## Use of local rock phosphate to manufacture Di-Ammonium Phosphate



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# This thesis is submitted as a partial fulfillment of the requirements for the degree of

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May 2019

#### Certificate

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## Dedication

Our work is dedicated to SCME and our beloved parents.

## Acknowledgement

All thanks to Allah Almighty for His countless blessings and rewards, who gave us the strength and ability to complete this thesis.

We would like to thank our supervisor Dr. Waqas Cheema for his support and guidance during our project. He has been very generous and supportive and helped us a lot to complete this thesis. We are thankful to other faculty of SCME as well, especially Dr. Bilal khan Niazi for his assistance.

We would also like to thank our industrial supervisor Mr Abdur Rehman for his guidance and help throughout the project and Fatima Fertilizer Company Limited for giving us such a useful project and providing us all the data and details needed for completion of project.

We are also thankful to our parents, without their support and encouragement; it would not have been possible to complete the project successfully.

#### Abstract

The document provides information about the work that was done on the process design of a Diammonium Phosphate Plant. With the growing economy and fast paced motion of the world towards more economically efficient and environmentally friendly processes, our design gives the best possible solutions for the local market requirements. Our design utilizes the local resources and therefore minimizes imports and extra logistical costs. Similarly it utilizes the use of latest software technology to test the validity and efficiency of our process through Aspen Tech Simulations. The traditional processes being used have been described and the problems they faced which make them less desirable have also been studied.

The main raw material involved is phosphoric acid which was previously imported, would now be produced within the country due to our process design and therefore, minimizing extra costs. With our process, we plan to reduce the cost of 50 kg DAP bag from \$ 23 to \$19. Our process also utilizes all the energy streams very efficiently making it a highly energy and cost efficient plant.

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### Nomenclature

- ΔTm Log Mean Temperature Difference
- m Meter
- mm Millimeter
- cm Centimeter
- m<sup>2</sup> Meter square
- m<sup>3</sup> Meter cube
- cm<sup>2</sup> Centimeter square
- cm<sup>3</sup> Centimeter cube
- KJ Kilo Joule
- kg Kilogram
- h Hour
- C<sub>v</sub> Heat Capacity of Vapor
- Cl Heat Capacity of Liquid
- gmol gram mole
- L Length
- μ Viscosity
- v velocity
- A Area
- y<sub>1</sub> Mole Fraction
- y<sub>2</sub> Mole Fraction
- T Temperature
- ΔT Temperature difference
- m mass flow rate
- Q Energy
- °C Centigrade
- °K Kelvin
- P Pressure
- ρ Density

- U<sub>a</sub> Overall Heat Transfer Coefficient
- W Watt
- H<sub>2</sub> Hydrogen
- N<sub>2</sub> Nitrogen
- ΔP Pressure Difference
- PCE Purchase Cost of Equipment
- hi Internal Pipe Heat Transfer Coefficient
- As Cross-Sectional Area
- d<sub>e</sub> Equivalent diameter
- s Second
- Ce Cost of equipment
- C Constant
- S Capacity of Vessel
- n index

## **1: Introduction to Fertilizer**

## **1.1 Fertilizer**

Fertilizers consist of chemical nutrients for the proper development of plants. These nutrients are used by plants to make food for their growth, flower production, and for the production of fruits and seeds. Photosynthesis is the process through which plants produce their food. The nutrients are taken up from the soil in dissolved form.

Fertilizers are classified as being either organic or inorganic based on their composition. Organic types are obtained from natural sources while inorganic are manufactured in the industry. And therefore, the fertilizer industry plays a vital role in agriculture sector.

#### 1.1.1 Nutrients and their classification

Table 1 Nutrients and their Classification (Ernest Kirkby et al., 2012)

Essential Elements	
Sourced from air, water and soil	Carbon
	• Hydrogen
	<ul> <li>Oxygen</li> </ul>
	• Nitrogen
Sourced from soil and/or fertilizers	Phosphorous
	• K
	• Ca
	• Mg
	• Sulfur
	• B
	• Zn
	• Cu
	• Mn
	• Fe
	• C1
	• Mb

Carbon, hydrogen and oxygen are the most essential elements for growth and development of plants. These elements are not provided to the plants artificially because these are elements that are already in abundance. A total of 90-95% of the dry mass of plants is made up of these essential elements. Carbon is extracted from the organic material in the soil such as compost; oxygen from air; and hydrogen is extracted from water.

Nutrients that are not present in abundance are the components to be concerned about. These nutrients are required for strong and healthy plant growth. The nutrients are fifteen in number and fall in the following three categories.

- Primary nutrients
- Secondary nutrients
- Micro nutrients

Their classification is shown in the table below:

Table 2 Classification of Plant Nutrients (Ernest Kirkby et al., 2012)

Used in large amounts macronutrients		Used in small
		amounts
Primary Nutrients	Secondary Nutrients	Micronutrients
Nitrogen	Calcium	Iron
Phosphorous	Magnesium	Magnesium
Potassium	Sulfur	Zinc
		Copper
		Boron
		Chlorine
		Cobalt

#### **1.1.2 Function of Nutrients**

These nutrients provide specific benefits to plant life. The different quantity needed for plants decides the classification of fertilizers.

Nitrogen is the most critical element. It is the primary element responsible for productivity of plants. This element is responsible for vegetative plant growth. Absence of this element would result in lack of lush green colour and stunted growth. The process of photosynthesis, the most crucial process in plant growth, cannot be performed in the absence of this element.

- Phosphorus (P) is responsible for string root systems, better fruit and flower growth. Resistance against diseases is developed in plants due to this element. Soils rich in phosphorus content produce the biggest flowers, more fruits, strong stem and root systems.
- The third element is Potassium (K), which promotes colour and vigour. Potassium is responsible for sugar production in fruits. It is also crucial in developing resistance against diseases, surviving extreme temperatures and providing protection from drought.

#### **1.1.3 Phosphorus Requirement**

The quantity of phosphorus required in the soil is dependent upon the species being grown and the level of desired product. The concentration between 0.2 to 0.3 ppm is adequate for a variety of crops. Maximum levels of corn grain yield may be obtained when solution concentrations are as low as 0.01 ppm if the yield potential is low, but high yield potential is associated with a level of ppm. Wheat requirement of phosphorus is greater than that of corn. Sorghum has similar requirement as that of corn. Soybean has much higher requirement. Two types of phosphorous in soils are used: Organic soil phosphorous & inorganic phosphorous. (Plaxton WC et al., 1999)



Figure 1 Importance of Phosphorous (Barrow NJ et al., 1980)

#### **1.1.4 Commercial Fertilizers**

Commercial fertilizer provides a supply of plant nutrients for soil use to feed crops if the soil cannot satisfy the total requirement for plant growth. Plants can absorb the nutrients they require only when the chemical compounds are easily dissolved. Organic fertilizers almost always have significantly lower plant nutrient concentration and common problems with

cost-effective collection, treatment, transport and distribution; which is why it is preferred that the nutrients be supplied in the form of inorganic compounds, made industrially.

The nutrients of plants are inorganic. (Ann McCauley et al., 2010)

Nutrient	Plant available form		
Nitrogen	NH4 <sup>+</sup> and NO3 <sup>-</sup>		
Phosphorous	H <sub>3</sub> PO <sub>4</sub> <sup>-</sup> and HPO <sub>4</sub> <sup>-2</sup>		
Potassium	K <sup>+</sup>		
Calcium	Ca <sup>+2</sup>		
Magnesium	Mg <sup>+2</sup>		
Sulphur	SO4-2		
Zinc	Zn <sup>+2</sup> and organic complex		
Iron	Fe <sup>+2</sup> , Fe <sup>+3</sup> and organic complex Fe		
Manganese	Mn <sup>+2</sup> and complex Mn		
Copper	Cu <sup>+2</sup> and organic complex Cu		
Boron	H <sub>3</sub> BO <sub>3</sub>		
Chlorine	Cl-		
Molybdenum	MoO4 <sup>-2</sup>		

Table 3 Plant Nutrients' available form (Ann McCauley et al., 2010)

Different chemical compounds exist that are capable of providing the plants with the nutrients in the above mentioned forms and are, thus, manufactured and used on a large scale all over the world.

#### **1.1.5 Classification of Commercial Fertilizers**

Synthetically prepared commercial fertilizers are classified on the basis of the nutrient they provide to the plant.

The commercial inorganic fertilizers are therefore classified as follows:

Nutrient	Fertilizer
Nitrogen	Ammonium sulphate
	Ammonium phosphate
	Potassium nitrate
	Calcium nitrate
	Urea
	Sulphur-coated urea
Phosphorous	Rock Phosphate
	Single superphosphate
	Triple superphosphate
	Monoammonium phosphate
	Diammonium phosphate
Potassium	Potassium chloride
	Potassium sulphate
	Potassium-magnesium sulphate
Calcium	Lime
	Dolomite
	Calcium sulphate (gypsum)
Magnesium	Magnesium sulphate (Epsom salt)
	Magnesium oxide
Sulphur	Elemental sulphur
Micronutrients	FeSO4.7
	FeEDTA
	ZnSO4.H2O
	ZnEDTA
	CIISOL SHIO

Table 4 Classification of Commercial Fertilizers (Ernest Kirkby et al., 2012)

#### **1.1.6 Multi-nutrient Fertilizer**

Many multi-nutrient fertilizers are available around the world in the market. Described below are the possible ranges of multi-nutrients of NP, NPK and PK fertilizers.

