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Forum & Technical Field Visit

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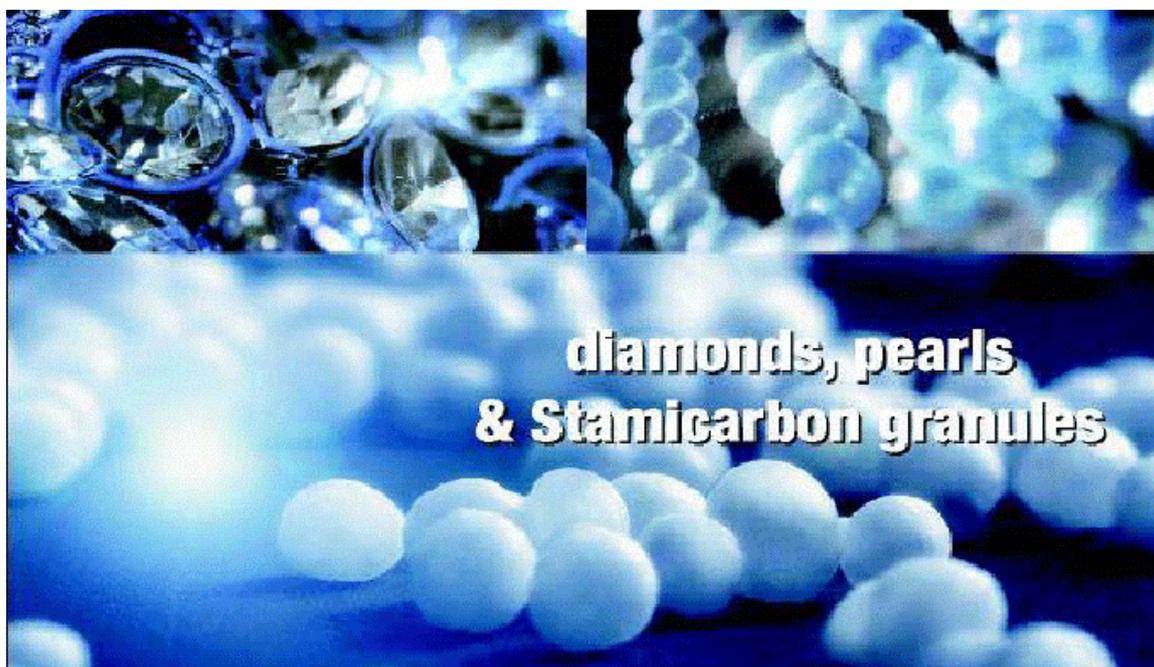
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STAMICARBON'S GRANULATION TECHNOLOGY DIAMONDS, PEARLS & STAMICARBON GRANULES

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1. Introduction

The name Stamicarbon is synonymous with know-how. Know-how in chemical processes, operations and training. As such, Stamicarbon has achieved a leading position in urea process licensing and continuously improved and innovated the urea technology resulting in the successful Urea 2000 plus technology. This innovative technology is commercially proven and applied in all recently awarded urea projects with capacities up to 3500 mtpd.

The changing market situation, with one of the major licensors of the fluid bed granulation withdrawing from the market, made Stamicarbon that commercialized its patented fluid bed urea granulation technology recently developed in the seventies and eighties, recently applied it in a 280 mtpd plant in GPO Azot, Grodno.

This plant is operating at or above design capacity with superior product quality meeting all product quality standards.

Stamicarbon is now in the position to license the total package urea technology: urea melt plant and finishing sections either prilling or granulation.

This paper will shed some light on the Stamicarbon granulation technology, the spraying nozzle principles, the process characteristics and the advantages of the Stamicarbon process.

2. History of DSM/Stamicarbon Research and Development of the Urea Granulation Technology

History

- 1976 DSM/Stamicarbon: orientation on granulation technologies
- spouted bed granulation
 - fluid bed granulation
 - pan granulation
- 1977 Research project within Fertiliser Division and Stamicarbon started
- 1978 Pilot plant testing (200 kg/h) in closed granulation loop
- 1979 Semi-commercial granulation unit (50 t/d) in operation at DSM urea unit in Geleen. Day to day operations by urea production department.
- 1980 Development and testing low pressure "film" sprayer concept 200 kg/h. Process design for a 1000 mtpd fluid bed granulation unit.
Granulation development costs more than Euro five million
Stamicarbon started cooperation with HFT
- 1994 HFT announces a restrictive licensing strategy for its fluid bed granulation
- 1997 Stamicarbon started cooperation with GPO Azot in Grodno, Belarus to install a 280 mtpd fluid bed granulation unit.
- 2002 Commercial production of high quality product in GPO Azot
HFT announces again a restrictive licensing strategy.

