

IFA Technical Conference

Beijing, China 20-23 April 2004

MANUFACTURE OF UREA-BASED COMPOUND FERTILIZER BY FLUIDIZED-GRANULATION

Li Tianwen, Dai Zhiqian and Liu Hongsheng Lutianhua Group Inc., Luzhou, China

ABSTRACT

A novel process of making non-uniform granular urea-based compound fertilizers of concentrated nutrients by fluidized-granulation is developed and tested. It is shown that this technology may bring out a breakthrough in the production of urea-based compound fertilizers.

Keywords: non-uniform granular urea-based compound fertilizer, fluidized-granulation, urea, MAP, DAP, TSP.

I. INTRODUCTION

With the development of modern agriculture more and more concentrated compound fertilizers are needed in planting instead of straight fertilizers. Although in the world and domestic markets urea remains the principal source of fertilizer nitrogen, urea is overproduced and its supply exceeds the demands year by year. Many producers are seeking for new ways to produce brand new products utilizing more urea. Therefore, urea-based compound fertilizer with high nutrient content appears to offer the maximum saving in costs of sale and application among the varieties of compound fertilizers. The key to making granular urea-based compound fertilizers depends on the granulation technique. Reportedly Hydro Fertilizer Technology B.V. practiced a pipe reactor that supplies ammonium phosphate and urea directly to a drum granulator. This process can produce granular urea-based compound fertilizers with different formulation of nitrogen, phosphorous and potassium.

However, the process has several drawbacks in reality. Firstly the addition of phosphate to the granulation system has the disadvantage of reducing the melting point of the mixture, which necessitates using lower temperature for drying. Secondly granulation may become more difficult as the percentage of urea in the formulation is increased. Thirdly, excessive amount of filler is required in granulation, so that granulation is deteriorated and the percentage of nutrients in formulation is decreased. Some Chinese chemical engineers put forward their granulation techniques for commercial manufacture of urea compound fertilizer. Pan/drum granulator and oil bath prilling are usual design of equipment. Nevertheless, hydrolysis of urea and slurry adherence in the operation are the main problems still to be resolved. So far there exists no unit with the capacity over 100 thousand tons.

Unlike the other kinds of compound fertilizers such as nitro phosphates, urea-based compound fertilizers are encountered with some difficulties in large-scale production due

Received for presentation at the 2004 IFA Technical Conference, 20-23 April, Beijing, China,

Address: East Section, Jianshe Road, 066003 Qinhuangdao, Hebei Province, China

Tel: +86 335 3161088

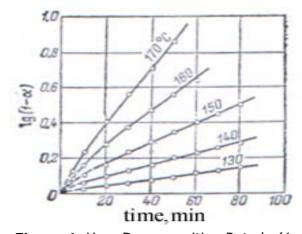
to its intrinsic physical and chemical properties. Though many efforts are made to break this bottleneck, no satisfactory technology is available to date.

The paper presents a brief introduction of our novel experimental process of making urea-based compound fertilizer by fluidized-granulation.

2. PHYSICOCHEMICAL FUNDAMENTALS

Chemically urea usually prone to produce high hygroscopic and eutectic salts when combined with other straight fertilizers, that is deleterious in the granulation process of urea-containing compound fertilizers. For example, urea mixed with ammonium phosphate may form a thick slurry with a melting point of $115\Box$, which is difficult to be solidified and easy to scale on the granulator wall. To overcome this more filler is stuffed in the compound fertilizer that results in the decrease of total effective nutrition and increase of transportation cost.

Compared with other phosphates like DAP, SSP and TSP, reactions of MAP with urea were studied more thoroughly. Thermal decomposition of urea-MAP composite proceeds at relatively low temperature ($115\sim120\Box$) due to increased content of urea. In the presence of urea ammonium phosphate dehydrates at temperature between $115\sim180\Box$, much lower than that of pure MAP (hydrolysis temperatures $200\sim350\Box$). MAP accelerates the decomposition rate of urea (Fig. 1) [1]. The composition of material disassembled from the mixture of MAP and urea is very complex. Also urea can stimulate the dehydration of MAP (Fig. 2) [1].