#### Table 5 Multi-nutrient Fertilizer (Ernest Kirkby et al., 2012)

Fertilizer type		%N	%(P2O5)	%(K2O)
NPK fertilizers		5-26	5-35	5-26
Ammonium Phosphates	MAP	16-18	42-48	
	DAP	11	52	
Nitro phosphates	NP	20-26	6-34	
PK Fertilizers	PK		6-30	6-30

Multinutrient fertilizers have the most notable advantages to the farmer, which are:

- Easy handling, transport and storage
- Easy application
- High nutrient content
- Even distribution in the field of nutrients
- Balanced fertilizing, i.e. nitrogen, phosphate and potassium available together
- High fertilizer efficiency.

#### 1.1.7 Fertilizer Grade

The type of fertilizers varies significantly according to the amounts and types of nutrients they carry. The right soil and plant fertilizer selection requires an understanding of the fertilizer label. Mixing is done by combining two or more fertilizer materials. There are significant differences in the proportion of the different nutrients contained in the mixed fertilizers. Mixed Fertilizer contains two or all three of the primary nutrients. Every fertilizer container on the market (sacks, buckets, etc.) has to be labelled with the nutrient content of a fertilizer guaranteed, by law. The information on the label and how the information is provided are specified by law and it guarantees the quantity of basic nutrients given by a series of three numbers, such as x-y-z, and is referred to as a "grade of fertilizer." A grade for fertilizer is a guaranteed minimum of primary nutrients, as a percentage of total nitrogen (N,  $P_2O_5$  and  $K_2O$ ). In this order, the grade of the fertilizer always indicates the nutrient content.

## Nitrogen (N) $\rightarrow$ x-y-z $\leftarrow$ Soluble Potash (K<sub>2</sub>O) $\uparrow$ Available Phosphate (P<sub>2</sub>O<sub>5</sub>)

The first number shows the nitrogen (N) pounds in every fertilizer of 100 pounds or kilograms. The middle figure refers to the amount of phosphate ( $P_2O_5$ ) and the last figure shows the pounds of potash ( $K_2O$ ) in each of 100 kilograms or kg of a certain fertilizer. You can specify the amount of each nutrient in the fertilizer by referring to the numbers printed in the bag. The labelling is the same across the country.

## 2: Fertilizer Industry of Pakistan

### **2.1 Introduction**

Pakistan's major economy is covered by the agricultural sector. Nearly 23.4 percent of Pakistan's economy is contributed to by the agricultural sector. It provides the citizens with livelihood and connectivity in the country. Around 70% of Pakistan's total population lives in rural areas. Nearly 45% of the workforce is active in farming. The provincial government of Pakistan focuses on increasing Pakistan's productivity in the agricultural sector, as a priority. Agriculture is a primary industry in which the downstream industry is supplied with raw material for production. Agriculture plays a fundamental role in reducing poverty and providing the country with job opportunities. Our main purpose is to diversify our agriculture from self-confidence to profitability.

Period	Growth rate	Share in GDP
1950s	1.76	47.70
1960s	5.12	40.68
1970s	2.32	34.50
1980s	4.10	27.62
1990s	4.54	25-34
2001-05	3.38	24.25
1951- 2005	3.55	34.36

 Table 6 Fertilizer Growth Rate (Nurul Islam et al., 2010)

The economic share of agriculture cannot go further, since many industries like textiles, sugar and fertilizer are also dependent on agriculture. It is believed that the low growth in agriculture in recent years in comparison with the total GDP was due to crop spoilage, adverse weather conditions and also to a relative increase in the share of the industrial sector.

In the future, however, the share of farming in GDP overall is expected to increase, as food demand increases.

Global consumption of fertilizers is closely linked to the country's macroeconomic growth. Similarly, in recent years Pakistan has experienced strong economic growth. GDP has grown by 7.55% in the last 3 years, with GDP growth expected to be positive for the future, which gives good predictions for the growth of use of fertilizers. The strong performance of the economy was accompanied by an increase in agricultural growth.

#### 2.2 Fertilizer consumption in Pakistan

Fertilizer consumption is low in Pakistan and the current consumption of fertilizer is about 160 kg per hectare. This is mainly due to the low yield per hectare of fertile land. Increased penetration of the fertilizer industry is expected. In future, economic growth will continue to increase this demand. Currently, the amount of fertilizer being consumed in the country exceeds the amount produced. Because of this, a huge amount of fertilizer needs to be imported resulting in the increase of food prices. More fertilizer needs to be manufactured in Pakistan if we wish to become a self-sufficient agricultural based economy.

Fertilizer use varies, based on the crops that are frequently harvested in the country. Based on the available data, urea is the most widely used fertilizer in Pakistan. DAP is the second most widely used fertilizer in the country.

Fertilizer consumption in Pakistan is determined by the geography, climate and availability of the different products (water availability), prices and timely availability. Therefore, the largest area for agriculture is Punjab, which uses the largest proportion of fertilizers followed by Sindh, NWFP and Baluchistan.

#### 2.3 Fertilizer Producers of Pakistan

There are policy changes and changes to the management of its domestic production, importation and distribution system in Pakistan's fertilizer sector. Sometimes the government controls the retail prices of fertilizer and engages in production, import and distribution of fertilizers directly or indirectly. There were reforms that began in 1986 and were completed by 1995; subsidies were eliminated on the sale prices of fertilizers, the reforms were dissolved, and the FID was disbanded by the Federal Ministry of Food and Agriculture, the Directorate for Fertilizer Imports. The government's engagement in the fertilizer sector was confined to policy planning and policy analysis (NFDC and FAO 2006). Public unit production was privatized in due course.

### 2.4 Historical Development in the Fertilizer Sector

Local producers of ammonium sulphate and mono super phosphate began production in the public sector in Pakistan in 1957. Engro Chemicals Pakistan limited was established in 1968. Dawood Hercules was established in 1971. The private enterprises owned their own distribution network. The government nationalized the marketing and distribution of fertilizer in 1973 and established provincial fertilizer distribution agencies. New fertilizer facilities were established; existing public fertilizer manufacturing units were operated and their production was marketed by the National Fertilizer Company (NFC), which is a public organization. In 1980, in Mirpur Mathelo in Sindh and in Haripur in KPK, NFC established two urea plants. NFC has been unable to meet rapidly growing fertilizer demand despite its efforts to increase domestic production. Accordingly, a urea facility was established by Fauji Fertilizer Company in Machhi Goth Sadiqabad in 1982.

The government subsidized the cost, but could not ensure that the supply was sufficient to meet demand and farmers were prepared to pay higher prices to satisfy their crop requirements. The government was then pressurized to phase out the subsidies while providing regular supplies by donors and domestic agents. In 1985, the government decided to deregulate and abolish the margin of fixed marketing and subsidies for nitrogen fertilizers, including urea, calcium ammonium and ammonium sulphate. However, the price of phosphate and potash fertilizers remained controlled and subsidized.

### **2.5 Market Players**

Four large companies dominate the market in Pakistan. A new company's entry is unlikely to reduce the concentration significantly. Fusions and acquisitions are also unlikely within the industry, so manufacturers are expected to expand.

The following fertilizer companies are operating in Pakistan:

- Fauji Fertilizer Company Limited (FFC)
- Fauji Fertilizer Bin Qasim (FFBL)
- Engro Fertilizer Limited
- Fatima Fertilizer Company Limited
- Pak-Arab Fertilizer Limited.
- Lyallpur Chemicals & Fertilizers Limited
- Pak-American Fertilizer Limited

#### 2.5.1 Diammonium Phosphate Producer

DAP is a fertilizer that is important for the seeding of new crops in the country. FFBL and Fatima Fertilizer Company Limited are the major producers of DAP in Pakistan. Currently, the DAP market share of FFBL is 30 per cent, with import supplies representing 70 per cent of the market.

Table 7 Market share of FFBL

	Amount of Fertilizer	Market Share
	(TTPA)	
Fauji Fertilizer, Bin Qasim	401	30%
Imports	1066	70%

FFBL production costs are very high compared to other fertilizer manufacturers. Because FFBL was the first producer of both Urea and DAP fertilizers, DAP production process is more expensive. The main part of production costs is natural gas as a raw material, 80% is gas as feedstock and 20% is fuel stock gas.

## 2.6 Market dynamics (Demand-Supply situation)

There is a difference in structure of the fertilizer industry in Pakistan. A single player entry or exit may influence pricing. The four largest companies are the companies that control pricing. Subsidies from the government prevent these companies from exploiting their market power by concluding price fixing contracts.

