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**Continuous Emissions from urea
plant finishing sections**

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Preface

Stamicarbon, the innovative, experienced and reliable licensor

Stamicarbon, founded in 1947, is the global market leader in the development and licensing of patented urea technology with more than 50% market share in urea synthesis and about 35% market share in urea granulation technology. It sells proprietary know-how and delivers services to existing and prospective urea producers. It has licensed over 250 urea plants located in over eighty different countries. Furthermore it has completed over 90 revamp projects in Stamicarbon and non-Stamicarbon plants. Its technologies are licensed through selected licensed contractors. In some cases, technologies are licensed to plant owners directly.

One of the main activities is licensing new urea plants, providing the urea producer with all the knowledge and resources to build a reliable, state-of-the-art, low cost and profitable urea plant. Another competence is revamping existing plants increasing capacity and improving efficiency with several debottlenecking services and tools. Stamicarbon's most distinguishing activity, compared with its competitors, is its commitment to Full Lifecycle Support, helping the urea producer to get maximum return on investment for 30 years and more. Full lifecycle support comprises from planned inspections and maintenance, troubleshooting, to round-the-clock emergency support. Reliable and durable equipment is integral to the success of urea production. That's why Stamicarbon supplies critical high-pressure equipment for an optimum performance for a maximum lifespan.

Innovations

Innovation drives performance in the fertilizer industry. Stamicarbon maintains its leading position by its continuous high-quality innovations in close cooperation with research institutes, suppliers and customers.

This has led to developing breakthrough innovations, amongst others contributing to investment cost reduction and operating costs reductions:

- AVANCORE® urea process (savings on plant height and equipment)
- Mega Plant Technology (economy of scale)
- Urea 2000plus™ Technology (integrated condenser / reactor and savings on plant height and piping)
- Urea Granulation Technology (formaldehyde just 0.3 wt% and run times >100 days)
- Safurex® stainless-steel material (savings on materials, equipment and maintenance)

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Abstract

A very interesting new development is the development of the Stamicarbon fluidized bed granulation technology. In this technology, a fundamentally different spraying technology is applied: Film spraying, surrounded by a high velocity hot air gas jet.

Sprayer and high velocity hot air gas jet are located in the bottom of a fluidized bed, which is kept in fluid state by the supply of cooling air through a fluidization plate.

Comparing the fundamental difference between this 'film spraying' concept with the 'fine droplet spraying' concepts, that are used in the classical granulation concepts can best be done by comparing the sequence of events in these processes. The quantity of urea formaldehyde can be reduced, without sacrificing product quality. There is no ammonia smell in the storage building.

2. Introduction

In modern urea plants, the main pollution to the environment originates from the finishing section. This finishing section may be prilling or granulation. In both cases the freight of pollutants from the finishing section outweighs the pollution from the wet section of the plant by at least one order of magnitude.

The present best available technologies for abatement of urea dust and gaseous ammonia emission from these finishing sections are discussed.

3. Wet section versus finishing section from an environmental point of view

Looking to a urea plant, one basically can recognize two sections:

On the one hand there is the 'wet section', comprising a urea synthesis section, decomposition and recirculation stages, evaporation/crystallization sections and waste water treatment. As a product this 'wet section' produces a urea melt, or concentrated urea solution.

On the other hand we can distinguish a 'finishing section', where the urea melt from the wet section is transformed into a solid product, ready for transport from the production plant. Several final shaping processes are applied, usually distinguished as either 'prilling' or 'granulation'.

In the wet section of the plant, emissions mainly arise from the purge of non-condensable gases that are present in the feedstocks (ammonia and carbon dioxide) for urea production. Moreover, air is supplied to the urea synthesis section for two purposes: protection of the stainless steels applied against corrosion on the one hand and catalytic combustion of hydrogen impurities in the carbon dioxide for safety purposes on the other hand. All these non-condensable gases have to be vented from the wet section of a urea plant. In modern urea plants, the ammonia content of these off-gases is reduced using absorption techniques. Since the flow of these non-condensable off-gases is relatively small, absorption of ammonia in cooled process condensate has proven to be a suitable technique in order to limit the freight of ammonia to the environment from this emission source.

