

# NUTRIENT- ENHANCED UREA PRODUCTION



**STAMICARBON**



**NEXTCHEM**

MAIRE Sustainable Technology Solutions



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Author(s)	Gerardo Duarte
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## ABSTRACT

This paper explores various aspects of fluid bed slurry granulation and its future potential for nutrient-enriched nitrogen fertilizer production. It delves into solid characterization, urea-solid compatibility, and solid particle size of the nutrient source as foundational components for slurry granulation processes. Pilot plant tests validate the proof of principle providing the pathway for scale-up. Effective fertilization practices have consistently emphasized the need for a balanced nutrient supply for crops, highlighting the significance of multi-nutrient-enhanced nitrogen fertilizers in improving nutrient use efficiency and food production. Historical reference is provided through the 1974 Stamicarbon symposium, which underscored the shift toward incorporating phosphorous and potassium into fertilization practices, thus strengthening interest in NPK fertilizers based on urea.

There is a critical need for nutrient- and efficiency-enhanced fertilization, aligning with the International Fertilizer Association (IFA) 4R Nutrient Stewardship principles to improve yield and reduce environmental losses. Enhanced nitrogen fertilizers offer a stable price alternative in the volatile commodity market, presenting opportunities for producers to diversify their portfolios. Despite evident benefits, adoption remains limited due to price sensitivity among large-scale growers and wholesaler focus on high-margin segments.

Fluid bed slurry granulation technology of Stamicarbon, the nitrogen licensor of NEXTCHEM (MAIRE Group), is presented as a solution to the limitations faced in traditional agglomeration methods for urea-based NPK fertilizers. Moreover, the paper outlines future directions for fluid bed slurry granulation technology, including construction material selection to enhance durability and reduce wear. The application of aqueous urea solution as slurry medium is proposed to improve processing efficiency.

Ultimately, Stamicarbon's slurry granulation technology seeks to enhance urea production by incorporating valuable plant nutrients through solid sources. This approach aims to increase production capacity and add value to the product, thereby improving profit margins. Explore the benefits of Stamicarbon's slurry granulation technology and experience the advantages of advanced solid processing firsthand.

## 1 BACKGROUND

Crops need a balanced supply of nutrients like nitrogen, phosphorous, and potassium (NPK) for healthy growth. Other nutrients like sulfur, calcium, magnesium, zinc, iron, boron and manganese are also important. Fertilizer applications should match soil and crop needs. Multi-nutrient-enhanced nitrogen fertilizers are here to improve efficiency and increase food production.

Stamicarbon hosted its fourth symposium in 1974, during which the third topic on the agenda was "Preparation of urea-based PK fertilizers" presented by C. Hoek<sup>1</sup> (Figure 1). This presentation served as a

follow-up to a subject previously introduced at the 1971 symposium. The interest in this topic was articulated under the following premise: *"After prolonged and exclusive use of nitrogen for fertilization, it will become necessary to incorporate  $P_2O_5$  and  $K_2O$ . Consequently, there will be a growing interest in NPK fertilizers based on urea, given that urea is the only nitrogen source abundantly available in many regions."* Five decades later, the motivation for this development has intensified, expanding beyond PK fertilization to include micronutrient fortification and efficiency enhancers, thereby advocating for nutrient-enhanced nitrogen-based fertilizers.

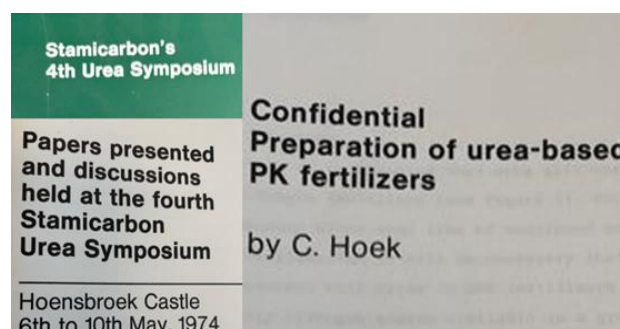


Figure 1: Presentation topic by C. Hoek at the fourth Stamicarbon symposium.

<sup>1</sup> Preparation of urea-based PK fertilizers, 1974.

Multi-nutrient and efficiency-enhanced fertilization is an important aspect of modern fertilizer management practices. The IFA 4R Nutrient Stewardship principles aim to provide balanced nutrients that meet crop and soil needs (Right Source & Right Rate), ensure nutrient availability throughout the crop cycle (Right Time), and minimize nutrient losses by applying them where crops can effectively use them (Right Place).

Correspondingly, compounded nutrient-enhanced nitrogen fertilizers hold significant potential for producers, given their relatively stable prices compared to the often-volatile commodity markets. By offering a compound premium, these fertilizers can help buffer against price fluctuations, presenting a compelling opportunity for producers to diversify their portfolio beyond basic products like urea or other PK commodities. Although the benefits are evident, nutrient-enhanced nitrogen granular fertilizers are rarely used due to price sensitivity, limiting larger scale growers' adoption. Additionally, wholesalers often focus on segments with higher profit margins.

Therefore, Stamicarbon is committed to delivering groundbreaking technologies for compounding nitrogen-based fertilizers, securing the future availability of nutrient- and efficiency-enhanced fertilizers. Stamicarbon is on the brink of offering its proprietary Fluid Bed Slurry Granulation technology to produce multi-nutrient-enhanced nitrogen fertilizers, with an initial focus on urea-based formulations and to be expanded to other nitrogen-based fertilizers.

Leveraging decades of experience, research and innovation, Stamicarbon's developed technology is poised to deliver an increased added value for urea-based formulations. The technological solution will feature all the proven and best-in-class advantages of Stamicarbon's globally licensed urea granulators, including extended run times, minimal formaldehyde consumption, low dust formation and emissions, high capacity, and numerous operational benefits. In addition, this new technology offers exceptional flexibility, enabling producers to efficiently manufacture a wide range of fertilizer grades on a single production line.

This technological solution is designed to enable fertilizer manufacturers and farmers to meet the increasing demand for nutrient-rich, efficient, and sustainable fertilizers. These elements are essential in guaranteeing food security and fostering responsible agricultural development.

## 2 INTRODUCTION

The use of urea in compounded fertilizers has long been a subject of investigation, driven by its global availability and affordability. This interest stems from the fundamental advantages that urea offers in terms of nitrogen content and economic feasibility. Researchers and industry professionals have explored various granulation methods and technologies to effectively utilize urea in creating nutrient-enhanced fertilizers.

The integration of urea with phosphorus (P) and/or potassium (K) sources poses considerable challenges, thereby limiting the feasibility of large-scale urea-PK production systems. A key approach to obtaining a dried final product involves minimizing water input during processing. Furthermore, other prevalent issues include diminished granule quality, marked by inadequate crushing strength and low critical relative humidity.

In 1971 Stamicarbon presented a vision for widening the field of application for urea fertilizers, which included the use of urea in mixed fertilizers. By 1974, Stamicarbon informed the Symposium audience of the development made in this field. It was summarized that unless a large amount of water for the granulation can be avoided, a large-sized dryer is required. Therefore, the dry granulation approach has been preferred, utilizing a highly concentrated urea melt and raw materials with low water content, such as solids.

