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9. ABSTRACT

Sulfur-coated urea (SCU) is the culmination of about 15 years of TVA work on development of controlled-release nitrogen fertilizers.

This report covers the development work from laboratory and bench-scale studies, through a small pilot plant of 300 to 600 lbs/hr (136 to 272 kg/hr), and then to a large pilot plant with capacity of 1 to 2 short tons (0.9 to 1.8 metric tons)/hr. The laboratory and pilot-plant equipment are described, operating conditions are outlined, and results of the test work are evaluated.

Agronomic tests are being made. Changes in equipment and operating conditions that resulted in the improved coatings are described. The economics of production of SCU were estimated, and results are given. Plans for a demonstration scale plant are outlined. Under the best practical conditions established in pilot plant tests, the nitrogen in SCU is estimated to cost only about 27% to 34% more per unit than regular urea. This is with urea produced at the facility at an f.o.b. price of $120/ton and sulfur delivered at $34/ton. Results from agronomic tests of SCU in 39 U.S. states, 2 U.S. territories, and 26 other countries are summarized. Significant advantages are shown for SCU on several crops and in a variety of cropping situations. Crops for which SCU showed particularly good results include rice under delayed or intermittent flooding, sugarcane, pineapples, fruits and vegetables, forages, and turf. The product also would be very good for horticultural and other specialty fertilizer use. In addition to the several significant advantages of controlled-release of nitrogen, the product has improved storage and handling properties and the sulfur is available as a secondary nutrient.

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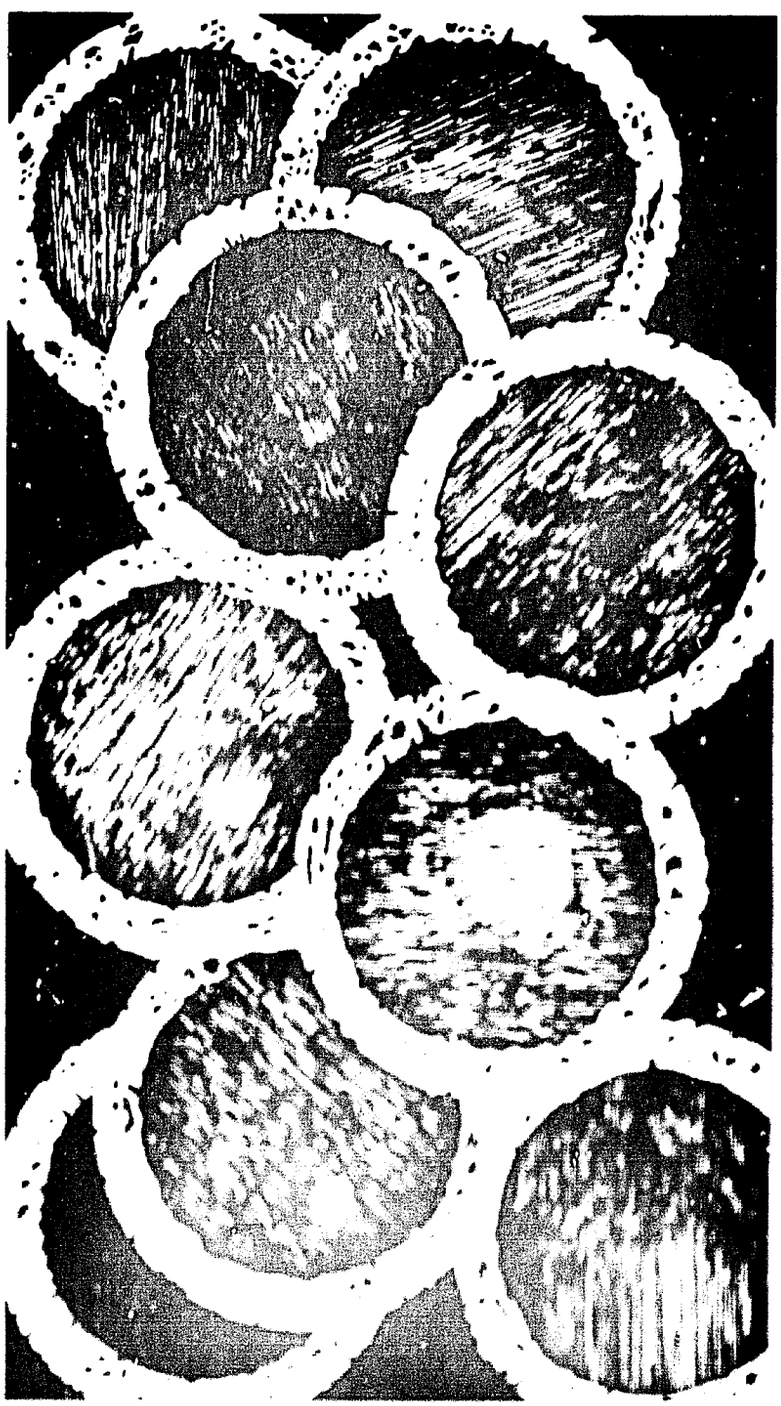