Excessive demand is one of the current industrial conditions. The national supply capability currently stands at 5.8 MTPA (million tons per year) and the demand at 6.8 MTPA. The Trade Corporation of Pakistan is satisfying the excess demand currently in place and imports foreign fertilizer at a subsidized rate to domestic producers and distributors. TCP only imports urea, and local producers sell urea at low rates (between two and three percent), whilst DAP is imported.

## **3: Raw Materials**

## **3.1 Introduction**

Two nutrients are provided to plants by DAP. Both of these nutrients are bound together as a single compound and come from two chemical species:

- Ammonia; provides the Nitrogen
- Phosphoric acid; provides the Phosphorus

Besides these main raw materials, water is also added to the reaction. Water and air are used as utilities, for controlling temperature and moisture content. A description of the main raw materials is provided below.

## 3.2 Ammonia

Ammonia, with the formula NH<sub>3</sub>, is a chemical compound. Ammonia occurs as a gas with a distinctive pungent smell at 20 °C and 1 atm pressure. Its principal uses are fertilizers, polymers and explosives. As a fertilizer, ammonia can be used directly, without additional chemical processing, by creating a solution using irrigating water. This permits the continuous growth without crop rotation of nitrogen-dependent plants like the maize (which leads to poor soils however). Ammonia is very suitable as a cooling agent because it easily liquefies under pressure and has been used in almost all cooling units prior to Freon's advent. The main raw materials for most military explosives include ammonia, soda ash, nitric acid and nylon, plastics, dyes, rubber, cooling and many more.



Figure 2 Ammonia Molecule (Wikipedia)

Urine fermentation by bacteria leads to the ammonia solution; therefore, in Classical Antiquity, fermented urine was used for washing cloth and garments, removing hair from hides to tanning, serving as a mordant in diet cloth, and removing iron rust.

With regard to Muslim alchemists, as early as the eighth century, the Persian Arab chemist Jabir ibn Hayyān first mentioned Ammonia. Similarly, Albertus Magnus referred to salmonic Ammonia in their Ammonia form. Dyers in the middle Ages also used it to alter the colours of vegetable dyes in the form of fermented urine. Gas ammonia was first isolized with calcined magnesium (Magnesium Oxide) by Joseph Black in 1756. In 1909, the Haber-Bosch process was discovered for production of ammonia from nitrogen. It was used in Germany for the first time on an industrial scale during the First World War. Previously, Hydrogen was produced through the electrolysis of water. As the steel industry came into being in the 20th century, Ammonia became a coking coal by-product.

### **3.3 Phosphoric Acid**

The world produces millions of tons of phosphoric acid, H<sub>3</sub>PO<sub>4</sub>, annually. Most of the acid (approximately 80%) is used for the manufacture of agricultural fertilizers and the remaining is used for detergent additives (approximately 10%), cleansers and insecticides, and livestock feed additives. It is also used in the dental, albumin derivatives preparation and textile and sugar industry. It acts as a fruit-like, acidic taste in foodstuffs. The crystalline solid (melting point 42.35 °C, or 108.2 °F) is pure phosphoric acid; the coloured sorbic acid is a liquid which is less concentrated. The raw acid consists of phosphate rock while white phosphorus consists of acid of high quality. Sulphuric acid is added to rock phosphates as the commercial method of preparation.

Pure phosphoric acid, anhydrous, is a white solid that falls to a viscous liquid at 42.35°C. Phosphoric acid acts as a triprotic acid with three ionizing atoms in an aqueous solution. It is a stronger acid, but weaker than acetic acid and hydrochloric acid.

Phosphoric acid salts may be produced by replacing one, two or three hydrogen ions. For example, a single mole of phosphoric acid with a mole of sodium hydroxide can form NaH<sub>2</sub>PO<sub>4</sub>, a dihydrogen phosphate of sodium.

Phosphoric acid is used in the manufacture of detergents for water softeners. Calcium and magnesium ions from hard water are removed by water softeners. If this ion is not removed, the soap and insoluble deposits are reacted and adhere to washing machines. Sodium

triphosphate, Na<sub>5</sub>P<sub>3</sub>O<sub>10</sub>, is the most frequently used phosphorous compound in solid detergent mixtures. Sodium triphosphates as water softeners bind with calcium and magnesium ions, which form soluble, complex or chelated chemical species. These complexes prevent the reaction of calcium and magnesium ions to soap and deposits. (Reda Hussein et al., 2017)



Figure 3 Phosphoric Acid Molecule (Wikipedia)

#### 3.3.1 History

Phosphoric acid for the commercial fertilizer sector was produced in Germany in 1870, and in the United States from 1890, for a short period of time. In 1900, about 12 companies made phosphoric acid for use in concentrated superphosphates by wet processes in Europe. Unfortunately, ore had to be imported from the U.S. This has made the production of acid by wet process operations uneconomic for several years.

In 1897, USA developed a process to make use of bones of Chicago stockyards for use in the production of calcium phosphate. The Staffer Chemical Society is considered to be a pioneer in the production of the wet process phosphoric acid. In 1912, the process was converted and food-grade acid was produced with phosphate rock. All phosphoric acid was produced by batch process up to that time. The first continuous process was initiated by Dorr Company in 1915.

In 1932, the production of phosphoric acid was investigated by precipitate hemihydrates of calcium sulphate instead of the usual method for forming calcium sulphate dehydrate. Phosphoric acid is 40 to 50% and  $P_2O_5$  follows this route. The marketing of this method was prevented by filtration problems.

Davison Chemicals studied in the late 1940s and early 1950s, a process of production of between 50% and 54%  $P_2O_5$  products in which a hydrate of calcium sulphate was formed, but this was never sold.

Chemical formula	H <sub>3</sub> PO <sub>4</sub>	
Molar mass	97.994 g·mol-1	
Appearance	White, deliquescent, solid viscous liquid	
Odor	Odorless	
Density	030 g·cm−3 (25 °C)	
Melting point	42.3 °C (anhydrous) 29.3 °C (hemihydrate)	
Boiling point	158 °C (316 °F; 431 K) decomposes	
Solubility in water	392.2 g/100 g (-16.3 °C) 369.4 g/100 mL (0.5 °C) 446 g/100 mL (14.95 °C)	

Table 8 Properties of Phosphoric Acid (Reda Hussein et al., 2017)

## 4: Di-ammonium Phosphate (DAP)

### **4.1 Introduction**

The most widely used phosphorus fertilizer in the world is Diammonium phosphate (DAP). It is made of two common constituents in fertilizer industries and is a very good choice for agricultural and other industries because of its relatively high nutrient level and excellent physical properties. For plant nutrition, DAP fertilizer is an excellent P and N source. It is very soluble and therefore it quickly dissolves in the soil, releasing phosphates and ammonium which are available to plants. The alkaline pH around the dissolving granule is a notable property of DAP.

When DAP granules release ammonium, the nearest plant and plant roots can be damaged. This potential damage happens more often when the pH in the soil is above 7 and is often associated with the dissolving DAP granule. Users need to avoid putting high DAP concentrations near germinating seeds in order to prevent such damage.

DAP ammonium is an excellent N source that is gradually converted into a nitrate of soil bacteria and is subsequently lowered in PH. Consequently, a temporary effect is the increase in soil pH of DAP granules. This initial increase in the pH of soil adjacent to DAP can influence the phosphate and soil organic matter microsite reactions

There are two main ammonium phosphate types: mono-ammonium phosphate and Diammonium phosphate. Mono-ammonium phosphate is produced with ammonia, centrifuge, and drying in a rotational dryer by reaction with phosphoric acid. In order to prevent loss of ammonia, Diammonium phosphate requires a two-stage reactor system. At the end of the reaction, a granulation process is followed by a rotary dryer heated by a fuelfurnace.

Diammonium phosphate (DAP) is a series of water-soluble phosphate salts, which can be produced when ammonia is reacted with phosphoric acid. The following expression and equation shows that solid Diammonium phosphate exerts the dissociation pressure of Ammonia. (E. C. Houston, L. D. et al., 1955)

 $(NH_4)_2HPO_4 \rightarrow NH_3 + NH_4H_2PO_4; \log P_{mmHg} = -3063/T+175 \log T+3.3$ 

Here,

P<sub>mmHg</sub> is the resultant dissociation pressure of ammonia
T is the absolute temperature in Kelvin
At 100℃ DAP dissociates at 5 mmHg approximately

As a fertilizer, DAP is widely used as plant food. The soil pH temporarily increases, but the soil is more acidic in the long term than it had been when ammonium was nitrified. It is incompatible in a high pH environment, as the ammonium ion of the substances is most likely to be converted into ammonia.

The ammonium phosphates resulting in this process are then screened. The particles under their size are recycled back to the granulation procedure while the oversized particles are first grinded before being recycled. The granules of the fertilizer are covered by special material after screening in order to control the dissolving process in the soil when used.