It should be noted that reduction of the flow of non-condensable gases remains an important factor in reducing the emission. In this respect, the introduction of better corrosion resistant material like the Stamicarbon/Sandvik development SAFUREX further reduces the emission from urea plants.

Some other sources of 'low flow – high concentration' ammonia emission sources in the urea plant wet section may be identified. Depending on the urea plant design, such flows may, for instance, arise from the recirculation condensers, or from the final ejectors in the evaporation section. Since these flows contain only low concentrations and low flows of non-condensable gases, absorption of ammonia in cooled process condensate also at these sources is a very suitable technique to reduce the ammonia emission.

Using these absorption techniques, large scale plants with wet section emissions of around 10 kg/hr of ammonia are in operation. Using the advantages of SAFUREX, and with further optimization of the absorption techniques used, in the near future plants with wet section losses as low as 1.0 kg/hr will come on line.

A major advantage of ammonia absorption in (cooled) process condensate is that the recovery of valuable ammonia from this absorbent is a relatively easy process. Using steam stripping, the ammonia is recovered from this process condensate in a rather concentrated form, which allows recycling of the ammonia to the urea synthesis section.

Also if we consider the liquid effluent from a modern urea plant, then the losses to the environment from a urea plant 'wet section' are low. Nowadays deep urea hydrolyses, combined with high efficient steam stripping technologies are standard in urea plants, making the environmental load of ammonia and urea from these sources as low as 1 kg/h for a production of 3000 ton of urea per day.

Combining the above liquid effluent and gaseous emission data from the 'wet section' of the plant, then we can conclude that the ammonia losses of this section of the plant for modern plants nowadays are in the 0.05 -0.005% range of the total ammonia feed.

If we now look to the finishing section of many present days urea plants, we see a big white plume with an appreciable ammonia smell. Quantifying the environmental load from these finishing sections, one finds that the losses over here easily go up to 200 kg/hr, bringing the losses of the back end up to 0.4% of the total feed to the plant; a figure that contrasts rather sharply to the losses of the wet section of the plant (0.005%).

It therefore may rightfully be concluded that efforts to lower the environmental load from urea production at this moment in time should mainly be targeting for the back-end finishing section of a urea plant.

Comparing the technologies that are applied in the 'wet section' to the 'finishing section', then it is obvious that the key difference lies in the amount of non-condensable gases applied. Whereas in the wet section only minor amounts of (mainly) oxygen and nitrogen need to be handled and purged, the amount of air applied in the back end of a plant easily goes up several hundred thousands cubic meter per hour. In all finishing technologies applied, be it prilling or granulation, this huge amount of air is contacting hot urea solution as well as solid urea and in these contacting processes the air gets loaded with urea dust and gaseous ammonia. Efforts to reduce the resulting environmental burden can be categorized as 'tackling the source' or 'end of pipe solutions'.

4. Dust from prilling

Prilling is a process in which the urea melt is divided into droplets using a prilling device. The droplets formed by falling down in a prilling tower, are contacted with a large amount of air. Whilst falling down, the contact with the air makes the droplets to cool down and solidify. Several kinds of prilling devices are used, such as rotating baskets or shower heads. In some technologies vibration is applied on these prilling devices in order to produce droplets of more uniform shape. Whatever device is used, they all have one thing in common: dividing the melt into droplets is done in the direct neighbourhood of the prilling device. The resulting droplets must directly have the size that is required for the final product; for fertilizer application therefore droplets of 1.5 to 2.5 mm size are the minimum that is required. Comparing this droplet size to what is usual in spraying technologies, then the droplets as produced in prilling must be characterized as 'very coarse'. As a result of this, the specific surface area of the droplets is rather small ($\sim 3.5 \cdot 10^3 \text{ m}^2/\text{m}^3$) as compared to e.g. a fine mist produced in a two phase sprayer ($\sim 1 \cdot 10^5 \text{ m}^2/\text{m}^3$). In the cooling process of the droplets, the heat transfer in the boundary layer air/melt is the limiting factor. The area of this boundary layer being proportional to the droplets surface area makes this heat transfer process for prills a relative slow process. Whilst this explains the big size of prilling towers on one hand, it also has a detrimental effect on the dust formation during the prilling process. The slow cooling of droplets causes a relative long period of time during which the droplets are in the liquid-phase. In liquid form urea has a low, but noticeable vapour pressure, and as long as the droplet surface is in the liquid form, evaporation of urea to the air in the prilling tower will occur from the droplets surface. The resulting urea vapour (which partly may be in the form of iso-cyanic acid) sublimizes on a short distance from the prill, as soon as the air from the boundary layer mixes with the colder surrounding air.

