Urea Product Quality

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Abstract **- In this article includes many studies and investigation reporting on the effect of various parameters to the prills quality like crushing strength, size distribution, abrasion and impact resistance, humidity factors urea moisture absorption, vacuum studies etc. The Nitrogen, moisture, Prill strength &Biuret contents and the size distribution of prilled urea are important factors of determining urea quality. High temperature of prilled product is a common in most of urea plant in India. In some plants in India the temperature of prills as high 80^{o}C in hot summer days at high load. Our plant temperature of prills is also 65-70^{0}C.This result in poor strength dust formation, increase in caking tendency. Granulated urea has definite advantage over prilled urea but expensive to produce till recently. We have installed BFC for line-1 & 2.A modification also done in Urea line –I plant in month of March 2018 to solve foaming problem in waste water section (distillation tower) and this modification proven and beneficial.**

Key words - Prills, urea, prilling tower, moisture, humidity, vacuum, Biuret, polymers.

INTRODUCTION

The National Fertilizers Ltd. Vijaipur have two ammonia plant M/S. Haldor Topsoe Technology, Denmark capacity 1750 & 1864 TPD for Line-I & line-II respectively and four urea plant of M/S. Saipem ammonia stripping process, Italy. The Line-I plant installed in 1988 and that of line –II in 1997. Urea-II plant was commissioned in 1st quarter of year 1997 and commercial production was declared on 31.03.1997. The capacity of Urea-I urea –II is 3030 & 3231 TPD respectively. The raw material used includes natural gas, water and power. Three Numbers Captive power plant of capacity 17 X 3 MW are used in this complex. The line –I plant installed casale's designed pre-concentrator in vacuum section while in line-II installed M/S. Saipem designed pre-concentrator, which is actual pre-concentrator, this is the 3rd stage of vacuum having designed pressure 0.34 ata. A different vacuum degree is maintained between the evaporator combination E-14 A/B (1st stage vacuum heat exchangers), 11-MV-6(1st stage vacuum separator) and that of E-15(2nd stage vacuum heat exchanger).MV-7(2nd stage vacuum separator) to allow the maximum water evaporation. Vacuum evaporation avoids the use of high temperatures, which would increase biuret content In the Urea. With evaporators running at different pressures, the solution passes from one to the other without the use of pumps. The separators must work empty to reduce the holding time of the solution. Outlet vapours from vacuum separators are condensed and recovered in wastewater tank and sent to waste water section where the ammonia recovered by distillation tower and urea hydrolysed with hydrolyser at the pressure of 34 bar and Temperature 233^{0}C The energy saving reported from both the pre-concentrator are 0.123 G.cal/ton of urea. The prills temperature of Line-I goes to maximum 75^{0}C while in urea line-II is 66^{0}C.Solex bulk flow cooler are installed to cool down prills temperature. Over a peak summer we take a BFC in line for one to two months. This prills temperature difference due the prilling tower height, the P.T. free fall height in Line-I is 72 meters while in line-II is 80 meters. The total height of P.T. in line –I is 96 meters while in line-II is 106 meters. The diameter of prilling tower in Line-I is 28 meter while in line-II it is 26 meter because the large amounts of air was bypassing in prilling tower resulted prills temperature high.

Detail of Urea product quality

The variable parameter in urea product quality are Nitrogen, Moisture, Biuret, Prill strength & size distribution. Nitrogen depends upon moisture, Biuret, Free ammonia & other impurities like corrosion product, dissolving urea dust grease etc. E.G. if Biuret in Urea product is 1.0 % then Theoretical Nitrogen in Urea is 46.66 %. In 1.0 % Biuret Nitrogen is 0.407 %, Moisture 0.5%, Nitrogen Nil, free Ammonia in prills is 120 ppm, Nitrogen is 0.0098%, Practically Nitrogen found in prills is 46.30 to 46.40 %. Performance of vacuum evaporation section is crucial as it affects the urea product quality. Less vacuum causes high moisture, high free ammonia and less crushing strength of urea prills the poor performance of vacuum evaporation section. A number of factors determine how much moisture urea will absorb during processing. These include particle shape and size, and varying chemical composition and purity levels. External factors including climatic humidity and how urea is stored and for how long also play a big part. Whatever the reasons, the costs of clearing adhesive lumps of caked urea from clogged conveyors can be substantial. Urea crystals formed when moisture absorbent dust particles or fines dry out can clog and damage many traditional conveying systems.

