

# TECHNOLOGY

## UREA GRANULATION – PART 1

### Granulation of Urea: Diamonds, Pearls and Stamicarbon Granules

Author:

Wim Roos, Senior Process Engineer  
Stamicarbon B.V., The Netherlands

#### SUMMARY

Stamicarbon is a reputed licensor for the urea process. Stamicarbon licenses the whole range of finishing technologies: prilling, granulation and pelletizing.

The fluid bed granulation technique results in better quality product because the granule is built up layer by layer. Before a new layer of liquid melt is applied on the particle, the particle is completely solidified. The granulation system was developed in a commercial plant in Belarus. Today, a plant is running with a capacity of 2,250 tpd. Due to the film spraying nozzle, the process results in a product with excellent quality. Considerable savings compared to other processes can be achieved due to a reduced formaldehyde content of the final product.

The filming characteristics of the nozzle of the Stamicarbon process results in low dust formation. Due to the low dust formation the plant can be operated during 2 or 3 months without interruption.

#### HISTORY STAMICARBON GRANULATION

In the 1970s, the fluid bed granulation process of Stamicarbon was developed in the

research facilities of Stamicarbon. This process was based on Stamicarbon's proprietary film-spraying nozzle.

A commercial test facility was contracted at Azot Grodno plant in Belarus. A small granulation unit was completely converted to

the Stamicarbon process.

In 2002, the first product was produced. After the introduction of the Stamicarbon granulation process, Agrium signed a contract for the installation of the Stamicarbon granulation process in their urea complex in

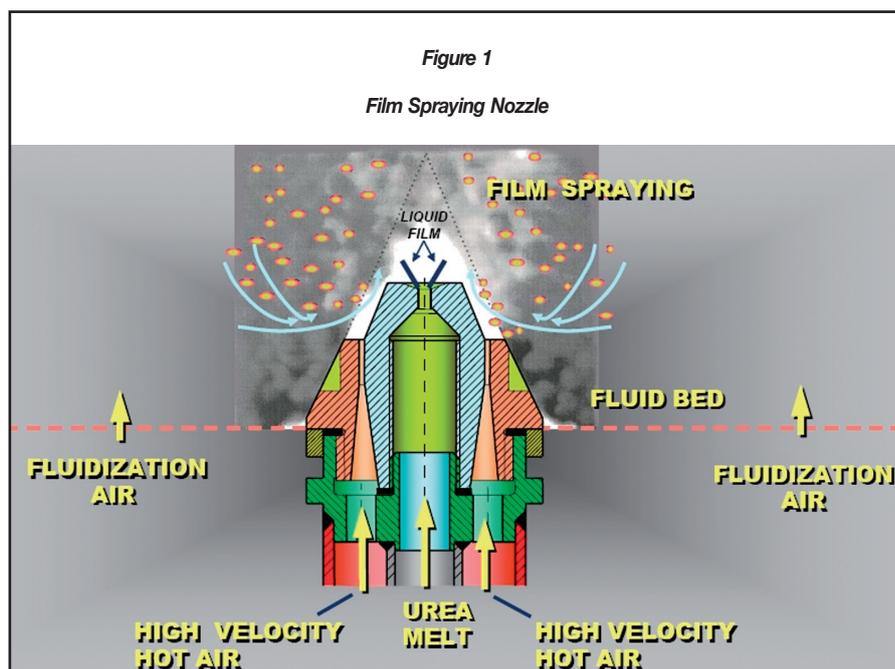
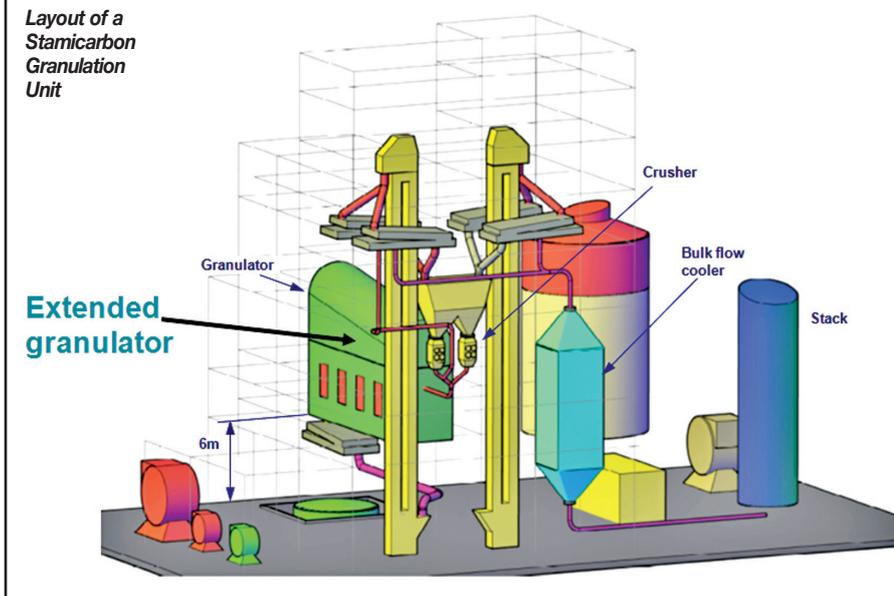