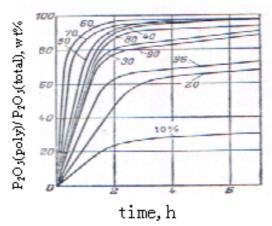


Figure 1. Urea Decomposition Rate lg (1- α) vs Time τ (N:P2O5=3:1).

Figure 2. MAP Hydrolysis Rate vs Time at 120□ with Various Percentages of Urea.

Furthermore, when urea and any super phosphate is blended, double salt CO(NH2)2·Ca(H2PO4)2 can be formed and releases crystallization water originally combined in Ca(H2PO4)2·H2O[2]. Another handicap is that no sufficient potassium salts such as potassium chloride, potassium sulfate can be dissolved in the urea melt when high concentration potassium fertilizers are needed.

According to the above analysis, we may conclude that it is not an easy task to granulate urea-based compound fertilizer containing phosphates by the way of melt slurry of urea and phosphate because of the occurrence of strong dehydration.

Traditional processes of the granulation of urea-based compound fertilizers are stuck to the idea of producing so-called uniform granules with uniform distribution of all nutrient elements in each granule. This idea cumbers the development of fluidization technology in the granulation of urea-based compound fertilizers though that technique has been practiced industrially in the manufacture of granular urea. The reason is that in the fluidized granulation process, melt or concentrated solution with few solid impurities must be prepared and the spraying nozzle must be made of high abrasion-resistant material. Unfortunately the development of such high-tech material is still not under way. Moreover, urea melt undergoes hydrolysis when ammonium phosphate or other inorganic impurities are present and tends to form biuret that is harmful to the sprouts of some crops.

However, noticing that fluidized granulation technology shows superiority to other granulation techniques by its large capacity, less energy consumption, lower investment, easy handling and high quality of product, more and more urea producers adopt this process to make granular urea of better product quality. Hydro Fertilizer Technology B.V., Toyo Engineering Co., Kaltenbach-Thuring SA have won the technical market in fluidized bed granulator, spouted bed granulator and fluid-drum granulator for many years both in China and other parts of the world. Many researchers have contributed a lot to get breakthrough in granulation of compound fertilizer by using fluidization. Brusasco et al.[3] of Agrimont in 1980's employed a spouted bed in granulation-drying of ammoniated slurry from nitric attack of phosphate or sulphuric-phosphoric acid mixture and carried out their industrial test on the granulation of diammonium phosphate and monoammonium phosphate. The production capacity of their pilot plant based on a single spouted bed was from 500 to 700t/d. But it is incapable of using urea as the main nitrogen source. Shakhova et al.[4] of pre-Soviet Union in 1969 made significant progress in this aspect by using a two fluid spray jet in a conical fluidized bed to produce layered granules of urea-based compound fertilizer. Because of the large difference between urea and ammonium phosphate, decomposition of the former takes place more seriously, and industrialization of this process was not reported afterwards.

Comparison of the different granulation processes of compound fertilizers suggests a promising prospect of fluidized-granulation technique in fertilizer industry[5]. It seems that application of fluidization-granulation in urea-based compound fertilizer is constrained by the traditional idea of making uniform composite fertilizer of homogeneous composition. On the contrary we have discovered the way to make non-uniform urea-based compound fertilizer by coating a thick layer of urea melt on the particles of phosphates or other composites. We call it non-uniform urea-based compound fertilizer to distinguish it from that by other processes[6]. Since most of plants absorb and digest the nutrients only gradually, this kind of fertilizer can applied to both agriculture and horticulture.