During the late seventies and early eighties DSM/Stamicarbon developed and tested a new fluidized bed granulation process for urea and ammonium nitrate. At the time, similar processes were being developed by a number of our competitors. More specifically, some of them were looking for a shaping technology that would give better performance than prilling.

The research project into fluid bed granulation was initiated and directed by the Fertilizers Division of DSM. Other participants in the project were DSM's Fertilizer Research Department, DSM's Equipment Testing Department and Stamicarbon BV.

In 1994 HFT, who was market leader in the granulation technology announced a restrictive licensing policy, leaving its licensed contractors and several prospect without a finishing technology. This was a drive for Stamicarbon to restart its research and development of its patented granulation technology. This resulted in the cooperation with GPO Azot, Grodno and in a joint effort an existing but partly scrapped plant was re-designed and installed.

Now that HFT again decided to restrict its licensing in the future, Stamicarbon has its own commercially proven technology ready for licensing and design packages for scaling up to world capacities have been prepared.

3. The GPO Azot, Grodno Fluid Bed Granulation Plant

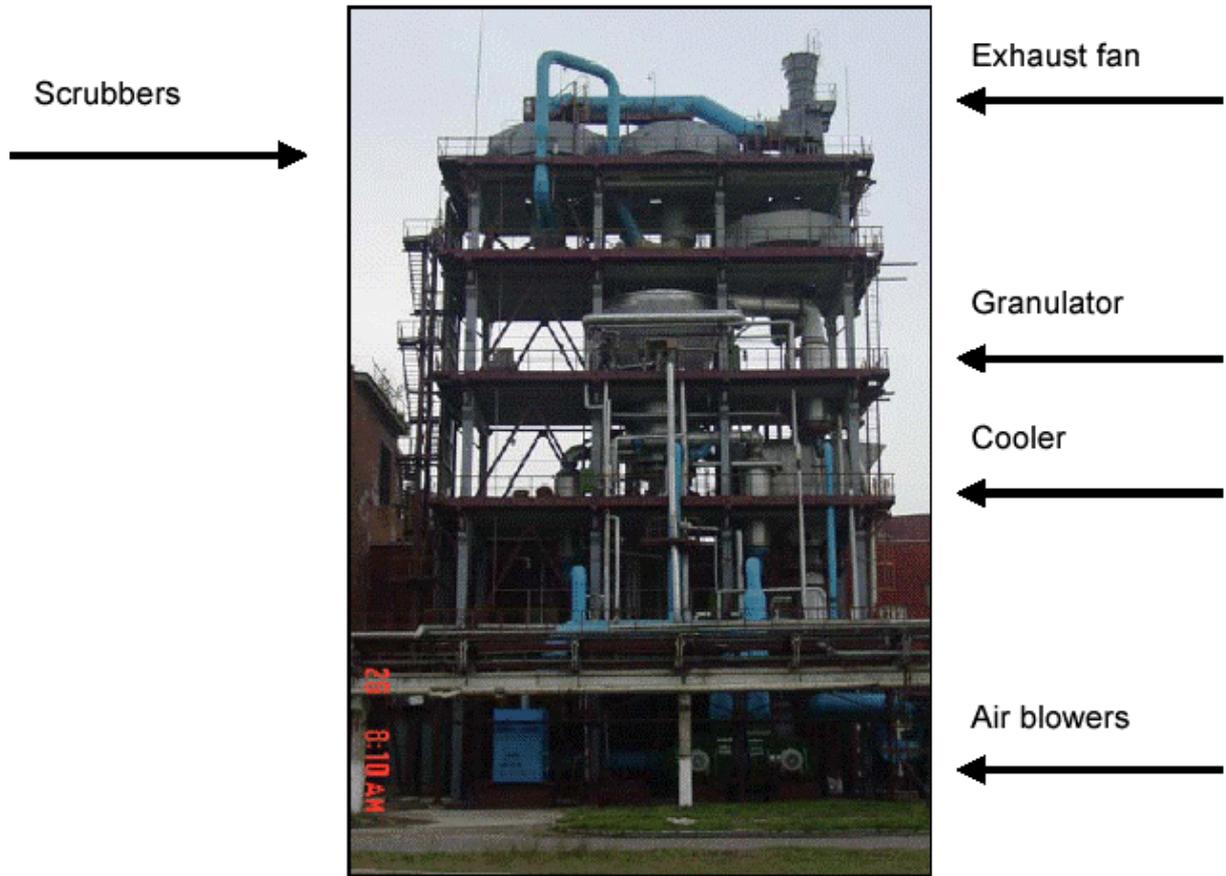


Figure 1: GPO Azot plant

With scrubbers, exhaust fan, granulator, cooler, air blowers, GPO Azot operates two urea plants in Grodno, Belarus. They happened to have its own designed granulation plant but this plant stood idle and was partially scrapped. GPO Azot and Stamicarbon decided to cooperate and to rebuild the Grodno granulator with Stamicarbon's technology. The design capacity for this unit is 280 mtpd. Use was made of the Grodno granulator housing but with the Stamicarbon designed fluidization plates and spray nozzles installed.²

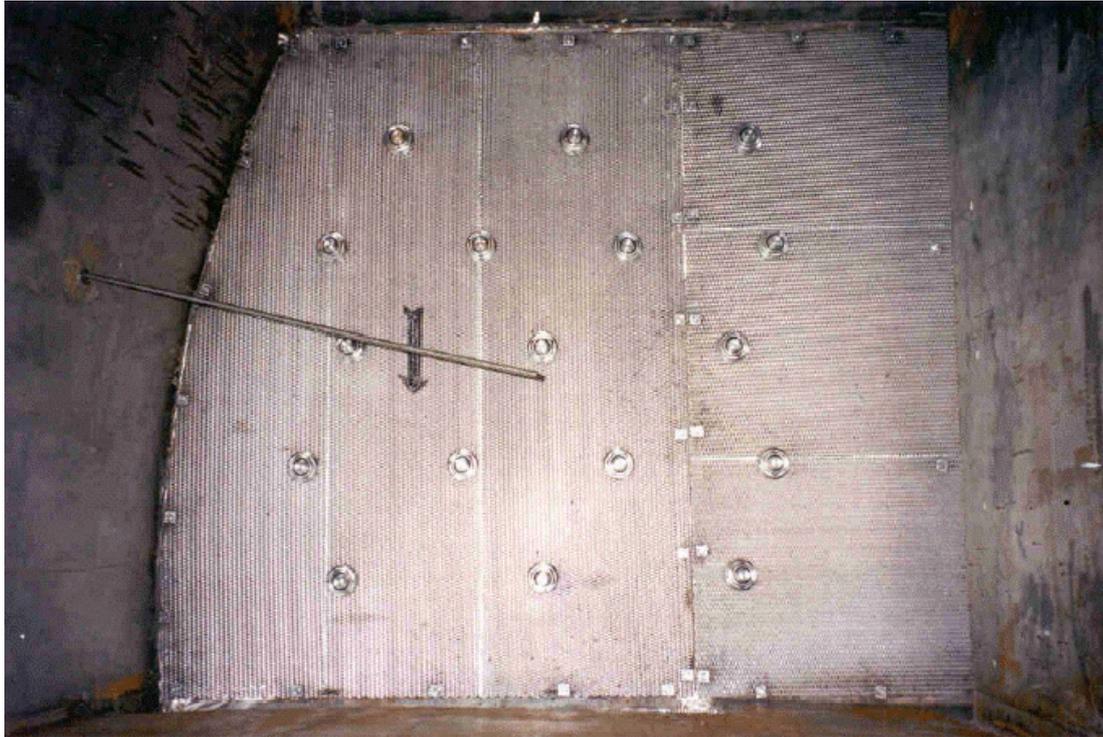


Figure 2: Internal fluid bed granulator

The plant went in operation in February 2002 and has been successful in operation at or above the design capacity since. The longest continuous run has been 25 days and was terminated due to problems in the urea melt plant. The product is of a high quality with a good roundness, good crushing strength and excellent size distribution.