Mr. C. Hoek examined several granulation methods to produce urea-PK fertilizers, including pan granulation, ammoniator-granulation, prilling, and granulation using a pugmill. All these techniques were found to be suitable for large-scale manufacturing of urea-PK fertilizers, each presenting its own benefits and challenges. At that time, the development of the Stamicarbon fluidized bed urea granulation had not yet been completed.

The Fertilizer Manual<sup>2</sup> reserves chapter 16.7 to describe the unique requirements for manufacturing urea-based granular compound fertilizer. It acknowledges the feasibility of manufacturing high-quality urea-based compounded fertilizers provided certain precautions are taken throughout the production steps. Rotary drum granulation is specifically mentioned as an option to control the agglomeration and facilitate granule formation.

A urea granulation process needs cooling systems to handle the heat generated by the molten salt crystallization. This heat can be removed by increasing the recycle ratio of solids, which adds more cold material into the granulator.

Drum and pugmill granulation use the increased recycle ratio cooling approach. The recycle to product ratio of 3:1 to 6:1 increases equipment sizing and energy use. More recycling also reduces plant capacity, raises dryer loading, and can cause screen blockages or storage caking due to increased product moisture.

In 2002, Kaltenbach Thuring S.A. showcased their design and operation of urea-PK granulation plants at the IFA Technical Conference<sup>3</sup>. These plants have single-train capacities of about 900 MTPD and utilize the agglomeration principle. This involves P-K sources being cemented by crystallizing urea solution sprayed onto a rolling bed within a fluidized drum granulator. Unlike conventional drum granulators, it includes an internal fluidized bed that provides cooling, featuring a nominal recycle rate of 1:1<sup>4</sup>.

Urea fluidized bed granulation plants are well-equipped for high-capacity single-train production units, seamlessly aligning with the output from the urea synthesis section and often surpassing 3000 MTPD. A typical dry recycle rate below 1, often around 0.6, supports this high throughput capability. Moreover, fluidized bed granules possess excellent mechanical strength, superior compared to agglomerated granules or prills, allowing them to withstand long and challenging shipping routes.

Selecting fluidized bed granulation as the technology for producing compounded urea PK fertilizers is a well-informed choice. This approach effectively addresses fundamental limitations such as insufficient drying, inferior product quality, and reduced production capacity. Despite these advantages, there are still challenges to overcome. The next section outlines the steps taken in the development of Stamicarbon's fluidized bed slurry granulation technology, to produce nutrient-enhanced nitrogen-based fertilizers.

### 3 ENRICHED UREA FLUID BED GRANULATION DEVELOPMENT PROGRAM

Stamicarbon's continuous and adaptive developments in fluid bed granulation technology aim to transform the production of urea-based compounded fertilizers. These advancements focus on product enhancement, affordability and flexibility.

The standard urea fluid bed granulation creates high-quality granules through layering, unlike agglomeration used in other methods. Each sprayed layer solidifies before adding a new melt layer, requiring a highly concentrated urea stream for quick crystallization. Rapid crystallization prevents sticky granules from agglomerating to lumps.

Due to Stamicarbon's proprietary film spraying technology, the process results in a product characterized by superior quality and operational advantages, such as minimal dust formation that supports up to 2-3 months of continuous operation without the need for cleaning.

An important technical consideration when using fluidized bed technology for granulating a mixture of molten urea and other plant nutrients, typically a solid mineral salt, is that the solid often does not dissolve or barely dissolves in the urea melt.

<sup>2</sup> T.P. Hignett (Ed.), *Fertilizer Manual*, 1985, <https://doi.org/10.1007/978-94-017-1538-6>.

<sup>3</sup> G. Chu & F. Ledoux, "Process technology of urea - based NPK and practical experience of industrial plants," [https://www.fertilizer.org/wp-content/uploads/2023/01/2002\\_tech\\_quifan-chu.pdf](https://www.fertilizer.org/wp-content/uploads/2023/01/2002_tech_quifan-chu.pdf).

<sup>4</sup> Patent US4749349A, <https://patents.google.com/patent/US4749349A/en>.

This results in the dispersion of solids in urea, creating a slurry that might fundamentally change the urea melt's properties, such as viscosity and density, and/or crystallization temperature.

Additionally, the undissolved solids in the granulating feed stream need to be within a specific particle size to be effectively incorporated into the granules by the layering mechanism.

Provided that these changes are effectively addressed, it is anticipated that fluid bed granulation of compounded urea-PK fertilizer will adhere to the same high standards of quality as commercially available Stamicarbon urea granulators.

### 3.1 Nozzle

The previous section generally addresses the working principle of Stamicarbon's proprietary thin film nozzle. The shape of the urea film is a hollow cone; the shape and thickness of the film is characterized by the nozzle geometrical dimensions and the urea flowing properties, as shown in Figure 2.

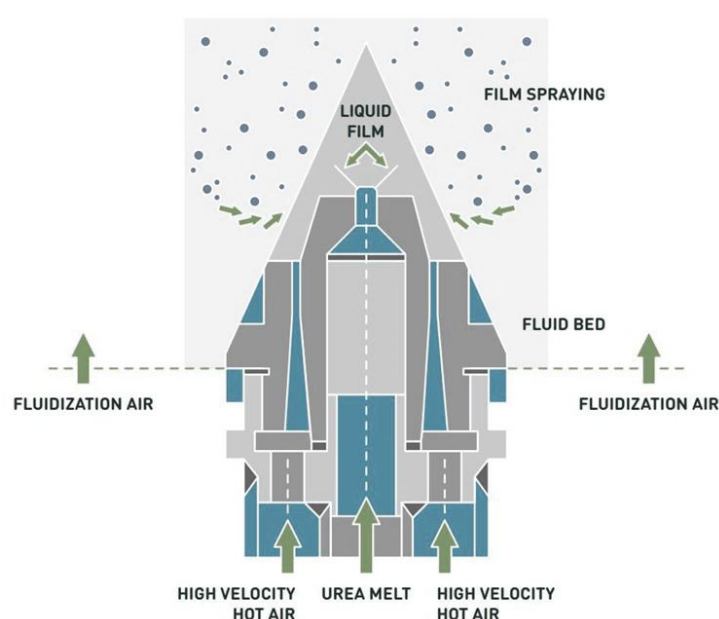


Figure 2: Representation of standard nozzle geometry and working principle with urea melt.

Adding solids to the urea melt changes its flow properties and affects film characteristics. To address this, a slurry nozzle development program was launched. The initiative was systematically divided into multiple stages, each contributing to the comprehensive understanding and advancement of slurry nozzles. The development process relied extensively on Computational Fluid Dynamics (CFD) simulations for the design of nozzle geometry. CFD simulates fluid flow and its interaction with surfaces defined by boundary conditions. It facilitates the study of multiphase flows through numerical solutions of the Navier-Stokes continuity equations for a given system.