Figure 2:

Layout of a Stamicarbon Granulation Unit



Fort Saskatchewan, Canada. Agrium operated two lines, each with a capacity of 625 tpd. These lines were originally installed in 1980 according to the UFT process. Agrium decided to convert these lines to the Stamicarbon granulation process. After a reconstruction of the granulator, the Agrium plant commenced the production of granules using Stamicarbon's license in 2003. Subsequently, several contracts were awarded to Stamicarbon (see the reference list).

### NOZZLE IN THE STAMICARBON PROCESS

When the nozzle design process commenced in 1980, a set of conditions for the design were formulated. The granulation nozzle was to have the following properties:

1. Low energy consumption;
2. Excellent final product quality;
3. Low dust formation; and
4. Low formaldehyde consumption.

The combination of these requirements resulted in the development of a nozzle based on film spraying. In particular, condition 3 above relating to low dust formation has resulted in the use of thin films on top of the spray nozzles in Stamicarbon's fluid bed granulation process.

The drying time of the thin film layer must be short enough to prevent agglomeration of wet particles. This requires a highly concentrated solution to enable fast crystallization. In Figure 1, a schematic drawing of the filming nozzle is shown. The red dashed line shows the level of the fluidization plate. The seed particles are sucked into the liquid melt film by the high velocity hot air flow. The nozzle is covered with a layer of fluidized urea granules. The wetted seed material flows into the area of the

granulation bed and exchanges the crystallization heat to the fluidized bed.

The nozzle density in the granulator is such that it ensures that the particle is completely crystallized before again entering a melt film on top of the nozzle tip.

The filming process leads to a granule with a very smooth outer surface. The sphericity of the final product is very good. This property is important in relation to caking behaviour. The contact area between pure spheres is minimal. Irregularity of shaped granules increases the contact area between the granules. Caking can result due to the sorption of moisture in contact areas between the granules. If the contact area is reduced, caking behaviour is improved.

### PROCESS DESCRIPTION

A standard lay out of a granulation unit is shown in Figure 2. The core of the granulation unit is the granulator casing, consisting of:

1. an under casing with the air compartments;
2. a fluidized bed in a rectangular box with the same outside dimensions as the under casing;
3. an upper casing with the free board zone, where the air is collected and flows to the scrubbing system. The upper casing is provided with a hemi spherical head.

The product is classified in the main screens and the crusher. All this equipment is located above the granulator casing.

At the outlet of the granulator, the product passes a lump screen. After passing the lump screen, the product is lifted with a bucket elevator to the classification equipment. All solid

product flows via gravity flow through the classification equipment.

The liquid melt is fed to the granulator with a temperature of 140 °C in a concentration of 98.5% urea plus biuret. In the granulator, the bed temperatures are maintained in a range of 102 – 110 °C. In the cooling zone of the granulator, the product is cooled to a temperature of 85 °C.

At this temperature, the classification in the main screens also takes place. The feed to the crusher is cooled to a temperature of 70 °C. At this temperature, the coarse material flows to the coarse bin before entering the crusher.

