of endangered species

4. Effects of air pollution

5. Impacts of soil contamination

6. Assessment of geological hazards

By examining these specific pathways of impact, we can gain a comprehensive understanding of the potential consequences associated with environmental factors and take appropriate measures to mitigate their effects.

9.2 Objectives

The objectives of environmental impact assessment are as follows:

1. Ensuring that environmental factors are taken into account during the decision-making process.

2. Identifying and minimizing any potential adverse environmental impacts.

3. Informing the public about the proposed project.

9.3 Advantages

There are several advantages associated with environmental impact assessment:

1. It allows people to evaluate the underlying necessity of a project.

2. It provides the opportunity for individuals to identify potential issues or concerns.

3. It helps project developers design initiatives that are more acceptable to the public.

9.4 Ammonia

Ammonia is a compound made up of nitrogen and hydrogen, represented by the chemical formula $NH3$. It is a colorless gas that has a distinct, strong smell. Ammonia boils at a temperature of -33.34 °C (-28.012 °F) under normal atmospheric pressure, which means it needs to be stored under pressure or at low temperatures in its liquid state. When combined with oxygen, it burns with a pale yellowish-green flame. At high temperatures and in the presence of an appropriate catalyst, ammonia can be broken down into its individual elements.

9.4.1 Hazard

There are several hazards associated with ammonia:

Fire hazards

1. Ammonia vapor in the air is flammable and can potentially explode if ignited.

2. It is chemically stable under normal conditions.

3. When heated to its decomposition temperature, it releases poisonous fumes.

4. There is an increased risk of fire and explosion when ammonia comes into contact with oxidizing agents.

Health hazards

1. Exposure to ammonia through any route can be dangerous. The main routes of exposure include:

   - Inhalation (inhalation of ammonia is highly toxic and can lead to death).

   - Skin contact (contact with the skin can cause irritation or burns).

   - Eye contact (contact with the eyes can cause permanent blindness or other severe diseases).

2. Ammonia is not considered to be carcinogenic to humans.

3. Ammonia is not considered to be a toxicant for human reproduction or development.

4. Acute inhalation of ammonia may result in irritation of the eyes and nose, sore throat, coughing, chest tightness, headache, and confusion.

5. Acute skin exposure to ammonia may result in severe burns.

6. Any leaks, spills, or failures of safety equipment should be immediately reported.

7. In the event of a spill or leak, individuals should immediately put on an escape-type respirator and leave the area.

8. Medical attention should be sought for all exposures to ammonia.

9.4.2 Protective measures

When working with ammonia, the following personal protective equipment (PPE) is necessary:

- Eye/face protection: Wear chemical safety goggles. A face shield, along with safety goggles, may also be required.

- Skin protection: Wear chemical protective clothing, such as gloves, aprons, and boots. In certain operations, a chemical protective, full-body encapsulating suit and a self-contained breathing apparatus (SCBA) may be necessary.

- Respiratory protection: For concentrations up to 250 ppm, use a chemical cartridge respirator with cartridges providing protection against ammonia. Alternatively, a supplied-air respirator can be used.

9.4.3 Spills and emergencies

If employees are responsible for cleaning up ammonia spills, they must receive proper training and have the appropriate equipment. In the event of an ammonia leak, follow these steps:

1. Evacuate personnel and secure the area by controlling access to it.

2. Ventilate the area to disperse the gas.

3. If the source of the leak is a cylinder and it cannot be stopped in place, remove the leaking cylinder to a safe, open-air location and either repair the leak or allow the cylinder to empty.

4. Move cylinders away from any potential fire hazards.

5. The threshold limit value of ammonia is 25 to 35 ppm.

9.5 Nitric acid

Nitric acid (HNO3) is a highly corrosive mineral acid. When the solution contains more than 86% HNO3, it is referred to as fuming nitric acid.

9.5.1 Hazards

1. Inhalation of nitric acid vapors may cause a burning sensation, coughing, unconsciousness, and even death. Symptoms may have a delayed onset.

2. Skin contact with nitric acid can result in serious burns and pain.

3. Eye contact with nitric acid may cause redness and pain.

4. Ingestion of nitric acid may lead to abdominal pain, a burning sensation, shock, and vomiting.

5. Prolonged exposure to concentrated nitric acid vapors can cause damage to the lungs.

9.5.2 Protective measures

1. If exposed to nitric acid vapors through inhalation, immediately move the affected person to fresh air. Use proper ventilation, local exhaust systems, and respiratory protection to prevent inhalation exposure. Artificial respiration may be necessary. Seek immediate medical attention.

2. In case of skin contact, remove contaminated clothing and rinse the affected skin with plenty of water or take a shower. Seek medical attention. Use protective gloves and clothing to prevent skin exposure.

3. If nitric acid comes into contact with the eyes, flush them with cool water for at least 15 minutes. To prevent eye exposure, use a face shield or eye protection along with respiratory protection.

9.5.3 Exposure Controls

1. Local exhaust ventilation or respiratory protection should be used. Secondary containment should be in place for storage and use if there is a risk of exposure to employees or the environment.

2. Depending on the quantities involved, regulatory permits and registrations may be required. Personnel working with nitric acid should receive comprehensive training on the hazards, safe handling, and emergency procedures.

3. Avoid all contact with the substance. Prevent skin and eye contact by using impervious gloves, clothing, boots, aprons, eye goggles, and a full face shield. If the airborne exposure limit may be exceeded and engineering controls are not feasible, appropriate respiratory protection should be worn.