Moisture in Product
Depends upon two factors

1. Internal. i.e. from process
2. External i.e. ambient

Infernal causes include poor vacuum condition process upset due to any reason urea recovery of lean solution. External causes include ambient condition, humidity, CRH of prills. Sometimes-atmospheric relative humidity is much more than CRH (Critical relative humidity) of Urea causing moisture adsorption by prilled urea. Under such conditions, air quantity to the prilling tower is to be decreased. CRH is a function of vapor pressure of saturated urea solution. The mechanical consistence of the formed lump will depend on the cycling frequency and on the quality of the urea lumps of hard prills properly treated for the anticaking and of under size distribution will disintegrate usually without structural damage. The mechanical resistance depends on surface structure and particle strength. Dust and fines normally arise during handling from Water absorption as shown in the figure-1. Molten Urea enters the rotating prilling bucket, which distributes it in the prilling tower where the air flowing in counter current causes its crystallization. Prilled urea is collected at tower bottom and sent to storage by means of a belt conveyor system. Its function is to cool the melted urea. Urea coming out of the prilling bucket in the form of drops falls into the tower and by means of an air current is cooled and solidified. Air rises through the tower by natural draft entering through a series of windows located at the bottom and, after cooling the urea dust particles (spm) vented to the atmosphere. Prilled urea collects on conveyor belt 11-MT -1(Product belt), which discharges it to hopper with a screen to separate possible lumps that may have formed on the walls of tower 11-ME-6(Prilling tower). The product coming out of the hopper is sent to the warehouse or direct loading in bagging plant. A belt to the dissolving tank 11-V-4(lumps dissolving tank) sends the rough product, which has separated on the hopper screen. A detail experiment done on dated 15/03/2018 with variable vacuum conditions of pre concentrator, 1st stage vacuum & 2nd stage vacuum and monitor moisture in urea product detail reading tabulated in table No.-3.

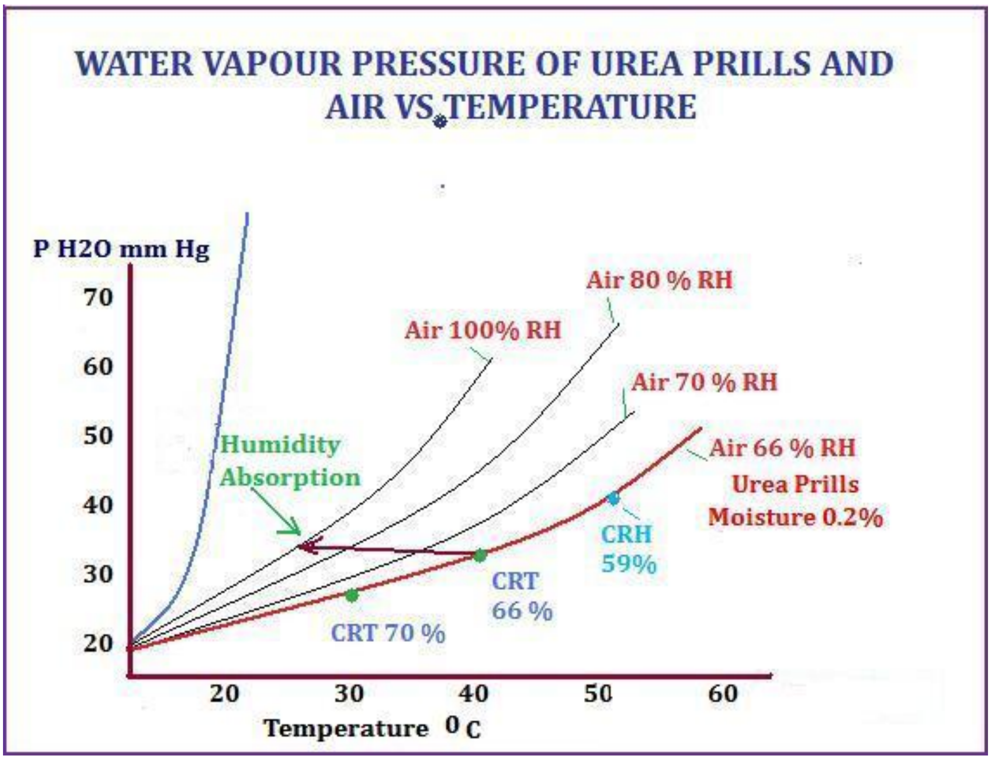


Fig-1

The choice of the commercial preferred form is normally associated with its application, solubility, crushing strength and free-flowing behaviour. The conveying and storage of urea must take into consideration the abrasive, hygroscopic and heat-sensitive characteristics of this compound.