Figure 4 DAP Granules (Rahul Dayal et al., 2013)

Table 9 DAP Properties (Rahul Dayal et al., 2013)

Chemical Formula	(NH4)2HPO4
Molar mass	132.07 g/mole
Density	1.619 g/cm <sup>3</sup>
Melting point	155°C decomp.
Composition	18%N, 46%P2O5 (20%P)
Water solubility (20°C)	588 g/L
Solution pH	7.5 to 8

#### 4.2 Uses of DAP

- Source of Nitrogen and Phosphorous. It has high solubility and therefore quickly dissolves in soil to release phosphate and ammonia that are available to plants. The alkaline pH around the dissolving granule is a significant property of DAP.
- As a result of the release of ammonium from dissolving Diammonium Phosphate granules, volatile ammonia can be hazardous to seedlings and plant roots. This potential damage is most common when the pH of the soil exceeds 7, a condition normally associated with the dissolving DAP granule. In order to prevent seed damage, care should be taken to prevent high levels of DAP in close proximity to germanised seeds.
- The ammonium present in Diammonium phosphate is an excellent N source and will then be gradually converted to nitrate by soil bacteria, which will lead to a drop in pH. This initial increase in soil pH adjacent to Diammonium phosphate can influence phosphate and organic soil micro-site reactions.
- DAP may also be used for irrigation because it is fully water soluble and can be used for liquid or suspension fertilizer production.
- DAP is used as a fire retardant in many applications. For example, in advance of the fire, a combination of DAP and other ingredients can be spread to avoid burning of the forest. It reduces the material's combustion temperature, reduces the maximum loss of weight and increases residue or char production. These are important effects in combating wildfires, as the temperature of pyrolysis and the amount of charcoal formed are reduced and the amount of fuel available are reduced. It is the largest component of a number of popular commercial

incendiary products. After the hazard of fire has passed it becomes a source of nutrients.

- DAP is used as fluxes for soldering tin, copper, zinc and metal in different industrial processes, such as metal finishing.
- It is commonly added to wine for yeast fermentation, mead brewing and cheese to promote the cultivation of cheese.
- It is also used as an addictive in certain cigarette brands allegedly as an enhancer of Nicotine and to prevent matches.
- DAP is used in sugar purification, and for controlling the precipitation of colloid dyes on wool, alkaline and acid insoluble.

### 4.3 Importance of DAP

The method gives us higher plant feed concentration while favouring the agromanic as well as physical properties. It can be also utilized for applications directly. DAP is non-poisonous, non-explosive and non-inflammable. As a base material, it is extremely important for blending and dry mixing for the production of other grades. In addition to that, it also provides us with high potassium chloride, ammonium nitrate and sulphate and urea concentrations. It is also very economical and gives a very sound method so as to fix ammonia in the solid form (Almost two times as much as mono-ammonium phosphate). There is also an increase in solubility in water of the products through contribution of phosphorous penta-oxide. DAP also provides some added advantages while being used for the production of N-P-K grades (wet mix).

- Decreased amount of reactants otherwise required in ammoniates.
- Lower moisture contents in formulation: which decrease drying requirement
- May contributes to higher production rates.
- Formulation using DAP is often less expensive than other conventional formulation.
- Improves physical stability.
- Lessens tendency of product caking in storage.

### 4.4 Manufacturing of DAP

These are the following processes which are available for the manufacturing of DAP.

- Tennessee Valley Authority (TVA) atmospheric saturator process.
- Tennessee Valley Authority (TVA) vacuum crystallizer process.
- DAP as by-product of coke-oven plants.
- Dorr-Oliver Granulation process
- TVA Pre neutralizer process
- TVA Pipe cross reactor process
- SA Cros Technology
- ERT Espendisa Technology
- AZF Process

(Kenneth J.et al., 1999)

#### 4.4.1 Atmospheric saturator process (TVA)

Diammonium phosphate (DAP) was manufactured in a pilot plant using an atmosphere saturator process in the end of 1990s from anhydrous ammonia and electric furnace phosphoric acid. This process comprises of thin, tubular crystal aggregates bound with a fine crystal film. The product was free of impurities but contained 5% mono-ammonium phosphate.

The DAP was produced in a continuous, single stage atmospheric saturator by continuously feeding gaseous ammonia and strong electric furnace phosphoric acid (75% to 85% H<sub>3</sub>PO<sub>4</sub>), at an ammonia-phosphate ratio of 2:1 into a saturated solution of ammonium phosphate at about 60°C to 70°C. DAP crystallized out of the solution and was recovered by settling, centrifuging, washing and drying. The ammonia and phosphoric acid mole ratio in the solution was maintained as low as practical without precipitating mono ammonium phosphate. This mole ratio was about 1.6, which corresponded to a pH of 5.8 to 6.0. The DAP crystals precipitate from this solution, were wet with a film of relatively more acidic mother liquor, a portion of which was displaced by water during washing, and the reminder crystallized in the surface of the DAP crystal. The pilot plant produced about 40 pounds of DAP per hour per square foot saturator cross section.

When it is compared with other fertilizer salts, the Diammonium phosphate (DAP) product was comparatively stable, resisted compatible physical characteristics and on exposure to  $30^{\circ}$ C and relative humidity up to 80%, moisture absorption and ammonia loss were negligible.

#### 4.4.1.1 Drawbacks

Attempt to use wet process phosphoric acid for production of DAP by the atmosphere saturator process was unsatisfactory because of the formation of precipitates that regarded filtration.

#### 4.4.2 Vacuum crystallizer process (TVA)

This process was started in the early 1950s when Tennessee Valley Authority (TVA) produced Diammonium phosphate (DAP) on demonstration scale in a vacuum crystallizer. The process previously developed in a pilot plant consisted of feeding into the vacuum crystallizer of gaseous ammonia and phosphoric acid. It was a crystalline coarse material containing 21% nitrogen and 53% phosphorous oxide.

#### 4.4.2.1 Drawbacks

Attempt to use wet process phosphoric acid for production of DAP by the atmosphere saturator process were unsatisfactory because of the formation of precipitates that retarded filtration. Same results were reported in vacuum crystallizer.

#### 4.4.3 DAP as by-product of coke-oven plants

The coke-oven by-product manufacturers began producing Diammonium phosphate (DAP) in the plants that were originally designed for ammonium sulphate production. Modifications were made in ammonium sulphate and Diammonium phosphate (DAP) to meet the market demand. The Colorado Fuel & Iron Company used a single stage saturated system. In 1956, Ford Motor Company, steel division, at Dearborn, Michigan began operation of a plant having two stages absorption for by-product of Diammonium phosphate (DAP).

As driven off in the paralytic distillation of coal, called coking contain approximately 1% by volume of ammonia. Therefore, an absorption type scrubber was used to strip the ammonia from the effluent gas before releasing the gas to the atmosphere.

Two types of the ammonia saturator or scrubber were most commonly used for the absorption of the ammonia. In the Coppers or single stage tube type saturator, the gas was dispersed into
the acid liquid phase by means of a submerged Spurger ring or cracker pipes. Secondary clean-up of ammonia from the gas was accomplished by sprays in the vessel above the tube.

#### 4.4.3.1 Drawbacks

The crystal formation and separation processes were essentially the same for Diammonium phosphate (DAP) as for ammonium sulphate. However, because of complication in settling resulting from a difference in specific gravity between Diammonium phosphate crystals and the liquor at the operating pH and temperature, approximately twice as much settling tank area and a non-turbulent feed were necessary for successful settling of DAP product crystal.

#### **4.4.4 Dorr-Olive Granulation process**

Phosphoric acid and ammonia are reacted in two agitated vessels, in series, under carefully controlled condition. Neutralization is carried to mono ammonium phosphate or slightly above in the first stage. The ammonia, carried off in the water evaporated, is not appreciable in this range and approximate characteristics of solution is 80% of DAP is effected in a second stage where the composition of solution is maintained approximately at or near the triple point. In early systems, final ammoniation was done in a third point.

Since minimum solubility is obtained at the triple point, slurry can be obtained with minimum water content. The ammonium phosphate slurry then overflows to the blunger. The slurry is distributed by a saw tooth weir arrangement or by a pipe spray header. Particle growth is by layering of the slurry on bouncing seed particles, and to some extent by agglomeration of the fine particles. A blunger normally produced spherical, smooth, comparatively uniform granules.

To obtain the desired smooth round granules, it is necessary to circulate the particles many times through the blunger and to apply a relatively thin coat each time. When granulating by agglomeration and coating, the slurry in the blunger will tend to stick together or agglomerate the undersized granules, broken oversized fragment and added dry salt crystals. For this purpose fine salts are required. The two types of granule formation, layering and agglomeration, represents only the extreme and in practice both will be formed in varying degree depending upon the conditions chosen, liquid phase, and content with respect to size, type , and quantity of solid particles. (Dorr Oliver Eimco et al., 2007)



Figure 5 Dorr-Olive Granulator (Dorr Oliver Eimco et al., 2007)

#### 4.4.4.1 Drawbacks

As in the process it is necessary to circulate the particle many time through the blunger and to apply relatively thin coat each time, if a thick slurry is used as an attempt to made to apply a thick coat at each passage through the blunger, the granules will tend to stick together small particles and become rough and irregular.