4. Nitric acid should be disposed of as hazardous waste. Contact the Waste Management Group for specific disposal requirements and procedures. Containers and materials contaminated with nitric acid should also be treated as hazardous waste. Leaking liquid should be collected in sealable containers. Any remaining nitric acid should be cautiously neutralized with sodium carbonate and washed away with plenty of water.

5. In the event of a significant release that poses a threat to employees or the environment, immediately evacuate the area and notify your supervisor.

6. Store nitric acid separately from combustible materials in a cool and dry place with good ventilation.

7. The threshold limit value for nitric acid is 2 to 4 ppm.

9.6 Nitro-phosphate fertilizer

Nitro-phosphate (NP) is a complex granulated fertilizer primarily composed of nitrogen (N) and phosphorus (P2O5) with small amounts of calcium (Ca). It has an acidic nature with a pH of 3.5 and is suitable for soils with higher pH levels.

1. Phosphate fertilizer manufacturing facilities produce air emissions through the combustion of gas or diesel in turbines, boilers, compressors, pumps, and other engines used for power and heat generation.

2. Air emissions from NPK plants include ammonia emissions from ammonization reactors, nitrogen oxides (NOX) such as NO and NO2 along with some nitric acid from phosphate rock digestion in nitric acid, fluorides from phosphate rock reactions, aerosol emissions including ammonium nitrate (NH4NO3), ammonium fluoride (NH4F), and ammonium chloride (NH4Cl) formed during the gas-phase neutralization reaction between ammonia and acidic components, as well as fertilizer dust from drying and cooling drums and other sources (e.g., screens, crushers, conveyors).

3. Fugitive emissions occur due to operational leaks from tubing, valves, connections, flanges, packing, open-ended lines, floating roof storage tanks, pump seals, gas conveyance systems, compressor seals, pressure relief valves, tanks or open pits/containments, and loading and unloading operations of products.

Wastewater

Effluent from NPK facilities using the nitro-phosphate route may contain ammonia, nitrate, fluoride, and phosphate. Ammonia is present in the effluents of condensates from ammonium nitrate evaporation or neutralization of nitro-phosphoric acid solution. Handling of solutions containing ammonium nitrate must be done with caution to mitigate explosion risks. The main sources of nitrate and fluoride in effluent are scrubber liquors from phosphate digestion and sand washing (removed from the process slurry). Sand washing also contributes to phosphate content in the effluent.

Hazardous materials

Phosphate fertilizer manufacturing plants handle, store, and distribute significant quantities of hazardous materials, including acids and ammonia.

Wastes

Non-hazardous solid wastes can be generated from certain phosphate fertilizer manufacturing processes, such as phosphor-gypsum from wet phosphoric acid production and quartz sand from NPK production using the nitro-phosphate route. Quartz sand should be separated, washed, and recycled as a building material. The generation of hazardous waste from phosphate fertilizer manufacturing processes is limited.

Noise

Noise is generated by large rotating machinery in phosphate fertilizer manufacturing plants, including compressors, turbines, pumps, electric motors, air coolers, rotating drums, spherodizers, conveyor belts, cranes, and fired heaters. Emergency depressurization can also contribute to noise levels.

9.6.1.1 Recommended measures

1. Reduce NOX emissions from the use of nitric acid in phosphate rock digestion by implementing measures such as controlling reactor temperature, optimizing the rock-to-acid ratio, and adding urea solution.

2. Install spray tower scrubbers to treat gases from the digestion reactor and recover NOX and fluorine compounds. The pH of the scrubber can be adjusted by adding ammonia.

3. Implement scrubbing systems to control fluoride emissions effectively.

4. Apply appropriate controls, such as multistage scrubbing or conversion into cyanides, to reduce emissions to the air from phosphate rock digestion, sand washing, and CNTH (Cyclone/Nozzle Tower Hydration) filtration.

5. During the design, operation, and maintenance of phosphate fertilizer manufacturing facilities, select appropriate valves, flanges, and fittings to minimize the potential for leaks and emissions.

6. Establish monitoring, maintenance, and repair programs specifically targeting areas prone to accidental releases, such as stuffing boxes on valve stems and seats on relief valves, to reduce or eliminate such releases.

7. Avoid the use of open vents in tank roofs and instead install pressure relief valves. Install vapor recovery units in all storage and unloading stations to capture vapors. Vapor processing systems can utilize methods like carbon adsorption, refrigeration, recycling, and combustion.

8. Treat wastewater through biological treatment processes that include nitrification/de-nitrification and precipitation of phosphorous compounds to remove pollutants effectively.

CONCLUSION

The P_2O_5 content of investigated rock phosphate of Hazara was found to be 22% which is quite enough to beneficiate economically. The two-stage direct and reverse floatation techniques was found to be suitable and effective technique for upgradation of this ore. However, the performance and efficiency of phosphate rock upgradation by floatation is sensitive to many parameters. Desliming of phosphate ore prior to floatation has significantly increased the efficiency and recovery of concentrate. The use of column floatation in this process enables us to achieve recovery closer to the perfect. The reverse floatation has improved the grade of final concentrate to a level that would not be possible with only direct floatation. The overall floatation process produced a commercial grade phosphate concentrate assaying 32%. The obtained concentrate can be utilized for the production of chemicals and fertilizers.

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