The CFD simulations utilize a Volume of Fluid (VOF) model under steady state conditions. The VOF model represents multiple immiscible fluids by solving one set of momentum equations and tracking the volume fraction of each fluid in the domain. A steady-state VOF simulation is suitable when the solution is independent of the initial conditions and there are distinct inflow boundaries for the individual phases. For example, the flow of water in a channel with a region of air on top and a separate air inlet can be solved with the steady-state formulation.

The system's shape and boundaries are based on the CAD drawing of the standard nozzle, which helps define fluid volume and changes in shape. Figure 2 depicts the general geometry of the standard Stamicarbon nozzle.



As an initial step, CFD simulations were conducted to evaluate the performance of the reference nozzle with simulated parameters for liquid viscosity, density, and flow pressure. Figure 3 displays the results of this CFD simulation, which serve as a benchmark for a stable and functional hollow cone film. The purple color in the images below indicates the area where air is present, and the sprayed film is shown in red.

A stable film is observed forming after exiting the nozzle orifice and reaching a specified height; the film thickness subsequently decreases with increasing distance from the orifice.

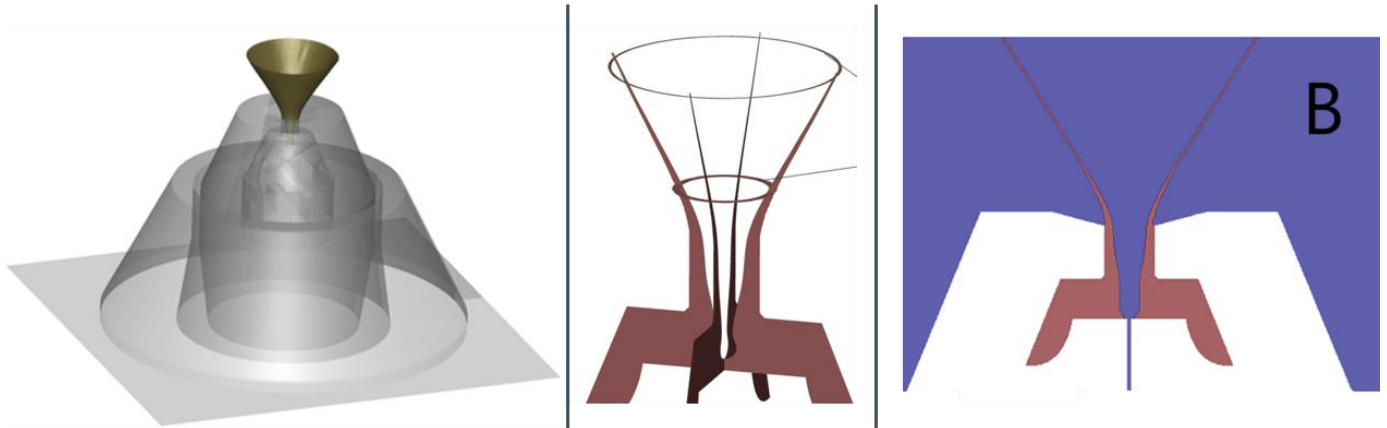


Figure 3: CFD representation of standard nozzle and urea hollow cone formation (colored cross-sections of liquid volume fraction).

Findings showed that hollow cone formation fails above a certain viscosity threshold, as shown in Figure 4. When comparing images from left to right, it is possible to observe the effect of increased viscosity on the spray behavior of the nozzle. At lower viscosities (see left figure), the air is still present deep into the nozzle body. On the other hand, at higher viscosities the presence of air decreases to the point where only a liquid jet is visible above the nozzle exit (see right figure). Changes in pressure and density did not significantly affect film formation compared to the standard case.

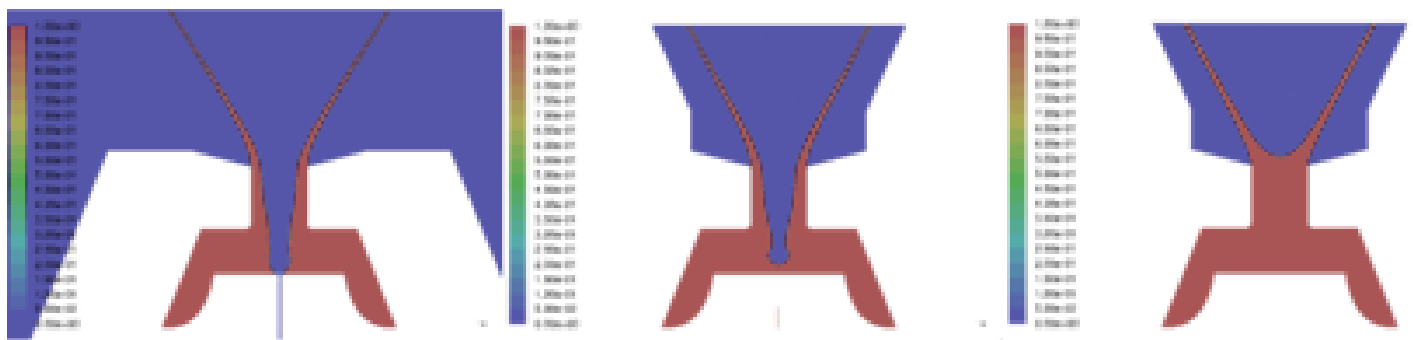


Figure 4: CFD simulations depicting the effect of increased viscosity from (L) to (R) (colored cross-sections of liquid volume fraction).

The inclusion of solids in the melt is anticipated to increase fluid viscosity by a factor of 10 to 100 times. This increase aligns with suitable solids loading for practical and commercially viable concentrations of multi-nutrient fertilizer grades. Therefore, it has been concluded that the standard Stamicarbon urea nozzle is not suitable for achieving optimal performance during slurry granulation.

In a second stage and derived from the initial results, two new slurry nozzles were designed: one for high-viscosity slurries and another for low- to medium-viscosity slurries. Multiple CFD simulations were carried out to optimize the nozzle geometry, resulting in hollow cone film characteristics that closely match the standard characteristics achieved with pure urea.

Furthermore, the design phase incorporated an enhancement in nozzle throughput. Slurry nozzles were engineered to operate at a nominal capacity of 450 kilograms per hour, which is double the standard urea nozzle capacity. The benefits of increasing the nozzle's capacity are significant. They can be utilized in installations with fewer nozzles, enabling the upgrade of various older installations, including non-Stamicarbon-licensed granulators.



In the third stage, both prototypes were manufactured to be tested. The first validation was carried out at a lab scale; a simulated liquid with an increased viscosity, glycerin in water, was sprayed to validate the CFD simulations (see Figure 5). The results predicted by CFD matched the experimental results within a reasonable range, and it was decided to proceed to the last development gate.