This urea desublimation process produces urea dust, which by the nature of its creation is a very fine urea dust. Typically, more than 50% of the urea dust from a prilling tower is smaller than $1 \mu\text{m}$ ("sub-micron dust").

Another aspect that is inherently to the prilling process is the large amount of air that is required. Also here, underlying is the relative small surface area of the urea droplets as they are formed in the prilling device. Whereas we already considered the consequences of this on the heat-transfer, it also limits mass transfer from the droplet to the surrounding air. Because of this limit in mass-transfer, only little water can evaporate from the urea melt. Since evaporation of water cools the urea melt, it follows that in prilling processes only little heat can be removed by evaporation, such that nearly all of the latent and crystallization heat from the melt has to be dissipated into heating up of the air flowing through the prilling tower. Since the specific heat of air is fixed, it follows that prilling requires large amounts of cooling air.

Taken the nature of the dust-formation process, that seems to be inherently to the prilling process, it is not surprising to see that no successful 'tackling the source' technologies for dust abatement in prilling have been identified so far. As to 'end of pipe' solutions, the combination of fine dust in a large amount of air makes the removal of this dust in an economical way to a challenging task.

Numerous dust-washing systems have been proposed, only few of them seem to combine the required qualities to make them successful also in commercial operation:

high dust collecting efficiency

low pressure drop

low investment cost

commercially proven

At this moment, Stamicarbon recommends scrubbers of BECO engineering company to be used on prilling towers, since they offer a reasonable price/performance ratio. At a power consumption of around 1 kWh / 1000 Nm³ of air, dust outlet concentration in the order of

25-30 mg/Nm³ can be realized. Higher collection efficiencies are achievable; however this goes to the cost of exponential growth in required pressure drop, and thus goes to the cost of a rapid rise in power consumption.

Summarizing, we may conclude that prilling is a cheap but environmentally unfriendly technology. End of pipe solutions to reduce the environmental load of prilling are expensive. Such end of pipe solutions therefore undoes the main advantage of prilling over granulation: low cost.

5. Dust from granulation processes

Granulation was developed in the 1960-1970 period as an alternative for prilling. In those days, the main drive for these new urea shaping technologies was improving the product quality. With the ever growing (and justified!) emphasis on the environmental impact of industrial activities over the years a second drive for alternative shaping technologies arose.

Several forms of the granulation process have been applied on an industrial scale. In the early days drum-granulation was a popular technology; later this role was taken over by fluidized-bed and spouted-bed granulation technologies. If we compare these classic granulation technologies to prilling, then two fundamental differences can be observed, that have a dominating influence on their performance from an environmental point of view:

Contrary to prilling, where coarse droplets are formed in a prilling device, in these classic granulation processes the urea melt is sprayed into a fine mist. Usually two-phase sprayers are applied, producing droplets in the 20-100 µm range. The design of the granulator forces a rapid contact between the liquid urea in this mist form and existing granules. Contacting of the small mist particles with the much larger granules causes almost instantaneous crystallization of the fine droplets on the existing granulate surface. As a result, the contact time between urea in liquid form and air is much smaller as compared to the prilling case. This also implies that the mechanism of evaporation of urea (and iso-cyanic acid) vapors from the urea melt, which results in the formation of very fine dust in the prilling process, is more limited (although not completely absent) in these classical granulation processes.

Contrary to prilling, after solidification, the urea is kept at elevated temperature for a longer period of time, either in the form of falling curtains (drum-granulation) or

in the fluid state of a fluid bed. This residence time at elevated temperature allows for an efficient drying process of the granules in the granulator, especially since layering is done on the outside of the granules surface. This drying process allows for urea melts that are higher in moisture content as a feed to the granulator. Since the heat of evaporation of water is considerable (certainly if compared to specific heat of air), the heat balance of these granulation processes allows for considerable lower air flows as compared to prilling.