The crushed product and the fine recycle flow are combined and recycled to the granulator.

The good product in the outlet of the main screens is cooled to storage temperature in a solid flow cooler. This cooler is cooled by cooling water. This way of cooling is far cheaper than fluidized bed cooling. A fluidized bed cooler in this position is combined with a refrigeration unit. The energy consumption of this unit is quite high and most often not included in the cost comparisons for granulation units.

The dust-loaded air from the granulator and the dedusting points is collected. This air flow is fed to a scrubber where the dust is washed out with a urea-containing washing liquid. The emitted dust concentration can meet all current standards. Most standards currently prescribe a maximum emission of 30 mg/Nm<sup>3</sup>. If the air has to be treated for ammonia, an acid scrubbing unit is required. The ammonia contained by the urea melt flow accounts for an emission of about 100 mg/Nm<sup>3</sup> in the stack. As soon as this emission has to be reduced, acidic scrubbing must be considered. The emission can be reduced by about 70 %.

### PRODUCT QUALITY

The final product properties are related to the chemical composition of the granules and their physical properties (e.g. caking behaviour and crushing strength).

#### Chemical composition

The requirements for the chemical composition of the granule product are very similar for all projects. A biuret concentration of 0.90 % wt is widely accepted. Most granulation processes will fulfill this requirement. In the Stamicarbon process, the reduced formaldehyde concentration and the low water content of the final product mean that the required nitrogen content in the final product can easily be met. The final nitrogen content is higher than 46.2 %. The formaldehyde content of the product is in the range of 0.28% to 0.35 % for Stamicarbon granulation units. For plants

licensed by other companies, a concentration of 0.40% to 0.60% is applied.

Experience with the operating plants has shown that under all circumstances the water content does not exceed the guaranteed value of 0.30 %. A water content in the product of 0.10 – 0.15 % is quite normal.

The free ammonia content of the granule is in the range of 5 to 30 ppm. In the storage building, no smell of ammonia is apparent.

**Physical properties**

An important property for the product is the size distribution. The appearance of the product at the first glance plays an important role for the qualification of the process. The client expects a required average diameter in combination with a good uniformity of the product. The Stamicarbon process produces a product with a high uniformity index, without negative influence on the dry recycle ratio. The classification of the product is achieved by the main screens. A good selection of the mesh size of the screen clothing and a proper setting of the crusher gap will result in the required particle size distribution. The granulation process of Stamicarbon has a very high granulation efficiency. Most of the product exiting of the granulator has the required diameter, resulting in a low dry recycle flow during stable operation.

The other properties like crushing strength and caking behaviour are connected. There is a strong relationship between water content, caking behaviour and crushing strength. Low water content results in a better crushing strength and a lower caking tendency. Caking plays an important role in the handling and transport of urea. Actually, caking is an intrinsic property of the product; good product can suffer from caking when it is exposed to bad conditions.■

Granulation plants					
Year of order	Total cap. mt/d	Units	Clients	Country	Contractor
2012	1,760	1	Bangladesh Government, BCIC	Bangladesh	China Chengda Engineering, China
2011	3,250	1	Pardis Petrochemical Company	Iran	Iranian
2011	1,575	1	Egyptian Chemical & Fertilizer Industries – KIMA	Egypt	Tecnimont
2010	2,700	1	Tierra del Fuego Energia Y Química S.A., Rio Grande	Argentina	China Chengda Engineering Co., China
2008	2,200	1	Petroquímica de Venezuela ("Pequiven), Puerto Nutrias	Venezuela	-
2008	4,400	2	Petroquímica de Venezuela ("Pequiven), Jose	Venezuela	-
2008	3,600	1	NPC – Golestan	Iran	Iranian (by NPC)
2008	3,600	1	NPC – Zanjan	Iran	Iranian (by NPC)
2008	3,600	1	NPC – Lordegan	Iran	Iranian (by NPC)
2008	1,400	1	PJSC Novomoskovsk Joint Stock Company AZOT, Tula Region	Russia	Chemoprojekt
2007	4,000	2	Egyptian Agrium Nitrogen Products S.A.E. (EANPC), Damietta	Egypt	Uhde GmbH, Germany
2007	2,000	1	PJSC Novomoskovsk Joint Stock Company AZOT, Tula Region	Russia	Chemoprojekt
2006	1,200	1	Grodno Azot, Grodno	Belarus	Chemoprojekt, Czech Republic
2004	2,000	1	Egypt Helwan Fertilizers	Egypt	Uhde, Germany
2003	2,000	1	Egyptian Fertilizer Company (EFC), Ain Sukhna	Egypt	Uhde, Germany
2003	2,000	1	Alexandria Fertilizer Company (AlexFert), Abu Qir	Egypt	Uhde, Germany
2003	1,250	2	Agrium, Fort Saskatchewan Nitrogen Operations	Canada	Agrium
1996	280	1	Grodno Azot, Grodno	Belarus	Grodno