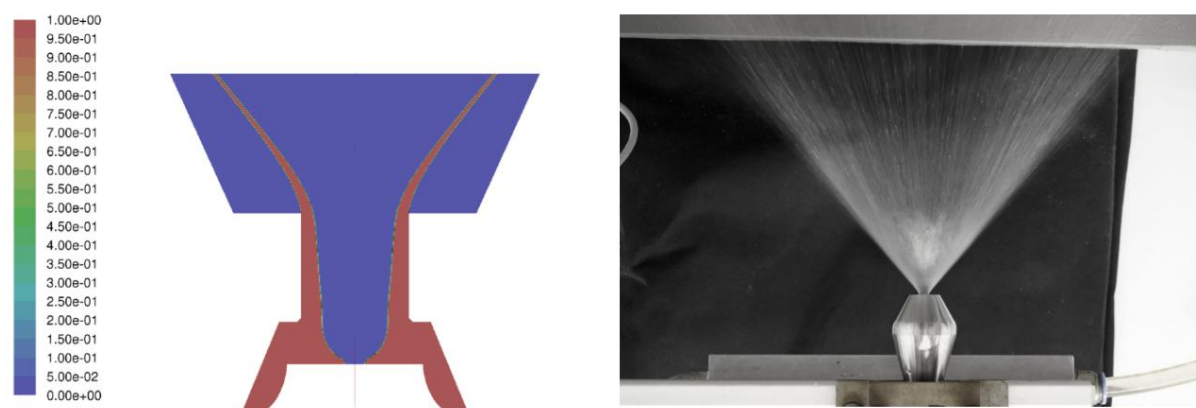


Figure 5: Slurry nozzle hollow cone film at increased fluid viscosity.

The slurry nozzles were tested in a pilot plant for proof of principle (POP) and scale-up testing of urea enriched with multi-nutrient solid mineral. For details, see Section 4.

## 3.2 Solid characterization

The success of slurry granulation is determined by several critical solid parameters. Proper handling of solids is crucial, as poor flowability requires special measures for transportation, storage, and dosing. Similarly, effective wetting of solids is essential; inadequate wetting behavior requires specific strategies to achieve homogeneous dispersion in the urea melt, as depicted in Figure 6 below.



Figure 6: Urea and nutrient(s)-bearing solid well-dispersed in urea melt (L) and settled (R)

The settling speed of particles is another key factor; high settling rates demand measures to maintain suspension homogeneity and prevent blockages in lines, valves, and nozzles.

As mentioned in the previous section, the viscosity of the resulting suspension is vital; high viscosity can negatively impact the efficiency of the granulation nozzle.

Additionally, particle size distribution (PSD) plays a significant role; a wide PSD, particularly when containing large particles, can adversely affect granulation performance. This point will be further addressed in the following section.

### 3.3 Urea-solid compatibility

The chemical reactivity between urea and solid materials is a crucial aspect that dictates the efficiency and outcome of the granulation process.

In general, urea is a weak base that undergoes protonation with acids, but it can also act as a Lewis base. At temperatures up to 190 °C, urea decomposes into ammonia and isocyanic acid, leading to compounds like biuret, cyanuric acid, and ammeline. Urea hydrolysis produces ammonia and carbon dioxide.

The selection of solids and conditions for mixing with molten urea must be carefully controlled to prevent unwanted reactions. Stamicarbon has developed extensive knowledge on the chemical compatibility of different solids to be incorporated in urea for NPK compound fertilizers, which is especially crucial when incorporating phosphate sources.

Phosphate fertilizers commonly cause a polycondensation reaction with molten urea. First, ammonium phosphate protonates urea. The protonated urea then acts as an electrophile, attacking another phosphate molecule. This forms a urea-phosphate intermediate, which combines with the initial phosphate, leading to the growth of the ammonium polyphosphate chain<sup>5</sup>.

As experimentally proven during the development program, the reactivity of orthophosphate with urea highly depends on the acidity of the phosphate source, and its availability to act as proton donor to urea and initiate polycondensations mechanisms.

Stamicarbon has developed methods to control phosphate basicity that limits urea decomposition while ensuring nutrient availability. This involves selecting solids with suitable properties, modifying mixing conditions, adjusting orthophosphate chemistry, or using a combination of these approaches.

Furthermore, selecting potash sources presents its primary challenges not only within the realm of technology processing, but also in agronomy.

The nitrogen-to-potassium (N:K) ratio is particularly important during the transition of plants from the vegetative growth phase to the generative phase, characterized by flowering or fruit-bearing. As fruits are rich in potassium, it is imperative to maintain an adequate supply of potassium during generative periods to ensure optimal development<sup>6</sup>.

Potassium chloride (KCl) is the most frequently used form of potassium in agriculture. However, certain crops are sensitive to chloride and are adversely affected by soil salinity. Salinity impacts plants in two significant ways: toxic stress and osmotic stress.

Some typical sensitive crops are beans, tomatoes, peas, apples, grapes, strawberries, lettuce, cucumbers, avocado, citrus fruits, tobacco among other crops. Fertilization practices suggest the use of low-chloride potassium in fertilizers in sensitive crops, but also in saline soils and saline irrigation water or any combined situation.

Despite this general recommendation, potassium chloride (KCl or MOP) is the major fertilizer used as the source of potash ( $K_2O$ ) over commercially available potassium sulfate ( $K_2SO_4$  or SOP) fertilizer or others. The main disadvantage faced by SOP is the relatively higher cost per kg of  $K_2O$ .

Salt tolerance data guides crop management to minimize costs while maintaining yield. These guidelines are useful for moderately sensitive or tolerant crops, but sensitive crops have limited flexibility due to quickly reaching toxicity thresholds.

Furthermore, high chloride content can be highly corrosive to processing equipment and piping. Utilizing potash sources with low chloride levels helps mitigate technological and agronomical adverse effects. There are various potential additives for providing low-chlorine potassium, in addition to standard produced sulphate of potash (SOP), such as evaporative salts with the potential to provide reduced carbon footprint.

<sup>5</sup> Yang, J. (2019). Evolution of the polydispersity of ammonium polyphosphate in a reactive extrusion process: Polycondensation mechanism and kinetics. *Chemical Engineering Journal*, 1453-1462.

<sup>6</sup> A.M. Van Der Zanden, "Environmental factors affecting plant growth," <https://extension.oregonstate.edu/gardening/techniques/environmental-factors-affecting-plant-growth>.

Stamicarbon's development seeks to facilitate the use of non-conventional sources, reducing carbon footprint and supporting circularity.

### 3.4 Solid particle size

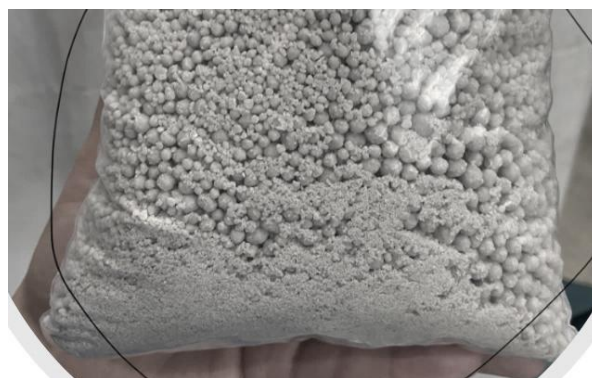
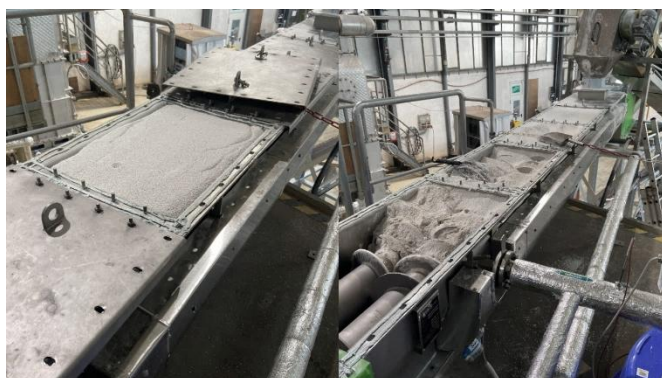
The solid feedstock to the urea melt must be as fine as possible while retaining acceptable flowability. Various PSDs were tested during the development program. For instance, Figure 7 illustrates the visual difference between "fine" and "coarse" grades.



