

# REDUCTION OF AMMONICAL NITROGEN FROM SURFACE CONDENSATE IN ANP

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**Abstract:-** Ammonium nitrate is a large volume industrial inorganic chemical, used mostly as a high grade (34% N) straight nitrogen fertilizer containing both ammonium and nitrate forms of nitrogen and is one of the most commercially important ammonium compound. The raw material used for production of ammonium nitrate are vapor ammonia and 60% nitric acid (HNO<sub>3</sub>). Ammonia and nitric acid react to produce 65% concentrated Ammonia. Ammonia is highly stable and is decomposed into hydrogen and nitrogen overheating. During the process of production of ammonium nitrate some ammonia is lost from the AN solution during evaporation in the form of ammonical nitrogen (NH<sub>3</sub>-N). The emissions of ammonium nitrate and ammonia to water and to atmosphere are the major problems of the existing technology and steam and electricity are consumed for treatment of waste water (process condensate, process steam and air from prilling towers or granulators) in order to find opportunities for better utilization and to prevent the loss of raw material and there is a need to reduce the excess ammonical nitrogen in surface condensate and make maximum and economical utilization.

**Keywords:** : Ammonium Nitrate, Efficiency, Raw material, Environmental impact, surface condensate .

## INTRODUCTION

**AIM:** Reduction of ammonical nitrogen from surface condensate in ANP (Ammonium nitro phosphate Plant)

### ➤ OBJECTIVES:

1. In this work, we are focusing to calculate the amount of ammonical nitrogen loss per ton of ammonia to analyse benefit to cost ratio and to determine the need of modifications for reduction of ammonical nitrogen in surface condensate.
2. To study the overall process description to understand the various reasons for ammonical nitrogen loss and the solutions for modification.
3. To suggest the possible control measures for source modification for reduction of ammonical nitrogen in surface condensate.

### ➤ RESEARCH BACKGROUND:

During the production of ammonium nitrate, some ammonia is lost from the solution during evaporation in the form of ammonical nitrogen. In order to prevent the loss of raw materials, it is necessary to trap the excess ammonical nitrogen in surface condensate. In this work we are focusing to calculate the amount of NH<sub>3</sub>-N (ammonical nitrogen) in condensate slurry and what system we should provide in order to reduce the percentage of ammonical nitrogen in condensate slurry. The experiments are planned to calculate the amount of surface condensate as well as the amount of nitrogen loss with its root-cause analysis to suggest proper solutions. The scheme is proposed for nutrient recovery by considering all the parameters like temperature, flash steam amount and cost estimation.

## I. EXPERIMENTS

### 1) Determination of NH<sub>3</sub>-N in slurry:

2.5 gm. of sample in 250 ml volumetric flask is taken then 20ml of 1 ml. HCl and 25 ml of 40% CaCl<sub>2</sub> and methyl Red indicator is added. Then 4 M NaOH till color changes to yellow added. Made it to 250 ml. Filtered the solution after shaking. Took formaldehyde 1:1 diluted in which Thymol blue indicator is added and neutralized it with Std. NaOH (0.1 N) till color changed to light green. Then 25 ml of filtered aliquot taken and neutralized it with NaOH. Then added 20 ml of neutral Formaldehyde and kept it for 20 mins after well shaking. To complete the reaction the above solution titrated against Std. NaOH (0.1 N) using orthocresolphthalein indicator till color changed to light violet[2] and Br reading is taken

$$\% \text{NH}_3\text{N} = (\text{BR} * \text{Normality of NaOH} * 1.401 * 250 / 25) / \text{Wt. of sample}$$

$$= (\text{BR} * \text{Normality of NaOH} * 1.401) / \text{Wt. of sample}$$

### 2) To calculate Ammonical Nitrogen (NH<sub>3</sub>-N) loss :

Basis: 1 m<sup>3</sup> dil. AN feed = 65% AN concentration 1 m<sup>3</sup> = 1.25 To = 1250 kg