## Urea/Ammonium-Sulphate (UAS) - killing two birds with one stone -

**Stamicarbon has developed a flexible and economic method for highly efficient emission abatement from urea granulation plants with simultaneous production of highly-marketable UAS-based fertilizer.**

Today, urea granulation plants are equipped with gas scrubbers to capture and reduce

ammonia and urea dust particles in the exhaust gases from the FB granulator and product cooling devices in the finishing section. These gas scrubbers are designed to achieve extremely high efficiencies and to meet the emission levels specified in the environmental permit, while keeping pressure drop low in the interests of minimizing energy consumption.

Usually, the scrubbers are vertically-disposed vessels with multiple tray stages and/or mist eliminators. Recirculated urea solution enters at the top of the dust removal section and flows down by gravity while the off-gas enters at the bottom of the scrubber and flows upwards in countercurrent. On its way down, the liquid flows horizontally over the trays or mist

eliminators and comes into contact with the off-gas rising through the tray openings, capturing its content of urea dust.

The NH<sub>3</sub> present in the off-gas is not efficiently removed by this mechanism for two reasons.

- The physical solubility of ammonia in the circulating urea solution is low on account of the low partial pressure of the ammonia vapour in the off-gas.
- The gas/liquid contact time is insufficient for significant transfer of NH<sub>3</sub> from gas to the liquid phase.

Therefore, the approach for removing ammonia is acidic scrubbing. A similar kind of scrubber internals is used for ammonia scrubbing, but acid is added to the circulating scrubbing solution to absorb/convert the NH<sub>3</sub> into the respective ammonium salt (AS). If done in a separate acidic ammonia scrubber after the dust scrubber, an AS solution contaminated with traces of urea will be produced.

Urea dust and NH<sub>3</sub> can be scrubbed together in a single scrubber vessel by circulating an acidified urea/salt solution, which absorbs/dissolves both the dust and NH<sub>3</sub> simultaneously. Under these acidic conditions, slightly more AS will be produced since a small part of the urea will be hydrolyzed to ammonia.

The selection of the scrubbing acid (HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub> or others) is influenced by the final destination of the salt product, but other factors such as the market price of the acid, its availability, logistics, its corrosion characteristics, market and customer preferences will play an important role as well. Sulphuric acid is usually preferred, since it is widely available and it contains sulphur, which is increasingly demanded as a supplementary nutrient in addition to the nitrogen already supplied in the urea granule.

Stamicarbon has proven scrubber

technologies for reducing urea dust and NH<sub>3</sub> emissions of your urea melt-granulation plant. They are found in the table below.

Based on design experience and commissioning of fluid bed urea granulation plants, Stamicarbon has developed several end-of-pipe expedients for NH<sub>3</sub> abatement. If the granulation plant is on the same site as an UAN plant, a solution of urea with ammonium nitrate in water resulting from the acidic scrubbing of NH<sub>3</sub> with HNO<sub>3</sub> can be easily fed to the UAN plant.

Alternatively, if the scrubbing solution is acidified with sulphuric acid, the AS produced in the acidic scrubbing stage and the urea solution from the dust scrubbing stage can be concentrated together in a separate evaporator unit and then routed to the urea granulator, in which case the entire urea production then contains approximately 0.05-0.1% S, or it can be processed in a separate finishing section to produce UAS fertilizer with a higher S content (say 5%) as a value-added by-product without producing any waste streams.