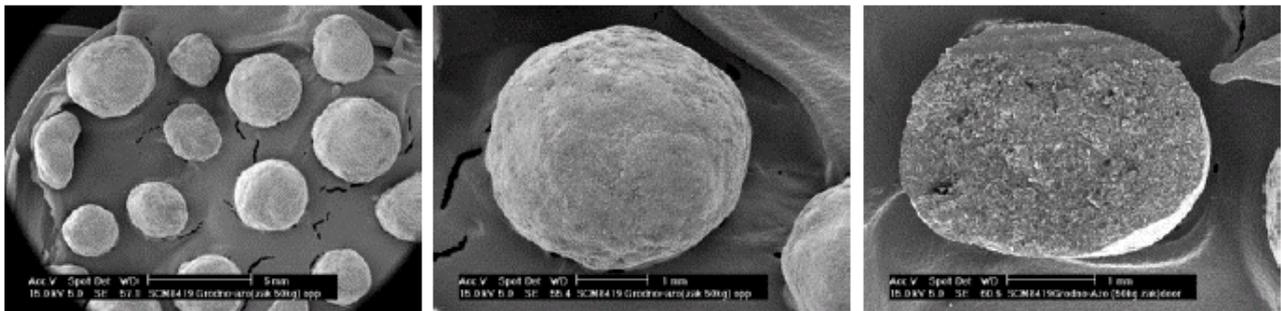


Figure 3: Grodno granular urea

Typical Grodno product quality:

Nitrogen content	46.3	wt %
Moisture content	0.25	wt %
Biuret content	1.1 (note 1)	wt %
Crushing strength	3.	kg (on 2.8 mm dia granule)
Roundness	> 80	%
Formaldehyde content	0.20	wt %
Size distribution (2-4 mm)	> 95	%

(Note 1: Grodno operates with extreme long urea melt feed line to the granulator. Expected figure biuret for a new plant: 0.8 wt %)

4. Process Characteristics

In the Stamicarbon fluid bed granulation process, granular urea is produced by low-pressure 'film' spraying of liquid urea onto seed material in fluidized state.

The basic characteristics of this process are:

- the liquid urea is a concentrated solution (melt);
- the spraying occurs in the core of a fluidized bed by means of a large number of spray heads;
- the particle size enlargement is achieved by scale granulation, i.e., growth of the seed or nucleus granules by continuous solidification of very thin layers of urea melt onto the initial particles;
- formaldehyde (in the form of urea/formaldehyde pre-condensate) is added to the urea solution before spraying, as an anti-caking agent.

In order to spray onto a large number of solid particles simultaneously with no agglomeration, it is necessary to keep them apart. Fluidization is a very effective method to avoid contact between particles over a long period of time until they have cooled down enough.

During the entire residence time in the granulation zone of the granulator, each granule is repeatedly covered with a thin film of urea melt. The particle size growth is uniform and progressive with the residence time and results in a uniform shape and good quality granules.