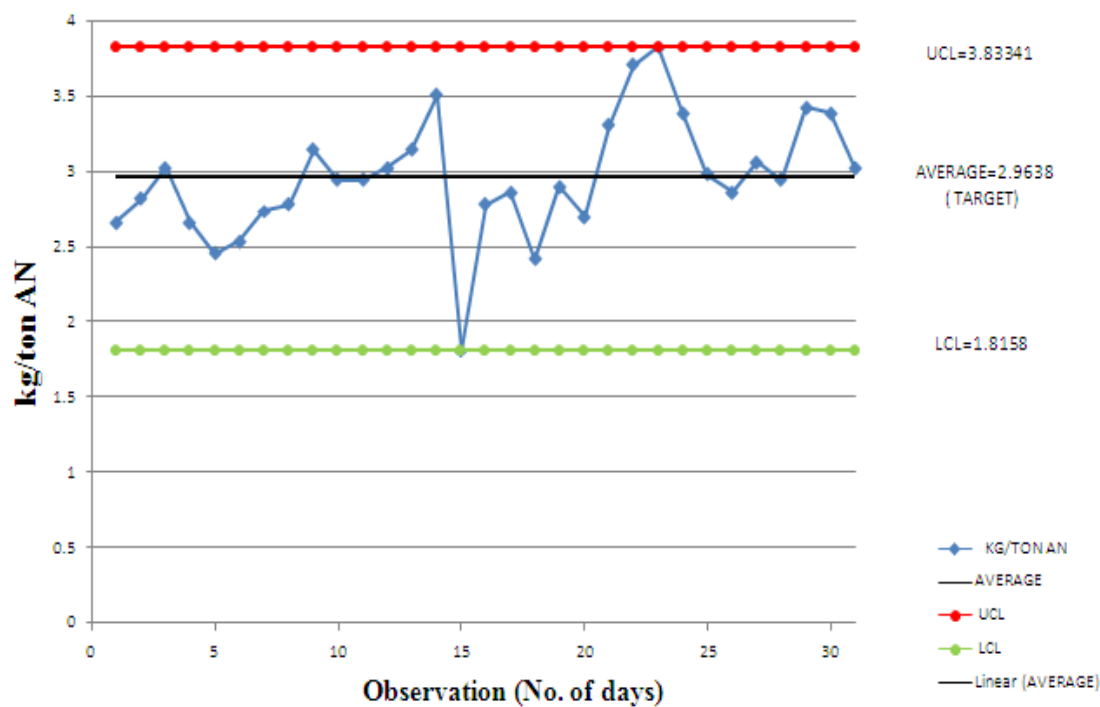
Specific gravity = 1.25

1 m<sup>3</sup> dil AN = 65(1.25)/100 ton of AN = 0.8125 ton/day

Dil. AN production = Dil. AN feed \* 0.8125 Around 27% moisture to surface condensate

Nitrogen loss = Average NH<sub>3</sub>-N \* 17/14 \* surface condensate / 100.

## 3) Graphical representation Kg of nitrogen loss per ton of AN feed vs No. of days



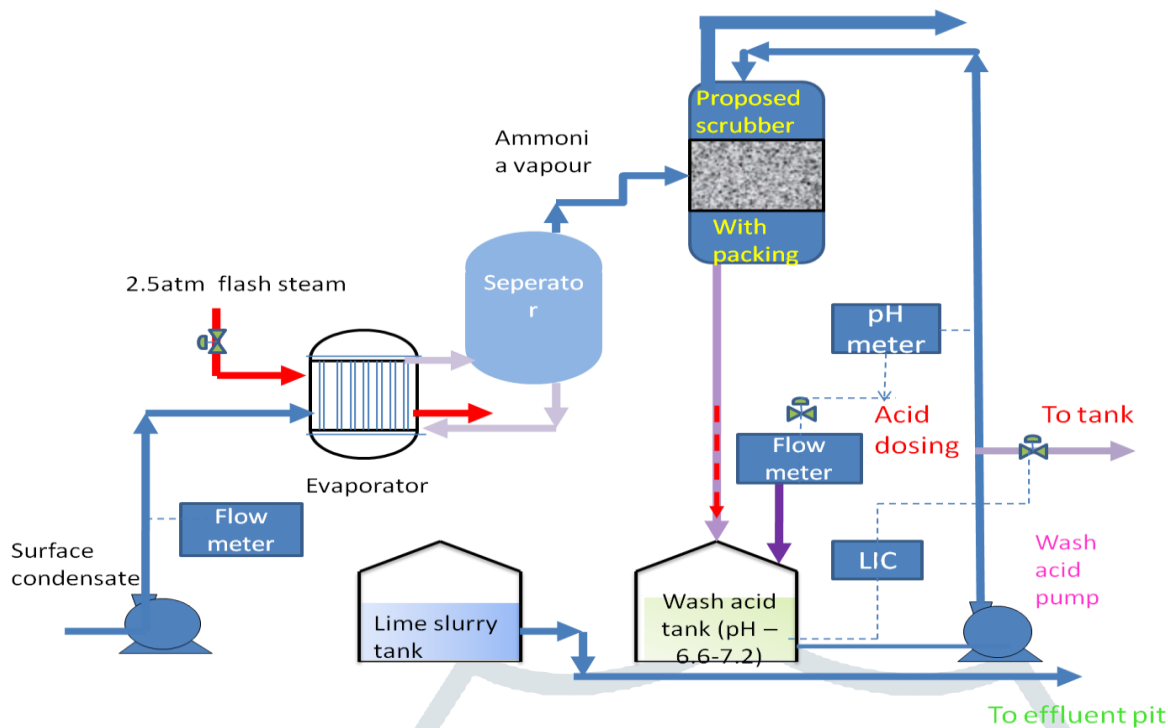
- 4) **WHY WHY ANALYSIS:** The higher amount of ammoniacal nitrogen in condensate arises due to some reasons -1) Higher pH of dil AN solution due higher pH in reactor or acid and ammonia flow variation . Another reason is no cascade control. 2)No provision for ammonia recovery from condensate due to its unavailability. 3)Failure of demister pad and fluctuation in vacuum separator due to load variation.Load is maintained as per tankers availability. 4) Error in online pH measurement and deposition of AN crystals on electrode.
- 5) **Solution and implementation:** Provision for cascade control for acid and ammonia should take place and for that ratio controller is provided. Maintain reactor pH between 7.0-7.2 and ratio as per stoichiometry.Daily cleaning and calibration with buffer solution of electrode.Ammoniacal condensate to be sent to R&D Nutrients recovery plants and fabrication of pipeline from ANP to nutrient recovery plant.[2]