*Figure 7: Differences in raw material PSD grades.*

For processing purposes each grade possesses advantages and disadvantages, the most evident being the poor flowability in the fine grade leading to overfilling of the solids handling equipment, as depicted below in Figure 8, left-hand side.

On the other hand, the coarse grade exhibited the well-known parasitic feeding effect, which is characterized by excessive production of undersized granules, which are returned to the granulator inlet, resulting in high dry recycle ratios.



*Figure 8: Overfilling of the screw heater (L) and parasitic seeding effect (R).*

Parasitic seeding refers to the introduction of large solid particles into the granulator through the slurry, which act as extra seed material and alter the particle balance number within the granulator. This process typically results in a product with smaller particle size.

This phenomenon is commonly observed together with a high and preferential solid dust entrainment. Figure 8, right-hand side, shows the granulator outlet under parasitic seeding conditions. Particle size optimization and reduction will be discussed in the following sections.



## 4 FLUID BED SLURRY GRANULATION PILOT PLANT TEST

### 4.1 Polyhalite as solid compounding material

Polyhalite is a mined evaporative rock (see Figure 9<sup>7</sup>) with formula:  $K_2SO_4 \cdot MgSO_4 \cdot 2CaSO_4 \cdot 2H_2O$ . It combines four different and essential nutrients:  $K_2O$  (14%), S (19%),  $CaO$  (17%) and  $MgO$  (6%). Compounding urea with polyhalite provides flexibility to produce different grades of multi-nutrient-enhanced urea fertilizer, making use of a less conventional fertilizer nutrient source.



Figure 9: Polyhalite mined rock.

### 4.2 Proof of principle

The main aim was to demonstrate the feasibility of co-granulating polyhalite made available in different PSD grades with urea using Stamicarbon's proprietary fluid bed slurry granulation technology for continuous operation.

The tests showed that it is possible to produce a urea-polyhalite granulate of good quality and homogeneous appearance, as depicted in Figure 10 clearly demonstrating the incorporation of polyhalite into the granule.

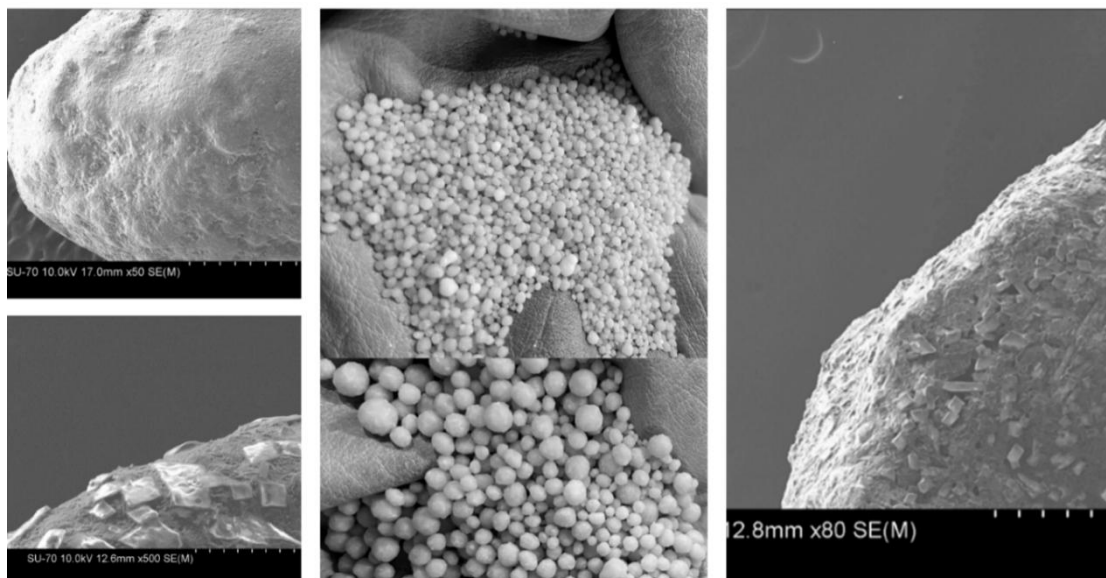


Figure 10: Incorporation of polyhalite particles into granule.

The evaluated product quality parameters for the urea-polyhalite product – like crushing strength – are very comparable to those for pure urea.

However, the granulator outlet consistently produced a product with a fine PSD, resulting in a coarse product recycle occasionally reduced to zero and fines recycle flowrate increased to unacceptably high level.

<sup>7</sup> T. Heap, "More than 1km under the North Sea is a climate-friendly mineral that could help feed the world," <https://news.sky.com/story/more-than-1km-under-the-north-sea-is-a-climate-friendly-mineral-that-could-help-feed-the-world-13047223>.  
Gerardo Duarte / Nutrient-enhanced urea production

The proof-of-concept testing encountered multiple equipment malfunctions and downtime, inherent to the endured learning curve associated with a novel operational approach. Most of them related to solid and slurry handling issues, which were eventually solved.

The limited granulation time due to downtime issues prevented conclusive results. Few definitive conclusions were reached:

- The dust entrainment increased by increasing both polyhalite concentration and particle size.
- All tested nozzles produced a co-granulated final product.

The influence of the granulation nozzle type on stability and performance was not determined, as inconsistent variations were observed, likely resulting from unexpected stops and restarts. However, the available data suggested that the standard nozzle quickly reached its operating limit as raw material load and particle size increased. This aligns with the presented CFD simulations, which show an upper operating limit at a specific viscosity threshold, which is mainly driven by solids concentration. Both newly designed slurry nozzles showed lower entrainment and larger particle growth compared to the standard urea nozzle but did not differ clearly from each other. It was decided to move forward with scale-up testing.

### 4.3 Scale-up

The scale-up test was designed based on observations, lessons learned, and results derived from the proof-of-principle test.

The main goals of this scale-up test campaign were to determine the optimal settings for the wet milling operation and identify the best slurry granulation nozzle. The goal was to match and maintain granulation performance and product quality equal to pure urea granulation standards.

To achieve sufficiently small particles, it is necessary to use a two-stage system of wet mills in series. The first stage, mill #1, should reduce the particle size by a factor of 2-3, while the second stage, mill #2, should further reduce the particle size by a factor of 3-5, all relative to the inlet stream at each stage.