Formation of Biuret

Formation of biuret takes place when urea is heated to its melting point it starts decomposition with evolution of ammonia, urea first isomerizes which dissociates into isocynic acid and ammonia.

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 7 Issue 04, April-2018

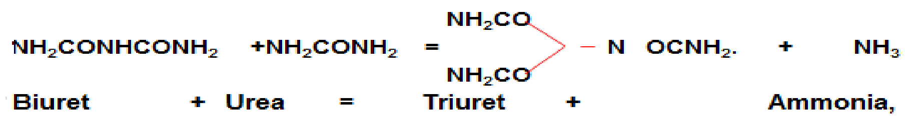
$CO(NH_2)_2 = NH_4CNO + NH_3$.
(UREA) (AMM.CYNATE)

The isocynic acid reacts with urea to form biuret.

$NHCO + CO(NH_2)_2 = NH_2CONHCONH_2$

In the presence of excess ammonia biuret is formed at substantially lower rate by direct reaction between urea molecules.

$2CO\ (NH_2)_2 = NH_2CONHCONH_2 + NH_3$.
(UREA) BIURET AMMONIA



Biuret Favorable Condition-

1. High temperature, low pressure
2. High residence time.
3. High concentration
4. Low Ammonia.

Prills Strength

Following strengths measures performances of the prills

1. Crushing strength.
2. Abrasion strength
3. Impact strength.

These parameters give useful information to the manufacturer as well as to the users regarding stability of a fertilizer to withstand normal handling without fracturing and tendency to form the dust due to abrasion and impact during course of its manufacture. Determining the crushing strength, or hardness, will help determine handling and storage requirements of a chosen granular product. generally, the crushing strength in our unit about 600 gm to 1.2 Kg per prill. The crushing strength of Granule Urea is about 1.3 to 3.2 kg per granual. The crushing strength of fertilizer particles differs greatly depending on the chemical composition. In National Fertilizers Ltd. All urea samples test experimentally to observe their breakage or failure behaviour under compressive loads. The mechanical resistance depends on surface structure and particle strength. The apparent strength calculates by dividing the failure load (force) applied, per cross-sectional area perpendicular to the force Dust and fines normally arise during handling from water absorption.

1. Poor surface structure and particle strength.
2. Low mechanical resistance.
3. Mechanical stresses in the handling chain.

Wear and tear from equipment (scrapers, screw feeders, grain trimmers etc) See also how to prevent dust formation. Crushing strength is the minimum pressure needed to crush individual particles.

Crushing strength.

Hardness test usually measure only one of these types of strength, however, in most cases good resistance to one type of mechanical action reasonable indication of good overall acceptability. As shown in the figure-2.

Abrasion Resistance

This test meant to determine degradation and dust formation due to abrasion between prills during handling of urea within the plant and outside. (Handling like conveyors, scrapper)

Impact Resistance-

Impact resistance is of interest mainly in connection with the impact imparted by fan type fertilizer spreader. In our country mechanical spreaders are not used in agricultural operation. (Used in conveyor to silo)

International Journal of Engineering Research & Technology (IJERT)
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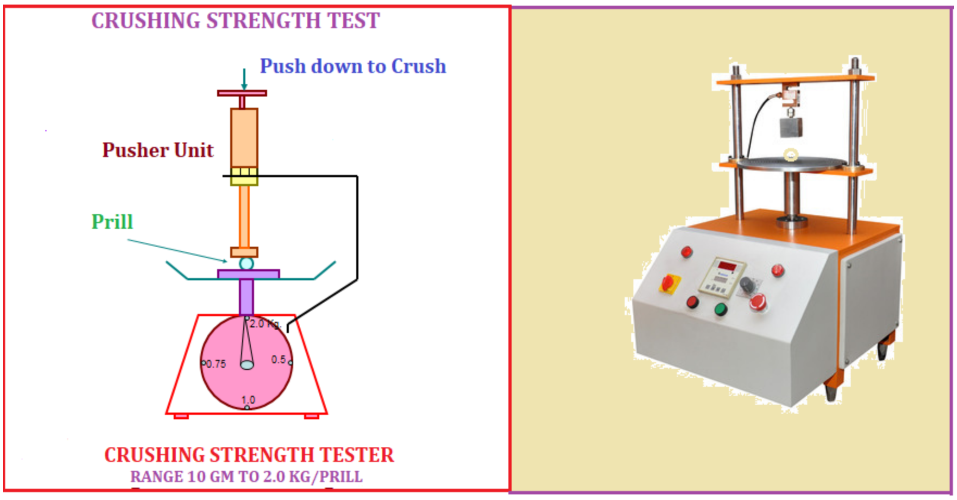