If we look to the off gas of a granulator, then we see dust. Now, where is this dust coming from? So far, three sources of this dust have been identified:

First there is the limited efficiency of the 'catching the fine droplets' process by the granules. The design of the sprayer itself, and the geometry of the granulator in the neighbourhood of these sprayers, will influence the efficiency of this process. Depending on these factors, a certain fraction of the fine droplets formed in the sprayer will be solidified before they get in contact to the surface of an existing granule. This fraction of premature crystallized droplets will get entrained as dust in the air leaving the granulator. Since this dust is originating from a spraying process, it is relatively coarse as compared to the fine dust produced in a prilling tower.

As a second source, in these classical granulation processes still some fine sub-micron dust is formed from the process of evaporation/desublimation from urea melt. The amount of fine dust formed here is considerably lower as compared to the amount formed in prilling processes.

As a third source, there is dust that is introduced via the recycle of solids. The nature of granulation processes is growth of existing granules. In a continuous production process, this of course also implies that somewhere new seed material has to be supplied.

Usually, this seed material to some extent is formed as overspray in the neighbourhood of the sprayers, and/or is supplied as broken oversize product. Especially this latter process, breaking of granules into finer particles, is a process that inherently produces some dust. If this broken product is recycled to the granulator without classifying, then this dust will be entrained with the cooling air in the granulator. By the nature of its formation, also this dust is a very coarse material.

Summarizing on these 'classical' granulation technologies, we see two major differences if compared to prilling in this respect:

Granulation requires less air

The dust from granulators is coarser.

The combination of these two factors makes 'end of pipe' solutions for the removal of this dust easier (and cheaper) as compared to prilling. Yet, the amount of dust to be handled and recycled in these classical granulation technologies is certainly not negligible (values in order of 5 to 20% of the entire production have been reported), still leaving quite some room for further improvement. Moreover, the presence of a (relative small) amount of fine submicron dust limits the efficiency of (economical) dust removal.

A very interesting new development in this respect is the development of the **Stamicarbon fluidized bed granulation technology**. In this technology, a

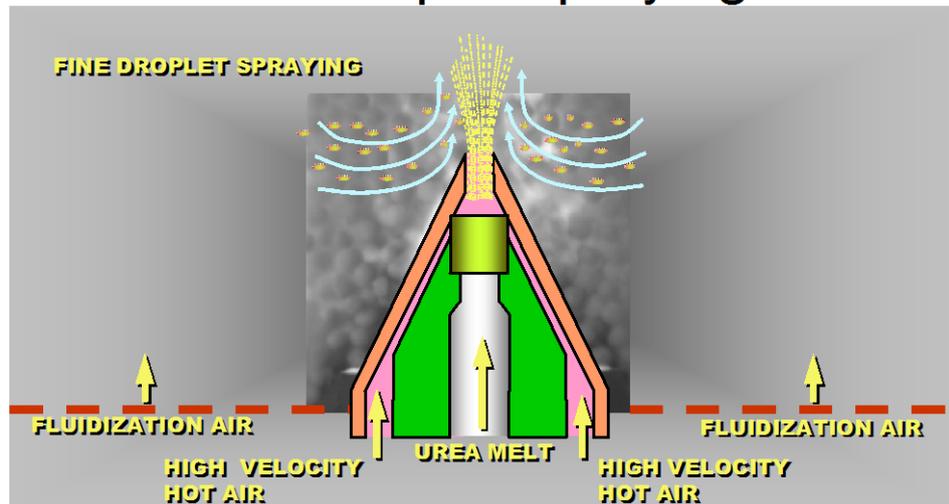
fundamentally different spraying technology is applied: Film spraying, surrounded by a high velocity hot air gas jet.

Sprayer and high velocity hot air gas jet are located in the bottom of a fluidized bed, which is kept in fluid state by the supply of cooling air through a fluidization plate.

Comparing the fundamental difference between this 'film spraying' concept with the 'fine droplet spraying' concepts, that are used in the classical granulation concepts can best be done by comparing the sequence of events in these processes.