The temperature increase over the mills was observed and correlated with granulation performance. When the milling intensity is high, indicated by a noticeable temperature increase in the slurry (see Figure 11), the granulation performance improves, and particularly dust entrainment and fines recycling are reduced.

The temperature increase over the mills should be monitored and controlled to avoid further biuret formation. A cooler placed downstream the wet milling section is used to bring the melt temperature down to typical granulation values.

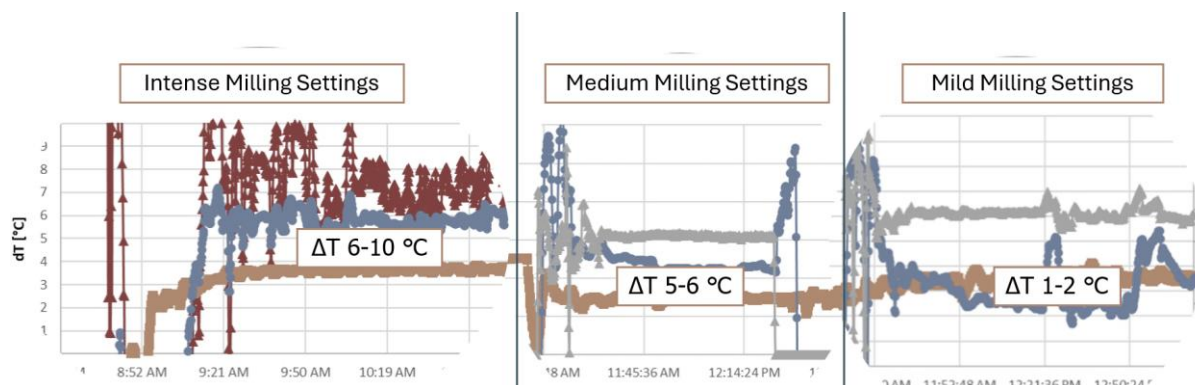


Figure 11: Temperature increase over online wet milling section at different settings.

On a given day and under exact previously tested intense milling settings there was a noticeable drop in the temperature increase (dT) during the test, as can be clearly seen from Figure 12 below. The observed dT trend correlates with the worsening of the granulation performance throughout the experimental run. The following day, the milling settings were reinstated, and another granulation attempt was made. This time, the observed temperature increase nearly disappeared (see Figure 12, right-hand side).

The reason behind the decline in temperature rise over the mills is directly correlated to the loss of milling actions due to the erosive wear later identified in the milling tools.

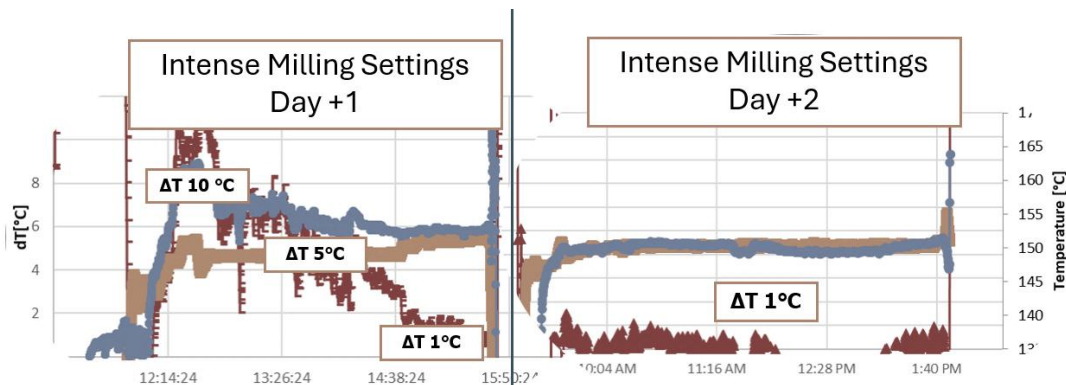


Figure 12: Drop of temperature increase during the test at the same milling intensity.

The milling tools (stator and rotor) of both wet mills were replaced with tools made of much harder materials, capable to withstand erosive wear (described in detail in Section 5.1). Furthermore, it was decided to execute another week of testing with the refurbished mills to achieve the campaign targets.

The produced urea-polyhalite granulate had a good, uniform appearance with an average PSD of d10 below 2.2 mm, d50 around 2.9 mm, and d90 about 3.8 mm. During the test, dust entrainment averaged just below 5% of the production rate, and the total dry recycle rate (including over- and undersized products) averaged just below 100% of the production rate (see Figure 13 for visual reference).

These results are within the upper range of the process performance target and may be reduced by optimizing gap settings at the coarse recycle crusher, thereby improving the granulation seeds quality.



Figure 13: Slurry granulation particle growth over time.

The quality parameters measured for the urea-polyhalite granules are largely comparable to those of pure urea granulate. The crushing strength was reported to be between 25-35 N, which is similar to the crushing strength of pure urea (~35 N). The nutrient contents of the final product closely align with theoretical values based on the polyhalite mass flow setpoint.

The SEM image of the granule from the proof-of-principle test, as depicted in Figure 10, reveals the presence of large polyhalite particles, ranging in the hundreds of microns, protruding from the surface of the



granule. Conversely, when a two-stage wet milling operation is applied in series, some polyhalite particles incorporated into the granule can be reduced to single-digit micron size, as depicted in Figure 14.

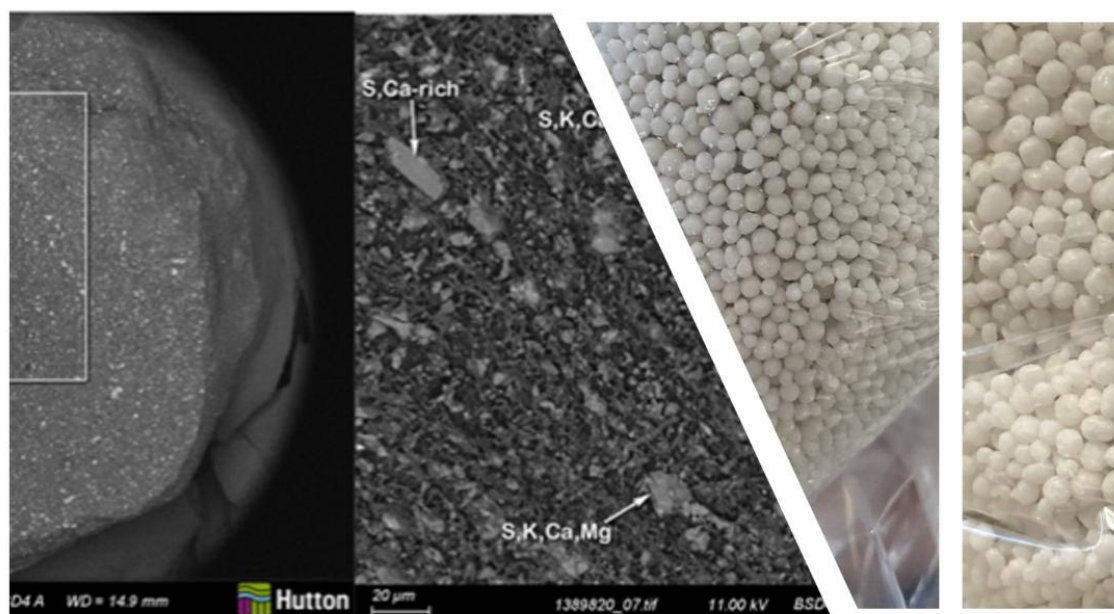


Figure 14: Scale-up testing SEM image of high-quality urea-polyhalite granule.