Fig-2

Urea line-II product quality

Sr. No.	Date	Nitrogen	Moisture	Biuret	Prill strength	Free Ammonia	Prills Temperature
		%	%	%	Gm/prill	ppm	^{0}C
1	27/11/2017	46.34	0.30	1.07	771.8	123	42
2	14/12/2017	46.33	0.31	1.11	764.5	125	43
3	28/01/2018	46.33	0.33	1.15	756.7	122	44
4	15/02/2018	46.32	0.34	1.18	732.2	126	45
5	01/03/2018	46.33	0.32	1.14	712.9	127	47
6	30/03/2018	46.33	0.32	1.16	703.5	135	44

Table-1

Prilling tower have variable opening louvers system, this helps to control air intake flow to prilling tower at bottom, which is very useful in rainy seasons as CRH (critical relative humidity) of urea becomes less than atmospheric humidity and prills tends to absorb moisture from air, As shown in the figure-6. Humidity of air along the height of tower increases due to evaporation of moisture from prill. Rate of change of humidity at the top is more than that at the bottom indicates most of the moisture is removed at the top when the prill is in the liquid stage. Under certain conditions of plants operations, it is not possible to remove the necessary amount of heat from the larger prills in order to attain thermal equilibrium before particle reaches to bottom of tower. This justifies high prill temperature for large diameter prills.

The critical relative humidity (CRH) = Ps/Pa

Where, Ps = Vapour pressure of saturated urea solution at given temperature.

Pa =Vapor pressure of pure water at same temperature.

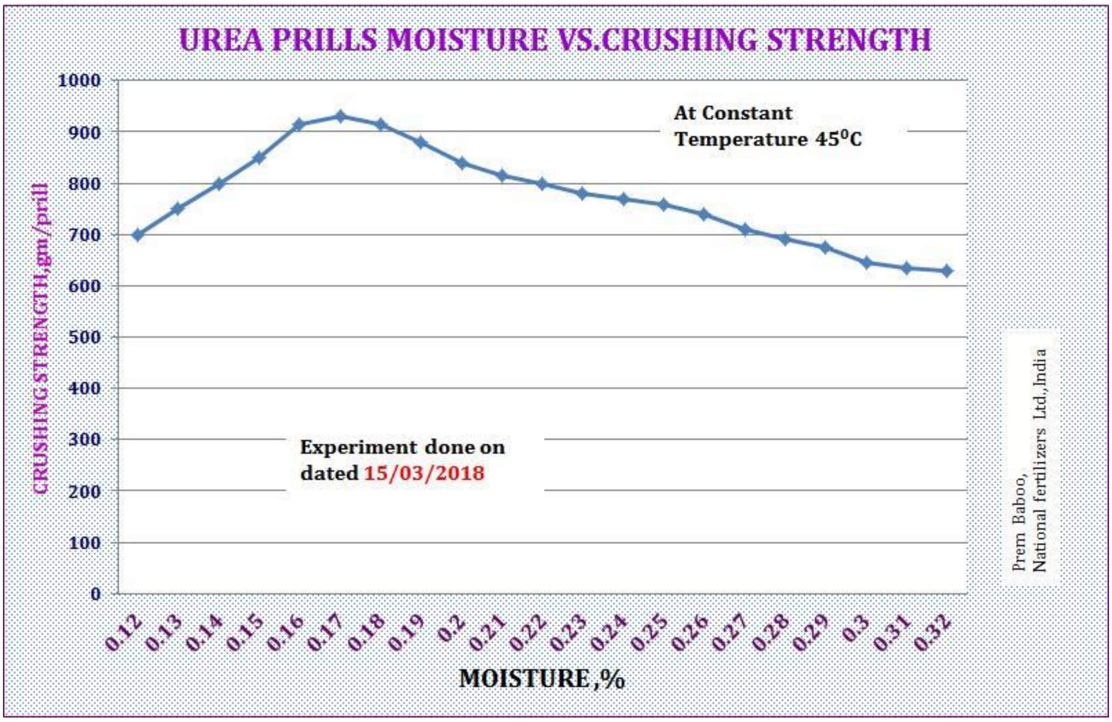
Critical relative humidity

At 20^0C : 81%

At 30^0C : 73%

With the help of variable opening louvers, we can control the air flow, temperature of prills and humidity inside the prilling tower. On dated 15/03/2018 detail experiment was done for variable prills temperature and with the help of vacuum & pre concentrator system moisture in prills control at different variables crushing strength recorded and shown in the graph 1 & 2.