3. PROCESS DESCRIPTION

A brief description of our experimental process is as follows (Fig.3). It consists of a cylindrical fluidized bed granulator $(300 \text{mm}(\text{diam.}) \times 1,800 \text{mm}(\text{height}))$ with a conical base section, two blowers separately connected with electric exchangers in order to generate hot air for fluidization and spouting, a cyclone to capture the dust resulted from

granulation, a packed bed scrubber with circulating mother liquor to wash the waste air from cyclone, a set of melt urea supply system, and the necessary flow rate and temperature measurement and controlling devices. The transportation of melt urea is driven by compressed air. Particulate phosphates or potassium salts as "seeds" were introduced into the granulator and fluidized at dynamic stable height of about 500mm. Melting urea of $140\square$ was sprayed into the fluidized layer from the nozzle embedded on bottom or top of granulator. Temperature of the fluidizing air should not be too high in order to prevent eutectic agglomerations between particles. In the operation we initially adjust the fluidized air approximately to $90{\sim}115\square$ and finally to ambient when the urea coated particles obtained the desired diameters.

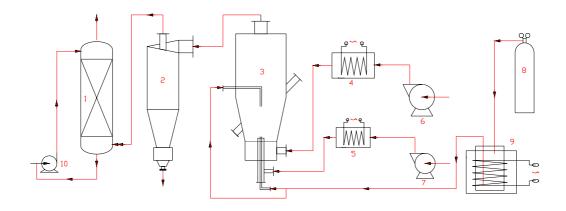


Figure 3. Sketch of the Fluidized-granulation Experiment.

- 1. scrubber
- 2. cyclone
- 3. fluidized bed granulator
- 4. 5. 9. electric heaters
- 6. 7. blower
- 8. compressed nitrogen tank
- 10. pump

It is well known that two mechanisms of granulation or coating exist in the fluidization-granulation process[7]. One is skin formation, by which the core particle is successively enveloped by large liquid drops sprayed from the nozzle into the fluidized nuclei, the drop is large enough to wet the whole particle and is subsequently solidified to form an onion-like layer. This structure of the granules leads to significant internal stresses that can result in poor mechanical properties, such as crushing strength and resistance to attrition. In addition problems may occur as a result that the material deposited is not dried completely before a new layer of liquid is deposited on the nucleus. The other is accretion formation, in which the nuclei are successively moistened by small droplets, and each time only a small portion of particle surface is covered with a thin layer of liquid material. In this manner the envelope is gradually built up, and thus formed granules have very fine structure and very great strength. For this purpose both hydraulic and

pneumatic spray nozzles were tested in the experiment to generate suitable melt urea droplets in the granulation. It is shown that pneumatic sprayer can provide droplets of much smaller sizes than that of hydraulic sprayer. Nevertheless, finer urea dusts should be purged to add the scrubbing load.

To clarify the detailed phenomenon of granule formation, urea melt was dyed before its introduction into the granulator.

4. RESULTS AND DISCUSSION

In the granulation experiment MAP, DAP, TSP were used as seed particles. Some sections of the samples of MAP as core produced at different temperatures of fluidized air were taken as follows. In Fig.4 the boundary between the MAP core and the coated urea layer is too blurry to be seen. It is shown that the adherence of melt urea to MAP is strong and the compatibility of urea and MAP is good. Particle has DAP core is sliced in Fig.5 in which the division of phosphate and urea compositions is quite obvious. Since TSP particle is neither soluble nor meltable, it almost keeps its initial shape when enveloped by urea melt (Fig.6).

Roughly speaking temperature of fluidized air plays a more important role in the granulation. If the temperature is too high eutectic phenomenon can arise. If too low, the compatibility between the core and coated layer may decrease and more dust of urea is generated. To be particularly mentioned, the hygroscopicity of urea mixed with SSP and TSP can be dropped sharply if the final product manufactured by this technique. The reason is that the released crystalline water resulted when urea reacts with SSP or TSP can be evaporated by the fluidizing air of high temperature.