The standard urea nozzle is not suited for higher polyhalite concentrations in the slurry – 20%wt and above – while both newly designed slurry nozzles are well-suited for the 20-40%wt range, and likely beyond.

The low- to medium-viscosity slurry nozzle type demonstrated optimal granulation performance parameters. Notably, the use of this nozzle type ensures a reliable transition from compounded to standard urea, thereby enabling the implementation of a multipurpose granulation line.

The final tests resulted in a positive outcome:

- An acceptable level of dust entrainment
- Acceptable levels of oversized and fines recycles
- No lump or agglomerates formation
- No fouling of the granulator
- Good product quality

Scale-up testing revealed challenges crucial for a commercially viable implementation of technology, primarily the wear on milling tools. Finding durable materials for the milling tools and slurry nozzles that resist erosion at a feasible effort and cost is essential. Additional information will follow in the next section, outlining the steps taken to identify appropriate material.

Another critical finding is the lower critical relative humidity (CRH) measured in the compounded urea-polyhalite final product, relative to pure urea. Stamicarbon confirmed the low CRH values of 55-65% through multiple analytical service providers.

This quality concern is inherent to the urea-polyhalite compounded product. Stamicarbon can provide a specific solution by coating the granules with a liquid agent containing surfactants and long chain hydrocarbons, reducing condensed water ingress. These added features limit moisture absorption, making it suitable for bulk granular handling.

The urea-water-polyhalite mixture gave rise to corrosion concerns. Corrosion results support the use of SS304L as the preferred, cost-effective material for both scrubber and evaporator inlet service, provided that regular inspections are performed and targeted upgrades are applied where needed.



## 5 THE FUTURE OF FLUID BED SLURRY GRANULATION

### 5.1 Material selection

During the pilot plant test, various materials were evaluated for their potential use in milling tools and granulation spray nozzles.

Initially, standard stainless 316L steel was used as construction material for milling tools in mill #1 and granulation nozzles. It showed significant erosive wear shortly after starting operations. Similarly, the super duplex stainless-steel type used for mill #2 tools also experienced rapid erosive wear.

The granulation spray nozzles were subsequently upgraded to a surface hardened stainless steel, which exhibited significantly reduced erosive wear compared to standard stainless steel. The enhanced durability of the hardened nozzle material indicates its potential suitability for commercial-scale applications.

Subsequently, mill #1 was upgraded using a surface hardened super duplex stainless steel, while mill #2 utilized a steel superalloy. Although these choices demonstrate enhanced erosion resistance, the observed improvement may not be adequate for commercial applications. The material upgrade doubled the nominal hardness.

A further increase in wear resistance, more than a fivefold increase, can be achieved by manufacturing milling tools from cemented carbide composites, as long as the brittleness is properly balanced to withstand the stress and deformation forces experienced during wet milling. Wet milling uses high-speed rotor-stator blades to draw in solid suspensions and mill them between the rotor and stator, generating strong shear forces.

The rotor and stator are inherently prone to wear due to mechanical action, demanding periodic replacement of parts. During the development program, preferential wear in the rotors was identified, indicating that anti-wear material reinforcement may be required only in specific areas.

In general, the extent of wear is primarily affected by the hardness of the solid particles and the material composition of the rotor and stator. Furthermore, optimizing operational parameters including power input, solids load, particle size, and viscosity is essential for reducing the forces that lead to wear.

By evaluating all relevant factors influencing wear, various material options can be recommended to address the different requirements for processing multiple solids through slurry granulation technology.

### 5.2 Technology outline

The slurry granulation technology has been validated through experiments based on an initial process concept, which was largely shaped by the operational limitations of the experimental pilot plant setup. Through continuous development, that initial slurry granulation concept has evolved into multiple-process configuration designed to offer simplicity and flexibility.

#### 5.2.1 Standard solid dosing

Figure 15 illustrates the fundamental process for producing a compounded nutrient-enriched urea granule. It features the benefits of incorporating aqueous urea solution in the slurry production process. Some key advantages include improved heat utilization, simplified equipment specifications, and enhanced processing of solids. The process concept avoids crystallization issues and enables higher solid incorporation into the final product.

Utilizing aqueous urea solution enables operation at temperatures below 100°C, unlike using pure urea melt, effectively reducing adverse effects linked to temperature elevation during wet milling. This approach eliminates the requirement for a cooling step that was imperative during pilot plant testing with concentrated urea melts.

Consequently, the heat generated from mechanical treatment remains available within the process. This offers a synergistic benefit during the water removal phase, as it reduces the amount of heat required for

evaporation. More importantly, the use of urea solution decreases the formation of the unwanted byproduct biuret during mechanical treatment.

Figure 16 presents further process integration with the off-gas scrubbing section and scrubbing liquid recycle. The off-gas from fluidized bed granulation is generally laden with fertilizer containing dust comprising urea and one or more fertilizer macronutrient sources added in the process. By subjecting the off-gas to dust scrubbing, fertilizer is recovered and can be recirculated upstream of evaporation section back into the process.

The water removal step can be combined with recycling scrubber liquid, enhancing heat transfer and water removal efficiency through economy of scale in a single evaporation section.

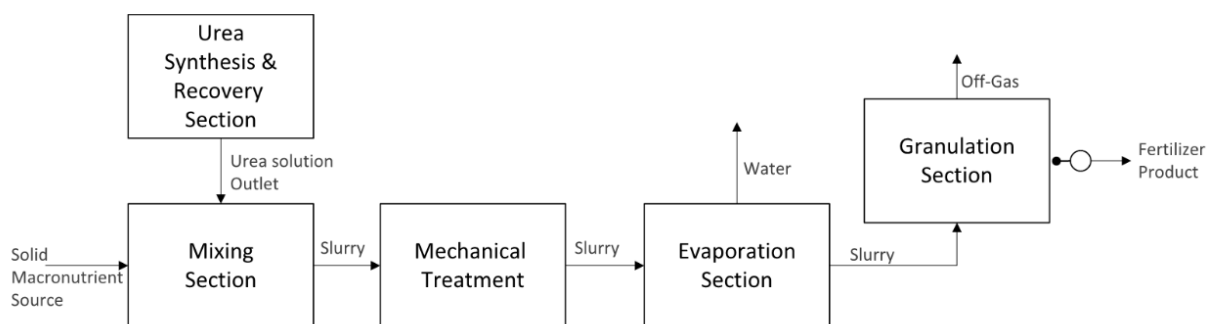


Figure 15: Urea solution slurry granulation (general).