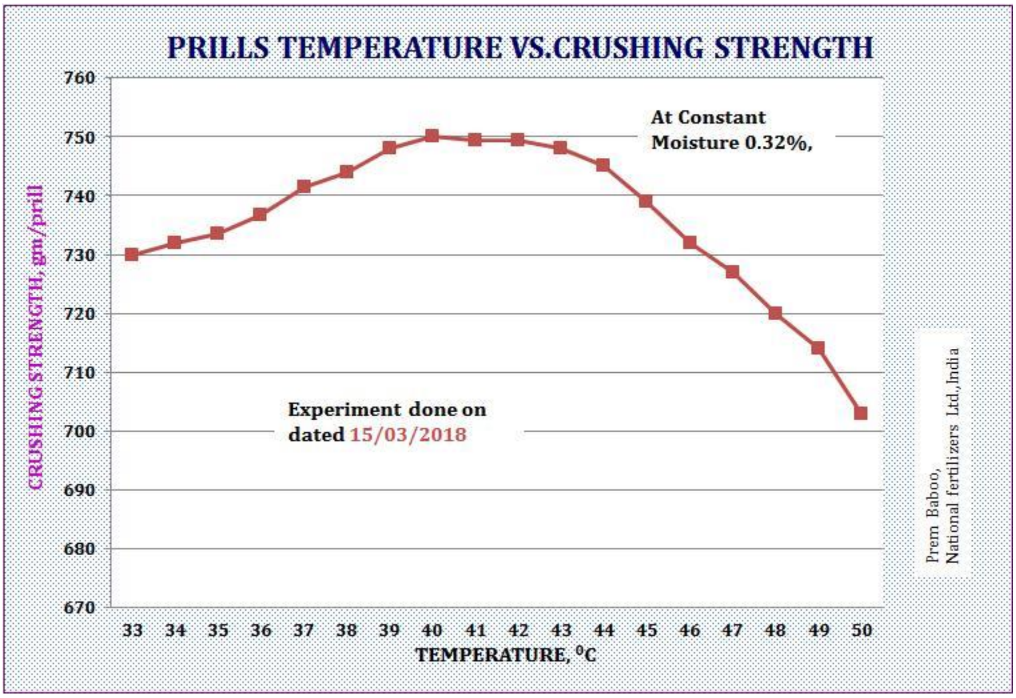
International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 7 Issue 04, April-2018



Graph-1

We can see from graph 1 & 2 the resulted prills strength is maximum at temperature 40^{0}C and moisture 0.1777%.

The detail test, although they cannot be generalized have a certain value in interpreting the expected result a certain treatment may have on the handling behaviour of prilled urea. But another important factor affecting dramatically the caking tendency of urea is the caking spreading due to humidity migration. Humidity migration phenomenon (as shown in the fig-1) is usually not considered in these detail experiments performed under constant temperature and constant moisture by variables louvers and all three stage vacuum system i.e. Pre concentrator (0.34 ata), 1st stage vacuum system (0.33 ata) & 2nd stage vacuum system (0.03 ata)



Graph-2

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 7 Issue 04, April-2018

Urea line-II product size distribution

DETAIL SIEVE ANALYSIS, UREA-LINE-II						
BUCKET No.		SIMCO BUCKET (SB-275)	TUTTLE BUCKET (TX 434)	Summary		
Sr. No.	Sieve (size distribution)	DATE-24/12/2016	DATE-27/12/2016	Simco	Tuttle	Size
		Prills %	Prills %	SB 275	TX 434	Detail
1	.+2.8	1.67	3.08	1.67	3.08	OVERSIZE
2	-2.8+2.36	6.43	5.42	96.64	95.37	NORMAL SIZE
3	-2.36+2.00	16.48	17.11			
4	-2.00+1.70	28.1	30.4			
5	-1.70+1.40	28.47	31.02			
6	-1.40+1.18	13.42	8.69			
7	-1.18+1.00	3.74	2.73			
8	-1.00+0.80	0.83	1.1	1.69	1.55	UNDER SIZE
9	-0.85	0.86	0.45			
	Total	100	100	100	100	

Table-2

FCO (FERTILIZERS CONTROL ORDER) AS PER INDIAN STANDARD.