5. General Process Description

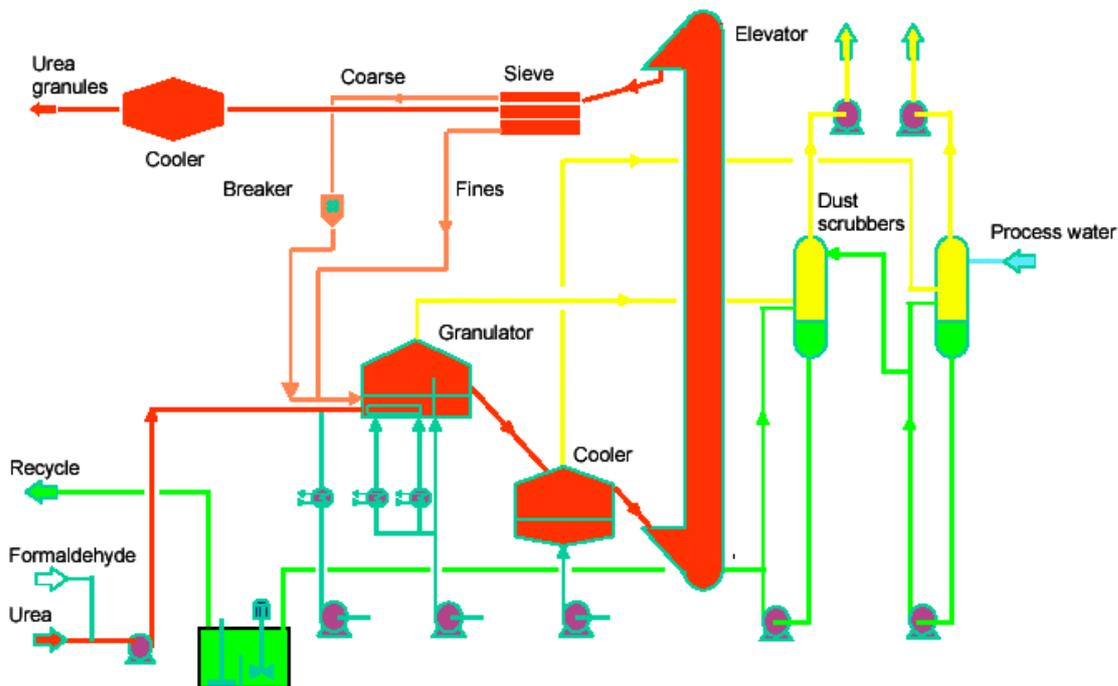


Figure 4: Process flow sheet granulation section

Urea melt, with a concentration of about 98.5 wt %, is transferred from the urea melt plant to the granulator by means of a urea melt pump

In the suction line of the pump urea formaldehyde is added. This injection of urea formaldehyde is used as granulation additive and anti-caking agent. It also improves the granule crushing strength and reduces dust formation during handling.

The formaldehyde containing urea melt is sprayed as a very thin film in a fluidized bed of urea particles in the granulator.

The granulator is divided in a granulation section and a cooling/conditioning section.

In both sections fluidization air is evenly distributed by a perforated plate to fluidize and cool down the granules. The fluidization air is delivered by a granulator air fan.

The seed (recycle) material is introduced into the first chamber of the granulation section, where it is sprayed upon by urea solution. As granules move along through the granulation section, their size steadily increases by layering to reach the required granule diameter at which the product finally flows out the granulator.

The urea melt is fed to the injection headers. The injection headers are connected to the urea melt line and the secondary air system.

Each injection header features vertically placed risers fitted with spray nozzles spraying the urea melt onto the seed particles. The secondary air, necessary to transport the granules through the urea melt film, is delivered by a sprayer air blower.

The granulated product flows from the granulation section into the cooling section (without spray nozzles) of the granulator to cool down and harden the granules before further processing.

The fluidization and secondary air and some urea dust are exhausted from the top of granulator by means of an off gas fan in the off gas line of granulator scrubber.

In the scrubbers the air is separated from the scrubbing solution. The clean air is thereafter exhausted to the atmosphere.

The scrubbing solution (diluted urea solution) is partly recirculated as scrubbing solution to the scrubber and partly pumped to urea dissolving vessel and recycled to the urea melt plant.

The product from the granulator is extracted by a extractor and flows through a lump screen which screens off any lumps to the granulate cooler.

The granulate cooler is a fluid bed cooler type. The fluidization/cooling air is delivered by a granulate cooler air fan.

The fluidization/cooling air, containing some dust, is exhausted from the top of granulate cooler and combined with the air from the product cooler and the dedusting air. This combined stream is cleaned in the cooler scrubber system.

By means of bucket elevator the cooled urea granules are lifted into main screens where granule selection occurs. The granules are classified in coarse product (oversize), final product (on-size) and fine product (undersize).

The final product is transported to the product cooler. In the product cooler the product is cooled down to a temperature of 40°C with air in a fluidized bed cooler. After the product is cooled down to the required temperature the final product is directed to the storage.

The dust loaded air from the granulate cooler, the product cooler and the dedusting air with some urea dust are exhausted from the top of granulator coolers and the dedusting points into a scrubber. The film type sprayer used in the granulator does not produce any fine size particles. The dust present in the granulator off-gas originates only from some attrition in

the fluid bed, as well as from some fine product produced in the crusher. Therefore relatively little dust is present in the off gas, and the dust that is present is relatively coarse ($> 10 \mu\text{m}$) Because of this (little, coarse dust) cleaning the off-gas from urea dust is relatively easy in our granulation technology.

The fine product is recycled to granulator and is used as seed material.

The coarse product is directed to crushers. After crushing the small-sized product is discharged to the granulator, or, alternatively to the granulate cooler.

In the urea-dissolving vessel the urea lumps from the lump screen are dissolved in the urea solution from the granulator scrubber.