At the end, the method produces urea compound fertilizer granules containing urea and at least one macronutrient source. These granules now include at least one or multiple macronutrients such as phosphorus (P), potassium (K), sulfur (S), calcium (Ca), and/or magnesium (Mg). The concentration of each plant macronutrient is evenly distributed throughout the granules' cross section.

The uniform distribution of nutrient concentration within the solid product provides significant agronomic benefits. This advantage is primarily achieved through the integration of slurry mechanical treatment combined with fluid bed granulation. That is, fluid bed slurry granulation.

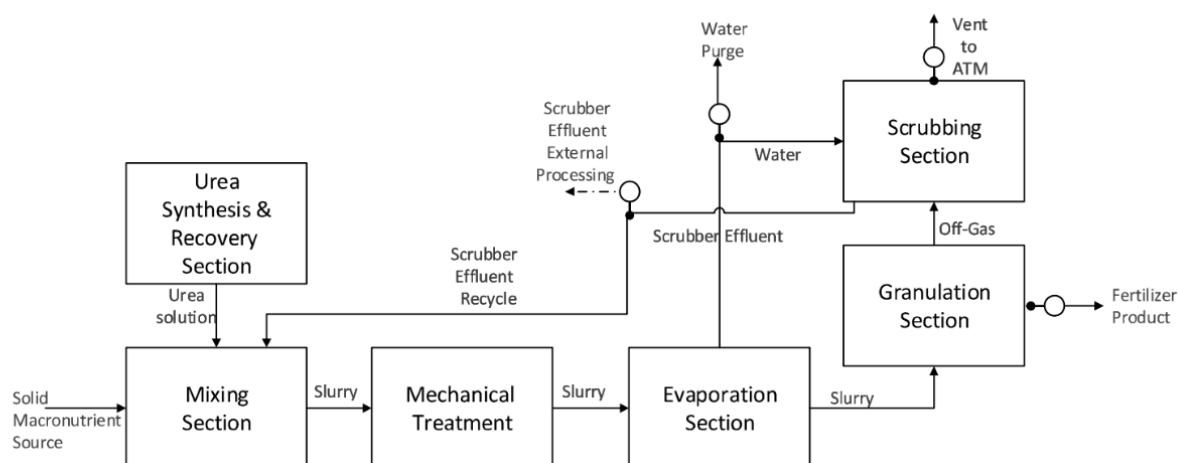


Figure 16: Urea solution slurry granulation (integration).

## 5.2.2 Low solid dosing

This design focuses on urea enrichment with solid micronutrients, efficiency enhancers, bio stimulants or granulation additives, where the solids dosing is low, typically below 5% by weight of the granulation plant throughput.

A key aspect of the design involves the split of urea melt feed. In this process, solids are fed and mixed into a reduced side stream, which is subsequently reintegrated into the main flow, as depicted in Figure 17.

The objective is to maximize the total solids content in the side-stream, up to 50% wt. or more. The splitting ratio of the urea melt feed is carefully chosen to ensure that the available urea volume sufficiently promotes wetting, dissolution and or dispersion of solids, while also minimizing residence time and thus limiting biuret formation. Ultimately, the ratio between these two streams is determined by: 1. The desired concentration of solids downstream mixing section in kg/mt, and 2. The desired concentration of solids in the mixing section in kg/mt.

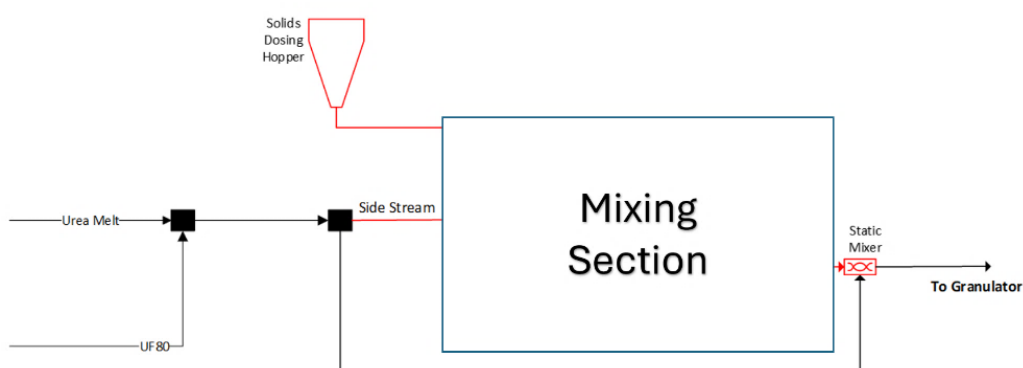


Figure 17: Low solid dosing on split feeding.

### 5.3 Technology added value

In both discussed dosing scenarios, the process concept can be implemented in urea granulation plants, whether it involves constructing a new facility (grassroot) or modifying an existing one (revamp).

The value proposition of the presented fluid bed slurry technology focuses on urea de-commoditization by integrating market-valued plant nutrients with urea to produce specialty fertilizers. This approach enables flexible manufacturing of diverse product formulations in a single multipurpose granulation line.

This innovative approach presents several benefits. Specialty fertilizers can mitigate price fluctuations, offering producers a compelling opportunity to diversify their portfolio beyond basic products such as urea or other PK commodities.

Economic success will not only depend on commodity pricing but also on production cost effectiveness. This refers to the technology's ability to enhance the overall production capacity of the urea-solid compounded fertilizer. By integrating solids containing plant nutrients with urea using Stamicarbon's slurry granulation technology, the process effectively increases the weight-based capacity of a production train.

Converting a urea granulation facility into a specialty fertilizer production unit can increase the original production capacity by approximately 20-30%, depending on the solid load and specific characteristics of materials.

In essence, the fluid bed slurry granulation technology presented in this paper offers the possibility to produce larger quantities of higher-value products, thereby achieving higher profit margins with fertilizer production.

## 6 CONCLUSIONS

In conclusion, slurry granulation technology offers a robust solution for processing multiple solids with varying compositions and characteristics. By addressing key factors such as material hardness and optimizing operational parameters, this technology can ensure maximized plant throughput at acceptable wear rates.

The solid dosing in the process configuration allows for the incorporation of multiple fertilizer grades, from pure urea to formulas tailored for nutrient enrichment and improved efficiency. The validated experimental concept has successfully transformed into a versatile multiple-process framework, making it a valuable asset in the field of enriched urea production.

Stamicarbon remains committed to continuous improvement and innovation, ensuring that its technology meets the evolving needs of the industry and delivers consistent performance. Explore the benefits of Stamicarbon's slurry granulation technology and experience the advantages of advanced solid processing firsthand.

Stamicarbon B.V.

REGISTERED OFFICE

Mercator 3, 6135 KW Sittard,  
The Netherlands  
P.O. Box 53 - 6160 AB Geleen  
P +31 46 4237000  
F +31 46 4237001

[g.duarte@nextchem.com](mailto:g.duarte@nextchem.com)  
R&D Engineer

[stamicarbon.com](http://stamicarbon.com)