#### 4.4.5 TVA Pre neutralizer process

With a single reaction tank, the anhydrous ammonia is normally sparged at, from 2 to 4 locations around the circumference in the lower portion of the tank. Either liquid or vaporized ammonia is used; more heat is available for evaporation in the reactor. Liquid anhydrous Ammonia is most commonly used as much as heat and additional equipment is normally required to vaporize the ammonia. The phosphoric acid is normally fed from source as 54% P<sub>2</sub>O<sub>5</sub> content. This varies in this range depending on the source of rock used in making the wet-process phosphoric acid, which in turn varies the impurities entering with the acid. The mole ratio of ammonia to phosphoric acid is normally held between 1.4 and 1.45. This area will normally give the maximum solubility for the slurry. The temperature of the slurry is normally maintained at about 115.5°C. For operating control, the mole ratio is determined by periodically checking the pH of slurry samples. At a mole ratio of 1.45 some free ammonia is lost in the steam from the pre neutralizer. Therefore, the exhaust from the pre

neutralizer is scrubbed with acid. One scrubber is generally used for the pre neutralizer and ammonia-granulator.

#### 4.4.5.1 Drawbacks

- Low conversion rates.
- Large reactor volume.
- High energy consumption.

## 4.5 Chemistry of the process

The reactions for the manufacture of Diammonium Phosphate take place in two steps. The first step is the neutralization of acid and base. The reaction is exothermic. During the first step a mixture of ammonium phosphate is found among mono-ammonium phosphate (MAP) formation is greater.

 $NH_3 + H_3PO_4 \longrightarrow (NH_4) H_2PO_4$ 

During the second step, further ammoniation of MAP takes place which yields DAP. This reaction is performed with liquid anhydrous ammonia at atmospheric pressure.

This reaction is also exothermic.

 $(NH_4)H_2PO_4 + NH_3 \longrightarrow (NH_4)_2HPO_4$ 

## 4.6 Process Flow Diagram



## **5: Material Balance**

## **5.1 Introduction**

When designing a new process or analysing an existing one, material balance is an important first step. In the solving process engineering problems, they are almost always necessary for all other calculations.

Material balance is nothing more than the application of the law of mass conservation, according to which mass cannot be created nor destroyed. For example, a single ton of naphtha reactor and two ton of petrol or gas or anything else could not be supplied. Only 1 ton of total input is given to total output of only one ton, i.e.

Total mass input= Total mass output.

An accounting for material is a material balance. Substantive balances are therefore often compared with current accounts balancing. In the industry, the mass flow rates of various streams enter or leave chemical or physical processes are calculated.

## 5.2 The general mass balance equation

Supposing propane is an input and output stream component of the following continuous process unit, these flow rates are calculated and measured.



If leakage does not occur and measuring properly, propane is generated, consumed or accumulated within the unit, which may be responsible for this difference. This general equilibrium can be drawn up and applied to the overall mass of this material or to any molecular or atomic species within the process for any material entering or leaving a process system.

Input +	generation	– output –	consumption	=	accumulation
(enters	(produced	(leaves	(consumed		(buildup
through	within	through	within		within
system	system	system	system)		system)
boundaries)	boundaries)	boundaries)			

Depending on the process involved, the general balance equation can be simplified. For instance, the accumulation term for a continuous process in the steady state is zero by definition.

The above equation can be simplified as

```
Input + Generation = Output + Consumption
```

As no chemical reaction occurs, the production and consumption conditions become zero, and the equilibrium for the physical process of steady-state is simply reduced to:

*Input* = *Output* 

Following are the results of mass balance calculations over the various parts of the plant.

## 5.3 Reactor-1

For more understanding of the location of the reactor 1 in the process, refer to the Section B of the PFD.

Law of conversation of Mass

Mass in = Mass out = 6,292,816 kg

#### **Mass of Inlet**

$H_2SO_4$	270078.1 Kg
$Ca_3(PO_4)_2$	2696491.1 Kg
H <sub>2</sub> O	918265 Kg
CaF <sub>2</sub>	119422 Kg

#### **Mass of Outlet**

Gypsum	4392364 Kg
H <sub>3</sub> PO <sub>4</sub>	1666378 Kg
NaF	126040 Kg

H <sub>2</sub> O	108034 Kg
------------------	-----------

## 5.4 Scrubber

Mass in = 9002.868 Kg mole

HF	3000.956 Kg mole
H <sub>2</sub> 0	3000.956 Kg mole
NaOH	3000.956 Kg mole

Mass Out = 9002.868 Kg mole

NaF	3000.956 kg mole
H <sub>2</sub> 0	6001.912 Kg Mole

## **5.5 Settler Tank**

25% H<sub>3</sub>PO<sub>4</sub> Recycled = 4251.22 Kg mole

75%  $H_3PO_4$  to Rector 2 = 12753.68 Kg mole

## 5.6 Reactor- 2

For more understanding of the location of the reactor 2 in the process, refer to the Section B of the PFD.

Law of conversation of Mass

Mass in = Mass out = 1684200 kg

#### **Mass of Inlet**

H <sub>3</sub> PO <sub>4</sub>	1249784 Kg
NH <sub>3</sub>	434416 Kg

Mass of Outlet

Diammonium phosphate (DAP)	1684200 kg

## 5.7 Granulator

Mass in = 12481.58 Kgmole

Mass out = 12481.58 kg mole

From Rector 2 to Granulator	12116 kg mole
From Cyclone to Granulator	365.58 Kg mol

## 5.8 Dryer

Mass In = Mass Out

Mass In = 12481.58 Kg mole

Mass Out

To Screen	12358 Kg mole
To cyclone	123.58 Kg mole

## 5.9 Cyclone

Mass In = Mass Out

Mass in = 365.58 kg mole

Mass out = 365.58 kg mole

## 5.10 Storage and Packaging

DAP = 12116 Kg mole

## **6: Energy Balance**

## **6.1 Introduction**

In the calculations of chemical engineering problems, the concept of energy conservation is expressed through the energy balance equation. An energy balance is essential to solve many issues.

The increasing cost of energy has caused the industries to examine the means of reducing energy balances which are used in examining the different process steps, over the whole process and from the raw material to the finish of the product, throughout the total production system. Energy conservation is stated by The First Law of Thermodynamics.

While it is not possible to create or destroy energy, it can be transformed from one form to another. Inner energy stored in molecular form can be transformed into kinetic energy; potential energy can be converted either into kinetic energy or to internal energy. Transfer of energy may occur via heat flow, mass transport (convection is otherwise known as mass transport), or through work performance. The overall energy balance process can be expressed as

Accumulation of Energy = Input of Energy into – Output of Energy from

## 6.2 Reactor-1

$$\operatorname{Ca}_{3}(\operatorname{PO}_{4})_{2} + 3\operatorname{H}_{2}\operatorname{SO}_{4} + 6\operatorname{H}_{2}\operatorname{O} \rightarrow 3\operatorname{Ca}_{3}\operatorname{SO}_{4} \cdot 2\operatorname{H}_{2}\operatorname{O} + 2\operatorname{H}_{3}\operatorname{PO}_{4}$$

We can calculate heat release or required during the reaction by this equation:

$$Q=dH=dH_{reaction} + (\Delta H_{f_p} - \Delta H_{f_{reactants}})$$

 $dH_{reaction} = \Delta H_{f_{Products}} - \Delta H_{f_{reactants}}$ 

 $dH_{reaction} = 1996202.36 \text{ KJ}$ 

 $Q = -5.69 \times 10^{10} \text{ KJ}$ 

Cooling water required =  $^{Q}/_{Cp\Delta T}$ 

Cooling water required =  $2.7 \times 10^8$  Kg

#### 6.3 Scrubber

 $HF + H_2O + NaOH \rightarrow NaF + 2H_2O$ 

Q=dH= $dH_{reaction} + (\Delta H_{fp} - \Delta H_{freactants}) = -7.09 \times 10^8 \text{ KJ}$ 

## 6.4 Reactor-2

 $2NH_3 + H_3PO_4 \rightarrow (NH_4)_2HPO_4$ 

 $Q=dH=dH_{reaction} + (\Delta H_{fp} - \Delta H_{freactants})$ 

 $dH_{reaction} = \Delta H_{f_{Products}} - \Delta H_{f_{reactants}}$ 

 $dH_{reaction} = (-1566910) - ((-46.2 \times 2) - 1271.66)$ 

 $dH_{reaction}$ = -202.85 KJ/mole = -2457730600 KJ

 $Q = -2.32 \times 10^9 \text{ KJ}$ 

Cooling water required =  $Q/Cp\Delta T = \frac{-2.32 \times 10^9 \text{ KJ}}{4.18 \times 35}$ 