Using a separate finishing section for the by-product provides the opportunity to conduct the solidification process in a pelletizer system; this is advantageous for 3 main reasons.

- For the small scale of by-product production, pelletizing is more economical than granulation (lower energy consumption);
- The main plant outlet will continue to be pure urea, if so desired; and
- Pelletizing easily facilitates customized blending with minor nutrients.

Such an arrangement, producing a separate by-product with a significant AS content and a salt-free urea main product, is the subject of a Stamicarbon patent application of 2009 (WO 2011/032786 A1). It is described below.

Although the acidic scrubbing system would be designed to take in concentrated H<sub>2</sub>SO<sub>4</sub>, it will work with lower concentrations, but at the expense of extra energy consumption for concentrating the solution. Ammonia reduction efficiencies of 75-90% or more can be attained. Make-up water is added into the scrubber to ensure that the concentration of urea and AS in the liquid phase remains within the solubility limits. The clean air leaves the scrubber via the exhaust.

This technology can reduce the ammonia content of the off-gas to 10-25 mg/Nm<sup>3</sup>. It is important to maintain the correct pH in the scrubber, because although lowering the pH of the recycle increases the NH<sub>3</sub> absorption efficiency, it also increases the hydrolysis rate of urea. Optimally the acid feed rate to the scrubber should be set so that the solution off-take from the scrubber circuit to the urea/ammonium sulphate (UAS) solution tank is marginally acidic (almost pH-neutral). Typically, this UAS solution has a composition of about 41 wt-% urea and 4 wt-% AS. The appropriate amount of NH<sub>3</sub> or aqua ammonia is introduced to neutralize its residual free H<sub>2</sub>SO<sub>4</sub>.

The neutralized UAS solution is sent to one or two falling-film vacuum evaporators. Depending on the configuration of this concentration section, the UAS solution is concentrated to a melt containing less than 5 wt%, 1 wt%, or even less than 0.3 wt-% of water. The evaporator off-gases are passing condensers, from which the non-condensable portion is sent to the granulation plant stack. The condensate is returned to the granulation scrubber to close the water loop and to reduce the consumption of external process water. To obtain a final UAS melt with whatever S/N ratio desired in the produced fertilizer to suit the agronomic conditions under which it is to be used, the appropriate amount of solid AS can be added to the UAS solution tank or to the concentrated UAS melt. Subsequently, the melt is transformed into UAS-containing solid particles. The solid shaping step may comprise granulation, prilling or pastillation.

Stamicarbon's development work on pastillation has been based around the Sandvik Rotoform pastillation machine, which is an environmentally-friendly process, with low energy consumption and is adaptable to handle different product compositions.

From a 2,200 mtpd granulation plant equipped with a combined urea dust and ammonia gas scrubber, around 56 tpd of solid 5 wt-% S UAS can be produced, well within the capability of a single Rotoform machine. The salt concentration (S content) can be varied over a significantly wider range if a separate pastillator is used. That enables the urea producer to generate precisely customized specialty UAS-based nutrient-enhanced fertilizers. All in all, it's a 'win-win' situation for everybody - farmer, fertilizer producer and environment alike. ■

**Table 1 - Summary of acidic scrubbing options**

Preferred salt outlet	aqueous solution				solid by-product	no by-product
Dust and NH <sub>3</sub> scrubbing	combined		separate		combined	combined
Acid used	H <sub>2</sub> SO <sub>4</sub>	HNO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	HNO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>
Main product	urea	urea	urea	urea	urea	urea +AS (0.2 wt-%)
By-product	UAS aq.	UAN aq.	AS aq.	AN aq.	UAS s.	none
Evaporation section required	no	no	no	no	yes	yes
Finishing section required	no	no	no	no	yes	no
Present in Stamicarbon portfolio	yes	yes	yes	yes	yes	yes

(AS = ammonium sulphate, UAS = urea/ammonium sulphate, AN = ammonium nitrate, UAN = urea/ammonium nitrate)