“ Fine droplet spraying, as applied in classical granulation technologies”

Fine droplet spraying



Here the sequence of events is:

1st event: The urea melt is fed to the gas jet.

This step can be accomplished in different ways, in particular regarding the “shape of the urea melt” as it is fed to the gas jet. In a DSM/Stamicarbon pilot plant following “shapes” were investigated:

- droplets
- film
- cylinder

Shape 1 and shape 2 were obtained through hydraulic-type means.

Shape 3 is automatically obtained upon exit of the urea melt from a cylindrical tube.

2nd event: The high velocity gas jet collides with the urea melt, and this collision results into fine droplets.

With shape 2 (film) the smallest amount of jet gas (energy) was required for this step. Shape 3 (cylinder) required the largest amount of jet gas energy.

3rd event: The Urea-droplets-load gas jet enters the FB

4th event: Granules from the Fluid bed are sucked into the gas jet and are rapidly accelerated. At the same time they are wetted by the fine urea droplets. Meanwhile the gas jet itself is rapidly slowed down.

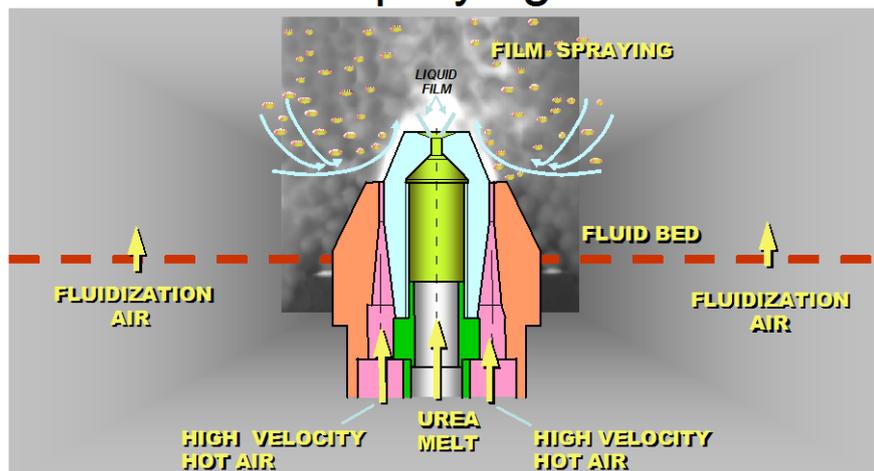
In our research program all sprayers of this concept showed the following common features:

good water evaporation due to high droplets surface area. Therefore relative high water content of the urea melt is possible (> 2 wt%).

Dust formation in the granulator is a critical factor; high levels of formaldehyde are needed for dust reduction.

“FILM spraying, as applied in the Stamicarbon fluid-bed granulation technology:

Film spraying



Here the sequence of events differs fundamentally from the previous one:

1st event: The high velocity gas jet enters the fluid bed.

The entrance of the gas jet is situated below the feeding point of the urea melt.

2nd event: Granules are sucked into the gas jet and are rapidly accelerated. Meanwhile the gas jet itself is rapidly slowed down to very moderate velocities (< 40 m/s).

3rd event: The urea melt is injected into the granules/gas system as a film. Due to moderate gas velocity, this film stays intact over a certain distance behind the injection point.

4th event: The accelerated granules move through the film, whereby the film covers the granules as a layer (compare to paint brush strokes).

All sprayers of this type tested in this film spraying concept showed the following common features:

Limited water evaporation due to low melt surface available for evaporation; therefore a low water concentration of the melt is required.

Zero dust formation, even with zero formaldehyde.

We will now discuss in greater detail the mechanism of dust formation in relation with the spraying technology:

Submicron dust.

The total set of experimental data from our pilot plant reveals that this type of very fine dust is formed along the following mechanism:

Evaporation of gaseous components (H₂NCO, NH₃, some urea) from the urea melt (~135°C)

Followed by desublimation of these components from the gas phase at the fluid bed temperature (~105°C) to form solid urea as submicron dust.

This of course is the same process as observed in urea prilling towers.