Cooling water required =  $1.59 \times 10^7$  kg

### 6.5 Cyclone

 $W = Q = mCp\Delta T$ 

 $Q = 4531353 \times 1.009 \times (82 - 45)$ 

Q = 169169013 kJ/Day

### 6.6 Dryer

 $m_p Cp \Delta T = m_A Cp \Delta T$ 

 $m_A = 233722 \ Kg$ 

## 6.7 Settler Tank

No  $\Delta T$  therefore

 $Q_{in} = Q_{out}$ 

## 7: Equipment Design

## 7.1 Design Procedure

- Choose a suitable type
- Collect all kinetics and thermodynamic data on desired reaction
- Collect physical property data required for design
- Calculate the parameters of reactor (Volume, length & diameter)
- Mechanical Design of the reactor (Jack Richardson et al., (2005)

### 7.2 Reactor-1

$$\begin{array}{rcl} Ca_3(PO_4)_2+3H_2SO_4+6H_2O & \rightarrow & 3Ca_3SO_4.\,2H_2O\ +2H_3PO_4\\ \\ CaF_2+H_2SO_4\rightarrow 2HF+CaSO_4 \end{array}$$

P = 1 atm

T = 75 degree Celsius

Reactor 1 is CSTR where Ground rock is reacting with sulphuric acid to produce Gypsum and phosphoric acid.

$$\tau = \frac{V}{V^{\circ}}$$

Residence time =  $\tau = 2$  hours

$$V^{\circ} = m/\rho = 155 m^3/hr$$

$$V = 2 \times 155 = 310 \ m^3$$

#### **Dimensions of CSTR**

Height = 12m Diameter = 3 m

#### 7.3 Reactor 2

$$2NH_3 + H_3PO_4 \rightarrow (NH_4)_2HPO_4$$

Rector is PFR where Ammonia and Phosphoric acid react to produce Diammonium Phosphate (DAP)

Rate of reaction =  $-r_A = K [C_{A\circ}(1 - X_A)]^n$ 

Where K= 0.123  $(Kg \ mole / m^3)^{\frac{1}{2}}$ n =  $\frac{1}{2}$ 

By inserting above values in equation we calculated the following values.

 $C_{A\circ} = 4.22 \ Kg \ mole/m^3$ 

 $F_{A\circ} = 0.29 \ Kg \ mole/m^3$ 

Design Equation  $= \frac{V}{F_{A\circ}} = \int_0^{0.98} \frac{X_A}{-r_A} dX_A$ 

**Dimensions of PFR** 

Length = 13m

Diameter = 0.44m

### 7.4 Granulator

Volumetric Flow Rate =  $0.7129 m^3/hr$ 

Residence Time = 50 minutes

Volume = Volumetric Flow Rate x Residence Time

 $= 35 m^3 (40 m^3 is maximum)$ 

$$\frac{L}{D} = 2$$

#### **Dimensions of Granulator**

Length 5.6 m Diameter = 2.8m

#### 7.4.1 Granular Growth:

$$\frac{dp}{dp^\circ} = \left(1 + \frac{1}{R}\right)^{1/2}$$

 $\frac{dp}{dp^{\circ}} = 1.2$ 

R = 10.5 rpm

Inclination from Ground =  $1/8^{\text{th}}$  of diameter

= 0.3 m

## 7.5 Dryer

Initial moisture content = 5.6% = 0.0056

Final Moisture Content = 1% = 0.001

$$q_T = U_a V \Delta T$$

Flow rate of Solid = 68648 Kg/hr

Flow rate of Air = 315 Kg/ hr

 $q_T = 194391 \text{ KJ/hr}$  $U_a = \text{Overall heat Transfer coefficent}$  $\Delta T = \text{Temperature difference}$ 

$$U_a = K^- G n / D$$
  
 $U_a = 200(n_f - 1)G^{0.67} / D$ 

 $U_a \Delta T = 6300 \ w/m^2$ Area = 56.84 m<sup>2</sup> area = 8.5m<sup>2</sup>

h = 4D  
$$(\pi \times \frac{8.5}{4.5})^{\frac{1}{2}} = d$$

#### **Dimensions of Dryer**

Diameter = 0.77m

Height = 3 mVolume =  $\frac{\pi d^2 h}{4}$  = 2  $m^3$ 

## 7.6 Scrubber

Liquid to gas molar ratio =  $L_m/G_m = 0.045$ 

$$\Delta P = \frac{L}{G} \times K \times \rho_g \times v$$
$$\frac{C_i}{C_o} = 3.47 \times \Delta P^{-1.43}$$

Where

 $\Delta P = 2.5$ 

L/G = 15

Liquid P = 200 Psig

Cut diameter =  $6\mu m$ 

 $G_{velocity} = 10$  ft. / sec

$$h = \frac{3}{2}d$$

$$Q = 33 \text{ m}^3/\text{s} = 1160 \text{ ft}^3/\text{sec}$$

Area =  $1.4 \text{ m}^2$ 

Diameter = d = 2.1 m

### 7.7 Screen

Power = 4 hp = 3 kW

Mass flow rate = 65.174 ton/ hr

Area =  $2.5 \text{ m}^2$ 

We know

$$L/W = 2.5$$

#### **Dimensions of screen**

W = 1.5 mL = 3.88 m

## 7.8 Crusher

Mass flow rate = 23.75 ton /hr

 $W_i = 9.92$ 

$$P = 0.3162 \times 9.92 \left( \left( \frac{1}{(50 \times 10^{-6})^{\frac{1}{2}}} \right) - \left( \frac{1}{(120 \times 10^{-6})^{\frac{1}{2}}} \right) \right)$$

P = 25.78 KW

## 7.9 Settler Tank

$$Volumetric flow rate = \frac{1666310.2 \ Kg/day}{1880 \ kg/m^3}$$

$$\frac{1666310.2 \ Kg/day}{1880 \ kg/m^3} = 886 \ m^3/day \times 1.5 \ hr$$

Volume =  $55 \text{ m}^3$ 

h = 4d

**Dimensions of settler tank** 

D = 1.5 m

H = 6 m

## 7.10 Cyclone

$$B_c = D/4$$
  
 $D_e = D_c/106$   
 $H_c = D_c/2$   
 $S_c = 5Dc/8$   
 $J_c = Dc/2$   
 $Lc = 1*D_c$   
 $Z_c = 2*D_c$ 

Particle size = 80 – 100 micrometre Efficiency = 98% Air Flow rate=  $28 \text{ m}^3/\text{sec}$ Velocity= 54 m/secArea of duct =  $0.5 \text{ m}^2$ 

$$area = \frac{\pi \times d_c^2}{4}$$

Bc	0.2 m
He	0.4 m
De	0.4875 m
Sc	0.4875 m
Le	0.78 m
Zc	1.56 m
Jc	0.39 m

## 8: Costing

## 8.1 Introduction

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

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

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

## 8.2 Capital Investment

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

- 1. Preliminary (approximate) predictions; typically 30% precision, used to make rough choices among the design alternatives in initial feasibility studies. The cost data and design details are limited.
- 2. The estimates for approval (budgeting), typically 10-15% accuracy. These are used to authorize funds to carry out the design to the extent to which a detailed and accurate assessment is possible. Approval may also include money to cover cancelation charges for any lengthy delivery equipment ordered at this design stage in order to avoid project delays. Such an estimate could be used in a contracting organisation, with a large contingency factor, to get the tender price. However, it would normally be necessary, if time allowed, to achieve accuracy of around 5% and to make a more detailed estimate where an enterprise has cost data and experience.

 Detailed (quotation), accuracy of 5-10% used for project cost control and fixed price contract estimates. These are based on the process design, firm equipment quotations and a detailed division and cost estimation (or almost complete).

The estimate costs rise from around 0.1% of the total cost to 30% accuracy, to approximately 2% for a detailed estimate with +-5% accuracy.

## 8.3 Fixed Capital Investment

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

It covers the costs of:

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

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

It includes the cost of:

- 1. Launching.
- 2. Initial Catalyst Loads.
- 3. In this process, raw materials and intermediates.
- 4. Inventories of finished product.

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

The working capital of a single product can range from 5% of fixed capital for a simple process with little or no finished product storage, to 30% for a process which produces a wide variety of product classes for an advanced market, such as synthetic fibres. A typical figure of 15% of fixed capital for petrochemical plants is estimated. (Jack Richardson et al., 2005)

## 8.4 Cost Estimates for the Proposed Design

Costing was done with reference from Coulson Richardson's Chemical Engineering Design Volume 6th. Following are the graphs and figures used in cost estimation.