SPECIFICATIONS OF FERTILISERS STRAIGHT NITROGENOUS FERTILISERS

Urea (46% N) (While free flowing)

Sr. No	Parameters	Value
1	Moisture per cent by weight, maximum	1.0 %
2	Total nitrogen, per cent by weight, (on dry basis) minimum	46%
3	Biuret per cent by weight, maximum	1.5 %
4	Particle size—[Not less than] 90 per cent of the material shall pass through 2.8 mm IS sieve and not less than 80 per cent by weight shall be retained on 1 mm IS sieve	

Urea (coated) (45% N) (While free flowing)

Sr. No	Parameters	Value
1	Moisture per cent by weight, maximum	0.5 %
2	Total nitrogen, per cent by weight, (on dry basis) minimum	45%
3	Biuret per cent by weight, maximum	1.5 %
4	Particle size—[Not less than] 90 per cent of the material shall pass through 2.8 mm IS sieve and not less than 80 per cent by weight shall be retained on 1 mm IS sieve.	

Urea (Granular)

Sr. No	Parameters	Value
1	Moisture per cent by weight, maximum	1.0 %
2	Total nitrogen, per cent by weight, (on dry basis) minimum	46%
3	Biuret per cent by weight, maximum	1.5 %
4	Particle size -—[Not less than] 90 per cent of the material shall pass through 4 mm IS sieve and be retained on 2 mm IS sieve. Not more than 5 per cent shall be below 2 mm IS sieve."	

Zincated Urea

Sr.No.	Parameters	Value
1	Moisture per cent by weight, maximum	1.0 %
2	Total nitrogen, per cent by weight, (on dry basis) minimum	43%
3	Biuret per cent by weight, maximum	1.5 %
4	Zinc (as Zn) per cent by weight, minimum	2.0 %

Table-3

International Journal of Engineering Research & Technology (IJERT)
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Polymer formation incresed after installation of Preconcentrator in Urea Line-II Plant

Before installation of preconcentrator the polymer deposition was found only second stage of vacuum(MV-7).because the 72% solution from L.P section feed to vacuum section there no deposition found in 1st satge of vacuum.But after installaion of preconcentrator the concentration of Urea solution increased fron 72 % to 83%.Hence the polymer deposition also found in 1st stage of vacuum(MV-6).