The huge effect of the urea spraying technology on this type of dust is easily understood on the basis of the much smaller surface area of melt that is in contact with air, when applying film spraying. Measurements in our pilot plant, as well as measurements in commercial operating plants applying the film spraying technique confirm the absence of sub-micron dust in this case.

Micron dust.

Our total set of experimental data revealed that the amount of this type of dust increases as:

Finer droplets of urea are formed

Sprayer design is such that these droplets need a longer time to contact a granule.

These findings point at something happening with the fine droplets while on their way to the granule, as the cause of micron dust. It can easily be calculated that very fine droplets (for instance below 25 µm) in air can cool down extremely quickly. This means that they can crystallize before hitting a granule in the fluid bed. The resulting fine crystalline particles do not stick to the granule surface, and are blown out of the fluid bed as micron (1-25 µm) dust.

The weight-average droplet size with the "Fine droplet" sprayers we tested was always considerably higher than 25 µm, typically 60 µm. Yet, even with an average droplet size of

60 µm, always a fraction of droplets below 25 µm is also formed. This is due to the random character of the droplet formation, which results into a wide droplet size distribution.

With "Fine droplet" spraying, this type of micron dust easily becomes a problem for process stability and/or a limiting factor for plant run time.

With "Film spraying" according to the Stamicarbon granulation technology, urea droplets below 25 µm are not formed, so there is no source for micron dust formation in the granulator.

Measurements in plants operating with the Stamicarbon film sprayers have confirmed the absence of sub-micron dust in the off gas of the granulator. The only remaining source of micron dust appeared to be the dust that is introduced into the granulator with the recycled crushed oversize product. The size of this dust is very coarse, making it easy to catch in low pressure-drop wet scrubbers.

The absence of sub-micron dust also greatly improves the opacity of the plume from the granulator vent stack.

6. Ammonia in the off-gasses from prilling and granulation

Apart from dust, the off-gas from prilling towers and granulators also contains ammonia. The main source of this ammonia is the free ammonia present in the urea melt feed to the prilling/granulation. In all prilling and granulation processes, the major part of this free ammonia is liberated from the melt/solid urea, and ends up in the main air off gas stream from the prilling/granulation process. Attempts to reduce the amount of free ammonia present in the urea melt have proven to be quite difficult. At temperatures above the solidification temperature of concentrated urea melt, biuret formation is a rapid process:



Especially since this reaction is accelerated by low ammonia concentrations, it is practically impossible to produce a concentrated urea melt without any free

ammonia. In actual industrial scale plants, urea melts are produced with free ammonia concentrations in the 100 – 1000 ppm range.

The amount of air used in prilling/granulation processes is in the range of 5 to 20 kg air/kg urea; such that the ammonia concentration in the off-gas without treatment will be in the 10 - 200 ppm range. The freight of ammonia to the environment from this source, without treatment, thus will be in the range 0.1 to 1 kg of ammonia per ton of urea produced.

For a long time disposal of such an amount of ammonia into the environment was considered acceptable. Nowadays however ever more signals are received indicating that our industry should do something about this. This is the more, since after reduction of the urea dust from prilling/granulation, the freight of ammonia in the off-gas represents the main source of pollutant from a urea production plant.

It has been proposed to tackle this problem at the source, by adding acidic components to the urea melt before it is introduced into the prilling/granulation process. Although such a process certainly is feasible, it has the disadvantage that the urea product gets polluted with an ammonia-salt. This makes the product unsuitable for certain (technical) applications.

In the wet section of a urea plant scrubbing with water has been applied successfully in reducing the ammonia in gaseous effluents. Unfortunately this technology cannot be applied in the finishing section of a urea plant, because of the huge amounts of air involved. As a result of these high air flows, the ammonia is present in a low concentration (~ 10 – 200 ppm range), diluted in a non-condensable gas (air). For instance, at 50 ppm ammonia in air, the partial pressure of ammonia at ambient pressure is only some 0.05 mbar. At 40°C, an ammonia water mixture containing as little as 20 ppm of ammonia also exerts an ammonia partial pressure of about 0.05 mbar. This implies that scrubbing water with less than 20 ppm of ammonia should be used in order to have any driving force for scrubbing ammonia from the gas to the liquid phase. In order to maintain such low ammonia concentrations, huge amounts of circulating water would be required.