6. Principle of the Low Pressure 'Film' Sprayer

After initial pilot plant tests using a single sprayer the process was tested in a 50 mtpd unit in DSM's urea plant. The regular shift operators of the urea plant operated this unit. The unit produced lots of about one thousand tonnes of product. This product was tested prior to shipping. The performance of this unit was highly satisfactory and yielded a lot of operating experience.

The 50 mtpd fluid bed granulation unit was designed on the basis of sprayer know-how available at DSM/Stamicarbon.

Concurrent with this project a new sprayer design was developed for an urea fluid bed granulation project. This new sprayer design, known as the low pressure 'film' sprayer, gave promising results and showed distinct advantages over the sprayer design used in the 50 mtpd unit.

The low pressure 'film' sprayer for urea fluid bed granulation was tested by DSM's Equipment Testing Department in a fully closed 200 kg/h granulation loop. This loop comprised all equipment normally encountered in a granulation loop, such as a granulator, granulate cooler, bucket elevator, screens, crusher, transport equipment, off-gas washing, fans, etc. These tests were very successful.

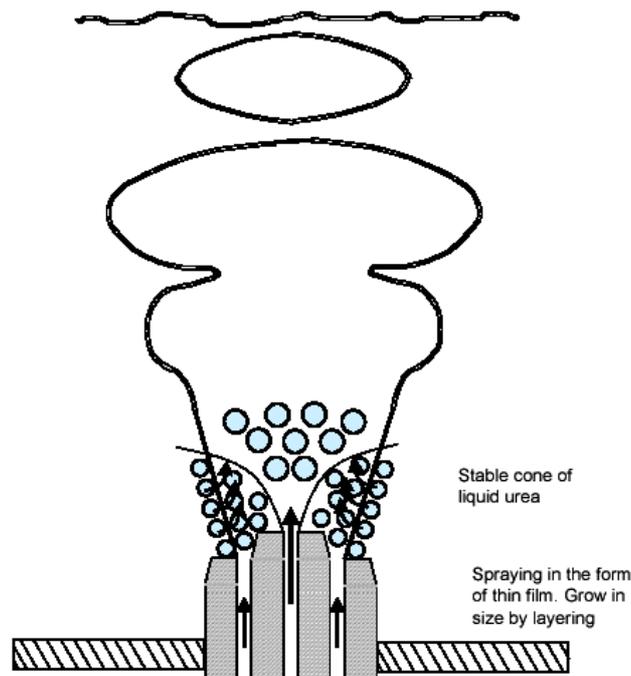


Figure 5: Cross section low pressure film sprayer

The 98.5 wt % urea solution is sprayed in the form of a thin film. Secondary air, supplied through an annulus surrounding the melt sprayer sucks the urea granules through this film. Each time a granule passes the liquid film it grows in size by layering.

Granulation by means of low pressure film spraying results in much less dust generation in the granulator as compared to high pressure spraying or two phase flow atomizing spraying, in which case the urea is atomized in a fine mist.

The low-pressure spraying technique as applied implies also that hardly any nuclei are formed in the granulator. To satisfy the number balance of granules in the granulation loop a certain number of nuclei are prepared by crushing and subsequent screening of the oversize particles.

Summarizing the advantages of low-pressure film spraying are:

- limited dust production in the granulator
- low spray energy per ton of urea
- formaldehyde content can be low
- low urea dust emission

7. Proprietary Equipment

The granulator and its internals are proprietary equipment and can only be purchased from the Stamicarbon approved suppliers. Stamicarbon will inspect points during the fabrication of the granulators.

The low-pressure 'film' sprayers can only be procured from Stamicarbon.

All other equipment has comparable dimensions as in other processes because the unit operations such as transporting, cooling, screening, crushing, scrubbing are comparable.

8. Emissions

The film spraying technique applied in the granulator ensures that no fine dust is produced in the granulator.

Because of the absence of fine dust present BAT values for urea dust in the granulator stack easily can be obtained using simple, low pressure drop, washing technologies.

If required, the ammonia concentration in the off-gas can be reduced using acidic washing technologies.

9. Conclusions

- Stamicarbon has a well proven fluid bed granulation technology
- Product quality achieved is excellent:
 - high roundness and uniformity
 - low formaldehyde content
- Low urea dust formation
- Overall investment cost is estimated to be comparable with all other processes on the market

- And last but not least the Stamicarbon granulation technology is available for licensing

Figure 6: Overall view Grodno granulation with direct loading facility.