## II. RESULT AND DISCUSSION:

NH<sub>3</sub>-N in condensate slurry was 0.8%. After heating, 80o C, NH<sub>3</sub>-N in condensate slurry was 0.2. It is observed that after heating the AN evaporator ammoniacal condensate slurry the amount of NH<sub>3</sub>-N has been reduced. Hence to reduce loss we can use multistage evaporator system. The effluent quality & quantity with only AN melt production in ANP plant without mixing sulphala (S-15)effluent Quantity of effluent at 350 -450 MTPD AN Load :: 320-500 M Quantity of NH<sub>3</sub>.N = 1300-1500 PPM (average) Quantity of No<sub>3</sub>.N= 300-600 PPM (average) Above said quality is as per ETP /Plant analysis reports. CC lab results shows much lesser NH<sub>3</sub> .N & NO<sub>3</sub>.N content in ANP effluent .Above range has been achieved after improvement schemes in AN melt such as :Reactor vapor NH<sub>3</sub> to Nitric acid control .Reactor Ph brought down to 7.0-7.2 from original 7.8-8.0 (further low Ph not possible with the existing two stage process technology of AN melt production .(Further Ph reduction in reactor is unsafe as AN can decompose at low Ph and higher temperature during concentration process.) Recycle of scrubbed water back to scrubbers. Use of STP reject water back to scrubber . Installation of new effluent pit with 3 compartment (settings/residence) etc. An evaporator ammoniacal condensate with 0.6 to 0.8% (6000to 8000 ppm) NH<sub>3</sub>.N is the main contributor for high NH<sub>3</sub>.N in effluent ,Which was earlier being used in PAP plant for gypsum washing/pH correction, this condensate m<sup>3</sup>/hr(80-100 m<sup>3</sup>/day max) is discharged to effluent.[1]

### ➤ PROPOSED SCHEME TO RECOVER NH<sub>3</sub>.N

Ammoniacal Condensate from evaporator surface condenser can be fed directly to redundant first stage AN evaporator heater using existing pump which is used to pump same to effluent pit via lime slurry(mixing) tank .Flash (captive) steam generated from AN evaporation units.(New& Old)steam condensate flash vessel (2.5kg/cm<sup>2</sup>)can be used to heat ammoniacal condensate in heater upto 80-90 deg under slight vacuum .As per lab trial, we have seen that dissolved free NH<sub>3</sub> liberates and NH<sub>3</sub>.N in ammoniacal condensate reduces from 0.8 to 0.1 % at this temperature. A redundant packed scrubber shall be installed at 15 mtr and vapor NH<sub>3</sub> released from heater can be scrubbed there using dilute nitric acid (60%) . Existing wash acid tank and pump ,earlier used for reactor regeneration using 60% nitric acid can be used for holding dilute nitric acid for circulation in scrubber.



Proposed scrubber vent can be connected to existing scrubbing system of AN melt plant. Ammonical condensate from heater separator outlet containing water only can be discharged to effluent. scrubbed  $\text{NH}_3$  gets converted to ammonium nitrate and concentration of dilute nitric acid drops. Spent dilute nitric acid shall be recycled to existing AN reactor periodically after testing its concentration and top up of dilute nitric acid to wash acid tank shall be done.

Modification should be done in-house using redundant evaporator, wash acid tank, scrubber with packing and pumps. These includes cost of piping fabrication and hook up and cost of modification of redundant scrubber and installation cost.

#### ➤ RESULT:

Intangible benefits due to reduction in ammonia in surface condensate according to the proposed solutions include Improved effluent quality and work environment. Responsibility and accountability improved. Striving for goal achievement increased. Improvement in scope of thinking. Team is developed to tackle various problems. Employees are now aware about loss of ammonical nitrogen. Employees are now aware about loss of ammonical nitrogen.

#### ➤ DISCUSSION:

Provision of cascade controller for controlling the ratio of acid and ammonia has to be done in order to lower the loss of nutrients. Maintaining the reactor pH up to 6.6-7.2 is important. As increase in acidity increases decomposition of ammonium nitrate into ammonium ions. Daily cleaning and calibration of pH meter has to be done. The plant must run on consistent basis only. Ammonia recovery using proposed scheme has shown sustainable profit and reduce the loss of nutrients.

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