Qualitatively saying, the proportion of N increases if the layer of urea of the compound granule becomes thick and decreases when the core materials have larger diameters. So the N:P2O5(:K2O) ratios can be adjustable on condition that the spray amount of urea melt or the transport speed of seed materials is controlled.



Figure.4. Section of Ureabased MAP Granule(4.0mm).



Figure 5. Section of Ureabased DAP Granule(3.6mm).

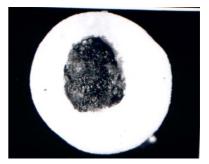


Figure 6. Section of Ureabased TSP Granule(4.0mm).

It is well known that serious problem of high hygroscopicity has not been solved for many years. But today we can show our best respects to phosphorous fertilizer industry. Thorough analysis should be carried out to give quantitative conclusions of the ongoing experiment. Some preliminary experimental results are listed in Table 1.

The experiment is still going on in order to get more information to complete our pilot plant design.

Table 1. Main Indices of Prepared Samples.

Test no.		1	2	3
Raw Materials				
Urea melt				
Concentration	wt %	96.5	96.5	96.5
Temperature		140.0	140.0	140.0
Feed rate,	kg/t(product)	15.0	20	10
Phosphates core		MAP	DAP	TSP
P2O5	wt%	42.0	48.0	16.0
N	wt%	13.0	18.0	44.0
H2O	wt%	1.5	3.0	5.5
Diameter	mm	1.0□3.0	1.0~4.0	1.0~4.0
Feed rate	kg/batch	10.0	10.0	10.0
Fluidizing air				
Flow rate	Nm3/h	900	850	1100
Temperature,		100	100	120
Spouting air				
Flow rate,	Nm3/h	130	130	120
Temperature		135□140	135□140	135□140
Bed temperature		100□115	100□115	□120
Product quality index				
N:P2O5		25-25-0	36-16-0	20-20-0
Granulometry				
>5.0 mm	wt%	13.7	10.6	20.3
4.0-5.0 mm	wt%	81.2	82.3	77.1
2-4.0 mm	wt%	5.1	7.1	2.6
Crushing strength				
4.0 mm	kg	0.32	0.34	0.28
2.5 mm	kg	0.41	0.50	0.37
Storage	day	□90	□90	>90

5. CONCLUSIONS

A novel process of making non-uniform granular urea-based compound fertilizer by fluidized-granulation technology is successfully implemented based on a careful analysis of the physical and chemical properties of urea, phosphates and potassium salts. From our preliminary experimental results we can roughly conclude as follows:

- 1. A breakthrough in making granular non-uniform compound fertilizer has been made in our ongoing fluidized-granulation experiment at the present stage. Fertilizers containing MAP, DAP, SSP, TSP and potassium salts as the materials were coated with urea melt is compatible and durable in storage. The ratio of the nutrients can be adjusted according the user's request.
- 2. Serious hygroscopicity can be prevented by coating urea on the surfaces of particular SSP and TSP.

6. REFERENCES

- [1] Кочетков, В. Н., Фосфородержащие Удобрения Спроавочик, Изд. □Химия □, 1982.
- [2] Lowrison, G. C., Fertilizer Technology, New York, 1992.
- [3] G. Brusaco, R. Monaldi, V. De Lucia and A. Barbera, Production of NPK Fertilzers with a Spouted Bed Granulation-Dryer., Port El Kantaoui, Tunisia, 1986. IFA Technical Conference proceedings/86/13.
- [4] Шахва, Н. А, Гришаев, И. Г., Кузнецов, В. Н., Подкопаев, В. Д., Мельников, Е. В., Петрова, В. В., Новый способ получения смешанных удобрений в виде двухслойных гранул, Хим. пром., 1971, No.1, c.37-42.
- [5] Ullemann, Hans, Kontinuierliche Wirbelschicht-Spr□hgranulation, Chem.-Ing.-Tech., 62(1990), Nr.10, S.822-834.
- [6] CN Appl. 00116135.0.
- [7] US 4,343,622.