#### Graph







#### Table 10 Cost Estimation Table (Jack Richardson et al., 2005)

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

#### This table helps in cost estimation of other equipment. Table 7. 2: Cost of Equipment

 Table 11 Cost Estimation Table (Jack Richardson et al., 2005)

Variable costs <ol> <li>Raw materials</li> <li>Miscellaneous materials</li> <li>Utilities</li> <li>Shipping and packaging</li> </ol>	<i>Typical values</i> from flow-sheets 10 per cent of item (5) from flow-sheet usually negligible
Sub-total	Α
<i>Fixed costs</i> 5. Maintenance 6. Operating labour 7. Laboratory costs 8. Supervision 9. Plant overheads 10. Capital charges 11. Insurance 12. Local taxes 13. Royalties	5–10 per cent of fixed capital from manning estimates 20–23 per cent of 6 20 per cent of item (6) 50 per cent of item (6) 10 per cent of the fixed capital 1 per cent of the fixed capital 2 per cent of the fixed capital 1 per cent of the fixed capital
Sub-total	В
Direct production costs A + 13. Sales expense 14. General overheads 15. Research and development	- B 20–30 per cent of the direct production cost
Sub-total	C
Annual production $cost = A + B + C$	=
Production cost £/kg =	Annual production cost Annual production rate

We used tables, figures and the following formula for costing of different equipment:

 $Ce = CS^n$ 

Where

 $C_e = Cost of equipment$  C=Constant S=Capacityn = index

#### 8.4.1 Storage Tank for H<sub>3</sub>PO<sub>4</sub>

 $Ce = CS^n$ 

n = 1.55

 $S = 540 \ m^3$ 

C = 4600

Total cost = \$146410

#### 8.4.2 Storage Tank for water

 $Ce = CS^n$ 

n = 0.55

 $S = 200 \ m^3$ 

C = 4600

Total cost = \$84785

#### 8.4.3 Storage Tank for NH3

Bare vessel cost = \$46000

 $S=440\ m^3$ 

 $m_f\!=2$ 

 $P_{\rm f} = 1$ 

Total cost = \$120000

#### 8.4.4 Reactor -1 cost

Bare vessel cost = \$45000

 $m_f\!=\!2$ 

 $P_f = 1$ 

Total cost = 110000

#### 8.4.5 Reactor -2 cost

Bare vessel cost = \$27000

 $m_{\rm f}\!=\!2$ 

 $P_{\rm f}=1$ 

Total cost = \$56000

#### 8.4.6 Granulator

 $Ce = CS^n$ 

n = 0.45

 $S = 35 m^3$ 

C = 31000

Total cost = \$153529

#### 8.4.7 Scrubber

Bare vessel cost = \$5500

 $m_f\!=2$ 

 $P_{\rm f} = 1.6$ 

Packing cost = \$25000

Total cost = \$30500

Similarly rest of costs of equipment were calculated from tables and figures.

### Table 12 Cost of equiment

Equipment	Cost (In Dollars)
H <sub>3</sub> PO <sub>4</sub>	1164100
H <sub>2</sub> O	84785
Ammonia Tank	240000
Sulphuric Acid Tank	1445000
Rock Tank	75000
Scrubber	30,500
Reactor 1	110000
Filter	54678
Settler	32108
Reactor 2	86000
Granulator	153529
Dryer	118388
Screen	130000
Crusher	56619
Cyclone	42000
Storage Tank	45,560
Pumps	192000
Blower	17500
Conveyor Belts	101000
Cooling Tower	7203050

#### Table 13 PCE cost

Cost(In Dollars)PCE11,433,817COST INDEX 2019197.8COST INDEX 2014140.2COST IN 201916131305FACTORS SUM3.15PPC66944917FUTHER FACTORS2.4FIXED CAPITAL1.61E+08WORKING CAPITAL32133560TOTAL CAPITAL1.93E+08		
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WORKING CAPITAL32133560TOTAL CAPITAL1.93E+08INVESTMENT1.93E+08	FIXED CAPITAL	1.61E+08
TOTAL CAPITAL 1.93E+08 INVESTMENT	WORKING CAPITAL	32133560
	TOTAL CAPITAL INVESTMENT	1.93E+08

#### Table 14 Variable and fixed cost

VARIABLE COSTS	
RAW MATERIAL	17671440
MIS	1606678.008
UTILITIES	3000000
SHIPPING	NIL
TOTAL	22278118.01
FIXED COST	
MAINTENANCE	16066780.08
LABOUR	316000
LAB	63200
SUPERVISION	1000000
PLANT OVERHEAD	158000
CAPITAL CHARGES	9640068.045
INSURANCE	1606678.008
LOCAL TAXES	3213356.015
ROYALTIES	16066780.08
TOTAL	48130862.22

Direct Production Cost = Fixed Cost + Variable Cost = \$70408980

Sales expense + General Overheads + Research and Development = 17602245.06

Annual Production Cost = \$8801125

Payback Period = Sales/Annual Production Cost

Cost of 1 bag = \$19

Sales = \$221920000/year

Payback Period = 2.5 years

## 9: Simulation

## 9.1 Introduction

In order to model and simulate phosphoric acid and di-ammonium phosphate production, ASPEN Plus software was used. Chemical reactions, filtration, flash separation, granulation, drying and screening were considered as the main processes units. For a large number of components, ASPEN PLUS stores physical property parameters in several databases. A property method is a collection of methods, models and the physical properties of all streams used by ASPEN PLUS to calculate the chemical and thermodynamic equilibrium. In this work, ASPEN PLUS was used to calculate all the physical properties required with the SOLIDS and Electrolyte NRTL options.

## 9.2 Procedure

So we simulated the process through the following procedure:

- 1. The properties of sokoto phosphate rock to characterize the raw material.
- 2. The component list was made.
- 3. The property package selected was ELECNRTL (used for process chemistry where the fluids are treated as ions) and SOLIDS (used for the operations like crushing, screening, filtration etc.).
- 4. After adding the components and selecting the package, the property table is automatically generated.
- 5. Simulation window is entered and all the unit operations are added and specifications are done. (Reactor-1 is added and given the stoichiometry and similarly all the other unit operations are added and specified)
- 6. Simulation was run and the results were noted and matched with the manual results.



Figure 8 Simulation

## 9.3 Conclusion

Our manual calculations were very similar to those calculated using the ASPEN HYSYS Plus model.

# **10: Instrumentation and Process Control**

### **10.1 Introduction**

A control system is a system for controlling the output required. A block diagram of a control system can be seen in the picture below.



A single block represents the control system here. As the output is controlled through different inputs, this name has been given to the control system. With some mechanism, we will change this input. We are going to study in detail in the next section the open loop and shutdown control systems the blocks use within the control system and how to change these inputs to get the requested answer.

Examples are Traffic lights control system & washing machine

An example is the traffic lights control system. Here, the input signal sequence of the controller is applied and the output is one of the three lights on for some time. The other two lights are off during this time. The light times can be determined on and off based on the transport study at a particular junction. The output is therefore controlled by the input signal. So the system for control of traffic lights works on a schedule basis.

## **10.2 Control Systems' Classification**

We can classify the control systems in the following ways, based on certain parameters.

#### **10.2.1** Continuous time and Discrete-time Control Systems

Continuous Time Control Systems and the Discrete Time Control System can be classified based on the signal type used.

All the signals are continuous in time in continuous time control systems. However, there are one or more discrete time signals in discrete time control systems.

#### 10.2.2 SISO and MIMO Control Systems

The control systems can be classified as SISO and MIMO controllers based on the number of inputs and outputs available.

SISO (Single Input and Single Output) control systems have one input and one output. Whereas, MIMO (Multiple Inputs and Multiple Outputs) control systems have more than one input and more than one output.

#### 10.2.3 Open Loop and Closed Loop Control Systems

Open loop control systems and closed loop control systems can be classified based on the feedback path.

Output is not returned to the input in open loop control systems. The control is therefore independent of the output desired.

The following figure shows the block diagram of the open loop control system.



Figure 9 Open Loop Feedback system

#### 10.2.3.1 Open Loop

Here, the controller receives an input and produces an actuating signal. This signal is an input to a controlled plant or process. The plant generates a controlled output. An example of an open loop control system is a traffic light control system we discussed earlier.

The output is returned to the input in closed loop control systems. The control measurement therefore depends on the desired output.

The following figure shows the block diagram of negative feedback closed loop control system.



Figure 10 Closed Loop Feedback system

#### 10.2.3.2 Closed Loop

The error detector gives a signal of error that differentiates the input and the feedback signal. This feedback signal is obtained by considering the output of the overall system as an input to this block from the block (feedback elements). The error signal is used as an input for the controller instead of the direct input.

The controller therefore generates an actuating signal that controls the plant. The output of the control system is automatically adjusted in this combination until the requested response is received. Automatic control systems are thus also referred to as the closed loop control systems. A closed-loop control system is an example of traffic lights control system with an input sensor. (Cooper D. J. et al., 2006)

In the following table, some differences are shown between the open and the closed loop systems.

Table 15 Difference between open an	nd closed control systems
-------------------------------------	---------------------------

Open Loop Control Systems	Closed Loop Control Systems
Control action is independent of the desired	Control action is dependent of the desired output.
output.	
Feedback path is not present.	Feedback path is present.
These are also called as non-feedback control	These are also called as feedback control systems.
systems.	