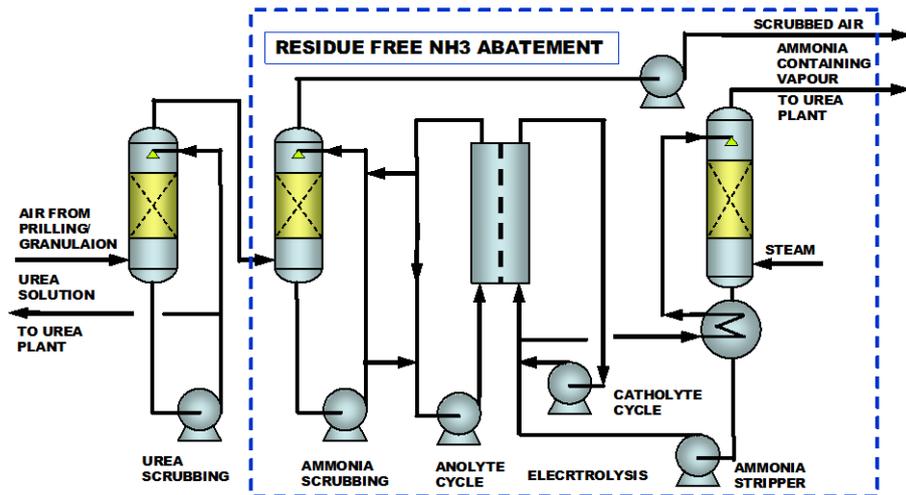
Moreover recovery of ammonia from ammonia-water mixtures at such low concentrations, on an economical feasible basis, would be a challenge on its own.

A rather obvious solution seems to be adding some acidic component to the scrubbing water. The ammonia vapor pressure of aqueous solutions rapidly falls off if the pH of the solution is reduced, making acidic aqueous solutions perfectly suitable as a scrubbing agent. In this way, very low ammonia concentrations in the gas phase can be obtained using standard gas/liquid contacting devices. As a serious drawback of this technology it should however be noted that in this case we produce an ammonia salt containing side stream. What to do with this?

In some industrial complexes, useful applications of such ammonia-salt solutions can be identified. For instance, if there is some UAN production within the industrial complex, we could use nitric acid, and send the produced ammonium-nitrate solution to the UAN production. For those locations where such solutions are not

possible (e.g. stand alone ammonia-urea production sites), Stamicarbon together with its licensed contractor UHDE now has developed a new “Residue free NH₃-abatement” process that does not produce any by-product.

Residue free ammonia abatement



The concept of this scrubbing operation exists of:

Acidic scrubbing of the prilling/granulation off-gas with e.g. sulphuric or nitric acid.

Electrolytic decomposition of the produced ammonia salts.

Stripping of the ammonia using steam stripping

Recycling of the stripped ammonia to the urea process.

Recycle of the sulphuric/nitric acid to the scrubbing operation.

The estimated investment cost for a 2000 MTD urea plant would be in the order of € 1.5 Mio.

Since the acid used for scrubbing is also recovered in the electrolyses unit, the consumption of chemicals for the process will be minimal (only make up of minor losses).

Status of this new developed process:

At present, this new process is not yet proven on commercial scale. All process steps, with the exception of the electrolytic decomposition of ammonia salts are well proven technology. Even the cells for the electrolytic decomposition of ammonia salts are well proven in other processes, e.g. Chlorine electrolysis. Only the optimum membrane is different.

Small scale experiments have been executed with quite encouraging results. A concentration of approx. 1 mol of acid was gained in the anolyte cycle and the desired ammonia concentration in the catholyte cycle was also achieved. Urea and some formaldehyde were added to the synthetic scrubbing solution in order to ensure a composition as realistic as possible. Both species are also destroyed in the electrolysis unit. Whether the by-products generated in the process contain any harmful constituents in significant amounts, which may demand additional measures, requires further extended test trials, preferably in an actual operating plant.

In order to come to a commercial scale implementation of this new concept, Stamicarbon at present is looking for a urea producer who would be interested in the further technical development of this concept.