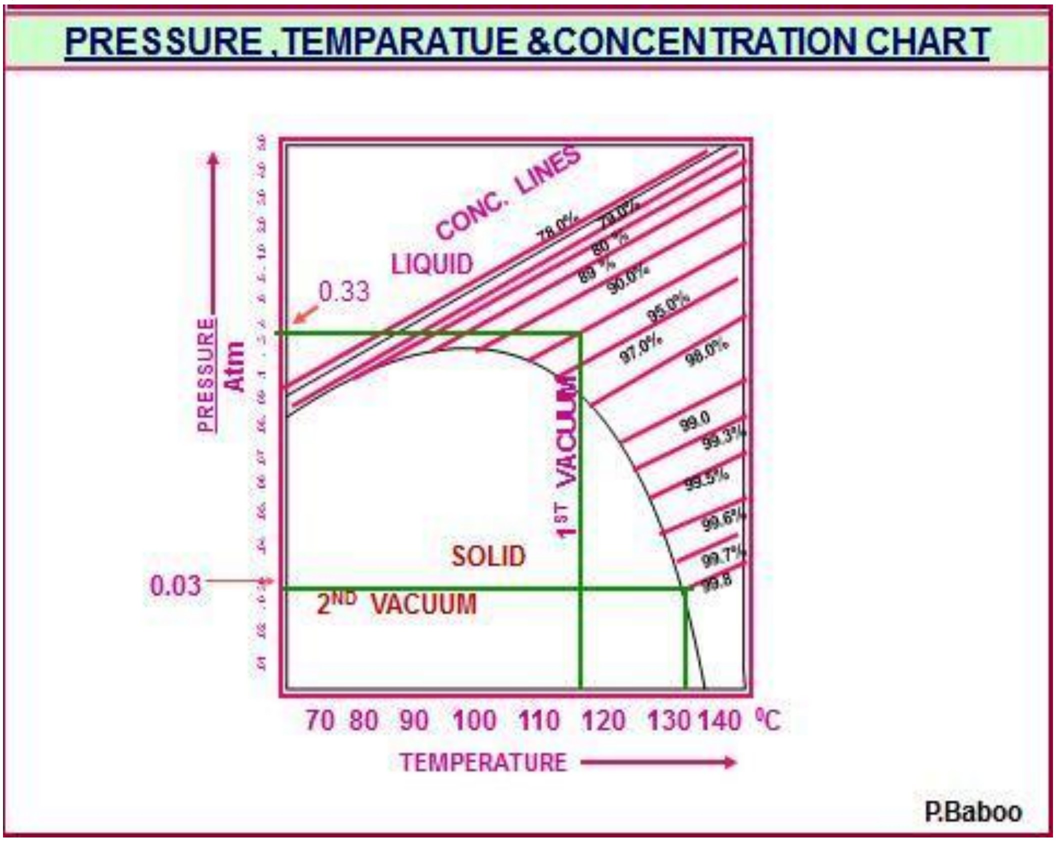

Fig-3

To overcome this problem the reguler flushing plan weeekly and the 2nd stage polymer flushing carries out with urea solution.As shown in the figure –4.The relation of vacuum,temperature & concentration chart sown in the figure-3.Some quantity of moisture in urea solution must be present to control the fluidity of the molten urea solution and to maintain the crushing strength of the urea prills. You can see from graph No-1, the crushing strength drastically decreased below moisture 0.177%.

Sr.No.	31MV-52 vacuum,ata	41 MV52 Vacuum,ata	31 PT-140,ata	41 PT 140,ata	31 PT 141,ata	41 PT 141, ata	Moisture in Urea product
1	0.54	0.55	0.46	0.45	0.075	0.077	0.41 %
2	0.44	0.42	0.40	0.43	0.068	0.065	0.35 %
3	0.34	0.35	0.37	0.38	0.04	0.059	0.30 %
4	0.333	0.345	0.36	0.36	0.038	0.0474	0.25 %
5	0.322	0.333	0.355	0.35	0.037	0.037	0.20 %
6	0.32	0.33	0.35	0.34	0.035	0.035	0.15 %
7	0.31	0.32	0.32	0.33	0.03	0.0029	0.10%

Table-4 ,(MV-52-Pre Concentrator,PT140- 1st stage vacuum,, PT-141-2nd stage vacuum

International Journal of Engineering Research & Technology (IJERT)
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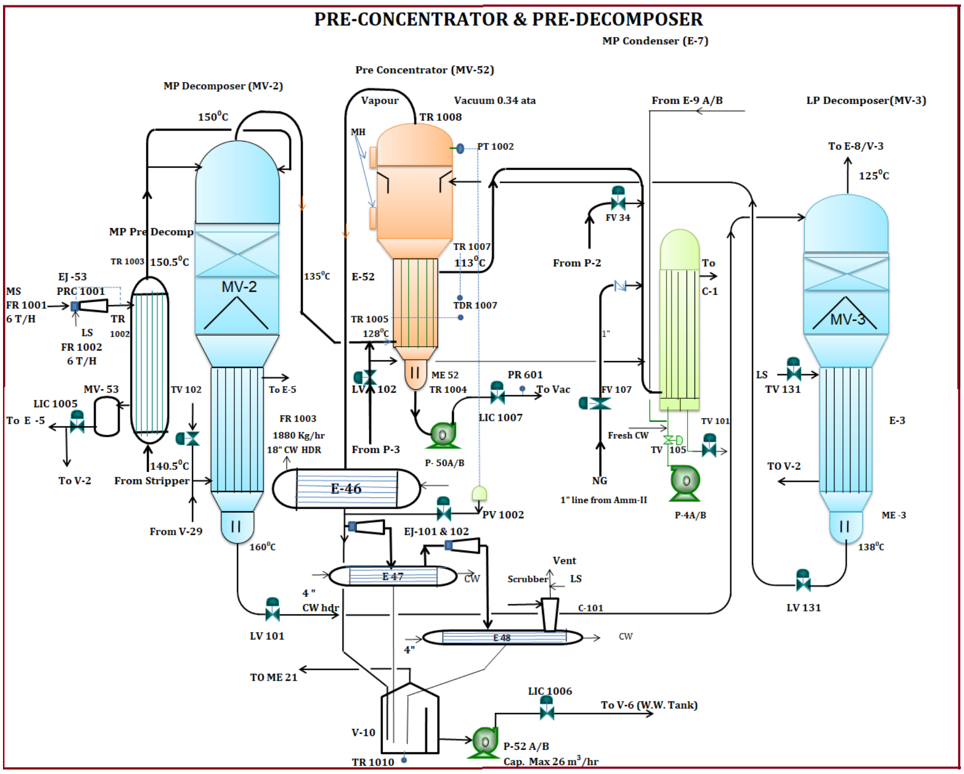

Fig- 4

Product Quality Influenced by recovery of spill urea in 2nd stage of vacuum rather than 1st stage in Urea Line-I plant.