Easy to design.	Difficult to design.
These are economical.	These are costlier.
Inaccurate.	Accurate.

It is known as feedback if any of the output or part of the output is returned to the input side and used as part of the input system. Feedback plays a key role in improving control systems performance. Let us discuss in this chapter the feedback and feedback types.

## **10.3 Types of Feedback**

There are two types of feedback

- Positive feedback
- Negative feedback

#### **10.3.1 Positive Feedback**

The positive feedback includes the input reference, R(s) and the output feedback. The diagram below shows the positive feedback control system block diagram.



```
Figure 11 Postive feedback
```

Later chapters discuss the concept of transfer function. Consider the transfer function of the positive feedback control system for the time being,

 $T=G_1-G_H$  (Equation 1)

Where,

- T is the overall transfer function or positive feedback system gain.
- G is the open loop gain, the frequency function.
- $\circ~$  H is a feedback path gain that is a frequency function.

#### **10.3.2 Negative Feedback**

Negative feedback reduces input, R(s) and system output error. The following figure shows a block diagram of the negative feedback system.



Figure 12 Negative Feedback

Transfer function of negative feedback control system is,

 $T=G_1+G_H$  (Equation 2)

Where,

- T shall be the transfer function or the overall negative feedback management system gain.
- $\circ$  G, which is the frequency function, is an open loop gain.
- $\circ~$  H is the feedback path gain that is the frequency function.

# 11: Hazard and Operability (HAZOP) Study

## **11.1 Introduction**

HAZOP is a structured and systematic technique used for system review and risk management. HAZOP is often used to identify potential hazards and problems of operability in a system that can lead to non-compliance products. HAZOP is based on a theory which assumes that deviations in design or operating intentions lead to risk events. Sets of guide words' are a systemic list of deviation perspectives to facilitate the identifying of such deviations. This approach is a unique feature of the HAZOP methodology that helps to stimulate team member's imagination to explore potential deviations.

As a risk assessment tool, HAZOP is often described as:

- Brainstorm technology
- A qualitative tool for risk evaluation
- An inductive tool for assessing risk, which means it is a "bottom-up" risk identification approach, where the success is based on the ability of SMEs to predict differences based on experience from the past and general expertise. When describing the HAZOP methodology, the following definitions are useful:

#### 11.1.1 Hazard

It is the possible harmful source. Design or operating intention deviations can represent or pose a danger. The HAZOP studies focus on hazards and note that a single hazard can lead to many forms of harm.

#### 11.1.2 Harm

It involves physical damage or damage to the health or the environment of people. Danger is the result of a hazard that can take many forms: safety for patients or users, employee security, business risks, regulatory risks, environmental risks, etc.

#### 11.1.3 Risk

It is the combination of the likelihood of harm and its severity. In strict terms, "risk" is not always explicitly identified in HAZOP trials, as no identification of probability or severity of harm is required under the core methodology (also referred to as rating). Risk evaluation teams may however choose to measure such factors to quantify and prioritize risk further where necessary.

## **11.2 Objectives**

The aim of this study is to provide the participant with a structured risk identification scheme, risk assessment, measures to control inacceptable risks, and control measures to ensure they are effective and have no new hazards introduced.

In every aspect of our lives, danger surrounds us. There are risks in air, food we eat and in the places we live.

It is a danger to nearly every aspect of life. In order to survive, we are all constantly identifying, assessing, monitoring and reviewing risks.

## **11.3 Applications**

It has an industrial workplace perspective; however, in domestic, recreational or voluntary situations, the general principles for risk management can be applied equally.

In figurative terms, we can round out any equipment, process, construction or organization to identify hazards, evaluate risks, and select suitable control measures through the risk management process.

HAZOP is best suited for the evaluation of hazards in installations, equipment and processes. HAZOP can evaluate systems in several ways:

- Evaluation of the capacity of the system to fulfil user requirements and safety standards
- Identification of system weaknesses
- Environmental assessment to ensure that the system is properly located, supported, serviced, contained, etc.
- Assessment of Engineered checks (e.g. automation), operational sequences, procedural checks (e.g. human interactions) etc.
- Evaluation of different modes of operation start up, standby, normal operation, stable & unstable states, shutdown norm, emergency shutdown, etc.

## **11.4 HAZOP Pros and Cons**

#### 11.4.1 HAZOP Pros

- Systematic testing is the HAZOP process.
- A multidisciplinary study is a team approach to HAZOP.
- Operational experience is used by the HAZOP team.
- The process includes both operational and safety aspects.
- The identified problems can be resolved. HAZOP considers operational procedures.
- HAZOPs cover human mistakes.
- The independent-leading HAZOP study.
- Results of the HAZOP study are registered.
- The process is easily studied and carried out by team members.
- No extensive technical expertise for technical formulation is required for a HAZOP.
- It provides rigor to focus on the system components and risks as a systemic process.
- The HAZOP process is a multi-purpose team effort.
- HAZOP analysis is supported by commercial software.

#### 11.4.2 HAZOP Cons

- A HAZOP concentrates instead of combining events on individual events.
- The HAZOP focus is on word guides, so that certain risks that are not linked to word guides are not overlooked. Training, particularly for the facilitator, is essential for optimum results.
- Typically, HAZOPs are very time consuming and therefore costly.


Figure 13 HAZOP Steps (Jack Richardson et al., 2005)

# 11.5 Reactor- 2

### **11.5.1 Temperature cooling water HAZOP**

Table 16 Temperature and cooling water HAZOP of Reactor 2

Guide Word	Deviation	Causes	Consequences	Action
NO	No Cooling	Cooling water valve not working	Reactor temperature increases	Install high temperature alarm
REVERSE	Reverse flow of cooling water	Backward flow of cooling water	Less cooling, runaway reaction possible	Install check valve
MORE	More cooling flow	Control value failure, human error	Reactor cools down	Instruct proper SOPs to the operators
AS WELL AS	Reactor Product in coils	Coils damaged	Off-spec product	Check maintenance procedure and schedules.
OTHER THAN	Another material besides cooling water	Water source contaminated	May be cooling ineffective and effect on the reason	If less cooling, TAH will detect, isolate water source. Back up water source.

#### 11.5.2 Reactor-2 Flow HAZOP

Guide Word	Deviation	Causes	Consequences	Action Required
NO	NO FLOW	Excess ammonia in reactor and release to work area.	<ul> <li>Valve fails A closed</li> <li>Pipe ruptures</li> <li>Phosphoric acid supply exhausted</li> </ul>	Automatic closure of valve B on loss of flow from phosphoric acid supply.
LESS	LESS FLOW	Excess reactor ammonia and discharge to the workplace. With quantitative supply reduction in the quantity released. A toxicity vs flow reduction member to calculate it.	<ul> <li>Valve A partially closed</li> <li>Partial plug or leak in pipe.</li> </ul>	A toxicity / flow reduction team member to calculate. Toxicity vs. flow measurement set point. Automatic valve closure for phosphoric acid supply flow reduction.
MORE	MORE FLOW	Overproduction degrades of phosphoric acid. No danger to the area of work.		

#### Table 17 Flow HAZOP of Reactor 2

# 11.6 Reactor 1

### 11.6.1 Reactor 1 Cooling water HAZOP

#### Table 18 Cooling water HAZOP of Reactor 1

Guide Word	Deviation	Causes	Consequences	Action Required
NO	No Cooling	Cooling water valve not working	Reactor temperature increases. Cooling water valve	Install high temperature alarm
REVERSE	Reverse flow of cooling water	Backward flow of cooling water	Less cooling, runaway reaction possible	Install check valve
MORE	More cooling flow	Control value failure, human error	Reactor cools down	Instruct proper SOPs to the operators
AS WELL AS	Reactor Product in reactor walls	Reactor walls damaged	Off-spec product	Check maintenance procedure and schedules.
OTHER THAN	Another material besides cooling water	Water source contaminated	May be cooling ineffective and effect on the reason	If less cooling, TAH will detect, isolate water source. Back up water source.

# Conclusion

Our DAP plant will equip Fatima Fertilizer with the ability to become the largest DAP producer in Pakistan from locally mined rock phosphate. This project will make Fatima the first fertilizer industry in Pakistan to do so. Furthermore, our process is highly efficient while also ensuring that we utilize our raw materials sensibly by employing recycling at various stages. The successful installation of this plant will give Fatima fertilizers an edge over its competitors. By utilizing the local resources, we also are cutting down the price of the DAP fertilizer by \$4 and if the plant is set up, we will be able to achieve our break even in 2.5 years which is a very suitable time period.

To summarize, I would like to state that if the plant is set up, not only will we be utilizing our local resources and minimizing the imports but we would also be selling the DAP fertilizer at a cheaper rate and will also be able to gain profit before the beginning of 3<sup>rd</sup> year of operation of the plant.

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