In the process of bagging/packing some of the bags ruptured causing spillage of urea that has to recover in urea plant. "When spill urea reclaims with process stream the neem oil is carryover with water vapour through vacuum evaporator section. The waste water generated in vacuum evaporators sent to distillation tower to recover ammonia from waste water. In this processing neem oil creates major problem of foaming in waste water section causing interruption in distillation tower & reflux accumulator, pressure of distillation hunts due to foaming." "For Solve this problem neem oil must be removed from urea solution generated in dissolving area with spill urea" To overcome this problem the activated carbon filter installed in Urea dissolving area which is not more effective and also the urea solution recovers in second stage of vacuum rather than 1st stage as shown in the figure-4. But recovery must be slowly and urea solution concentration not less than 60 % if below than recovery divert to 1st stage of vacuum and partially recovers in 2nd stage of vacuum as shown in the figure -5. After this modification the waste water section foaming problem completely solved. But quality of urea some time influenced. The neem oil also goes to urea product which is also beneficial. The moisture flashed due to high vacuum. However, the crushing strength is same as before.

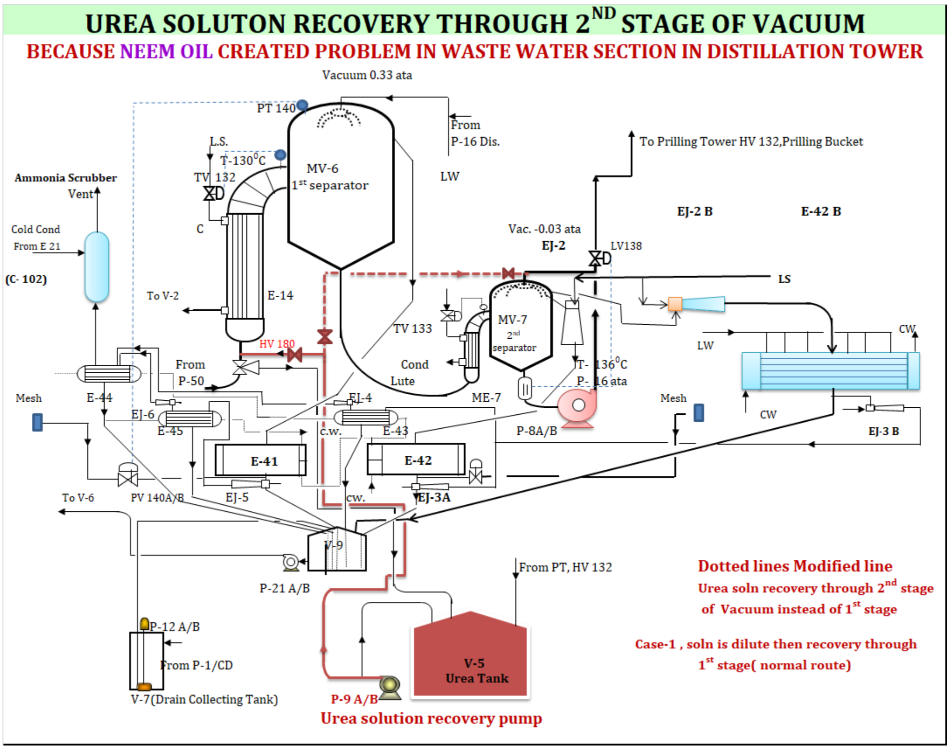

Fig-5

Fig -6

CONCLUSION

The strength measurement showed that samples of prills urea have a higher strength as their prominent size and internal structure. The hollow and large size prills having less crushing strength while the average size prills having more crushing strength. The mechanical consistence of the formed lumps will depend on recycling frequency of solution and on the quality of the urea. When molten urea introduced into the prilling tower undergoes a sub cooling which makes the final crystalline structure of the prills more fragile to abrasion and impact forces. The handling and storage of urea requires special attention to several product related properties and external factors. Most common problems are associated with moisture pick up, caking, dustiness and particle segregation. The factors that most affect the moisture absorption-penetration characteristics of urea are its chemical composition, particle porosity, particle surface area, and degree of crystallinity. Other variables which control the Urea Prills are: Urea Feed temperature; pressure composition; tower diameter; Forced or natural draft; air velocity in tower; height of free fall; ambient conditions and Pollution Control, dust & Ammonia emission from prilling tower.

Legends

BFC- Bulk flow cooler, CRH- Critical Relative Humidity, TPD-ton per day, MW-megawatt, G.cal-Giga calorie, IS-Indian Standard., Ata-absolute pressure, ppm-parts per million. Gm. – gram,SPM- Suspended Particulate Matter.