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SUMMARY

Sulfur-coated urea (SCU) is the culmination of about 15 years of TVA work on development of controlled-release nitrogen fertilizers. In the early years of work a large number of individual compounds were screened, but none were found that had both suitable properties and potential for low enough cost to be used as fertilizers. The early laboratory work with SCU indicated good promise for controlling the release rate of nitrogen by applying varying thicknesses of sulfur coatings. The product appeared to fulfill the need for a controlled-release fertilizer that would release its nitrogen at the right time and in proper amounts required by growing plants. Early economic evaluations indicated that cost should be low enough for use as a fertilizer.

This report covers the development work from laboratory and bench-scale studies, through a small pilot plant of 300 to 600 lbs/hr (136 to 272 kg/hr), and then to a large pilot plant with capacity of 1 to 2 short tons (0.9 to 1.8 metric tons)/hr. The laboratory and pilot-plant equipment are described, operating conditions are outlined, and results of the test work are evaluated.

Relatively high coating weights of sulfur were required in the early work. As work progressed, the coating weights were decreased markedly. Wax and microbicide were added on top of the sulfur coatings in most of the work. Purpose of the wax was to seal imperfections in the sulfur coating; a microbicide appeared to be needed to counteract attack of soil microbes on the wax. In the latest work, the wax and microbicide were eliminated and good controlled-release properties (based on dissolution rates in water) were indicated with a somewhat heavier coating of sulfur alone. This would simplify the plant and its operation and would substantially decrease required investment. Agronomic tests are being made.

Changes in equipment and operating conditions that resulted in the improved coatings are described.

The economics of production of SCU were estimated, and results are given. Plans for a demonstration scale plant are outlined.

Under the best practical conditions established in pilot-plant tests, the nitrogen in SCU is estimated to cost only about 27% to 34% more per unit than regular urea. This is with urea produced at the facility at an f.o.b. price of $120/ton and sulfur delivered at $34/ton.

Results from agronomic tests of SCU in 39 U.S. states, 2 U.S. territories, and 26 other countries are summarized. Significant advantages are shown for SCU on several crops and in a variety of cropping situations. Crops for which SCU showed particularly good results include rice under delayed or intermittent flooding, sugarcane, pineapples, fruits and vegetables, forages, and turf. The product also would be very good for horticultural and other specialty fertilizer use. In addition to the several significant advantages of controlled-release of nitrogen, the product has improved storage and handling properties and the sulfur is available as a secondary nutrient. SCU is also compatible with other fertilizer materials, such as unammoniated superphosphate, that are not compatible with regular urea.

BACKGROUND INFORMATION

Fertilizers that release their plant nutrients slowly throughout the growing season or even longer continue to attract the attention of technologists and agronomists. There are a number of proved or potential advantages for such fertilizers. These include: increased efficiency of uptake by plants; decreased cost of application due to fewer applications; minimization of losses by leaching, fixation, or decomposition; elimination of luxury consumption; and avoidance of burning of vegetation or damage to seedlings.

The Tennessee Valley Authority (TVA) has been interested in slow- or controlled-release fertilizers for many years. A number of organic nitrogen compounds were studied initially in the laboratory and greenhouse. Some proved to be readily available, some slowly available, some inert as a fertilizer, and some actually toxic to plants. Among those showing promise as a slow-release fertilizer were oxamide, glycouril, cyanuric acid, ammeline, and ammelide. Because no practical or economical process has been devised, none of these materials have been manufactured commercially for use as a fertilizer.

After this early experience, emphasis of TVA research and development work shifted to the application of coatings to conventional fertilizer granules. The advantage of coating conventional fertilizers with slowly soluble material is that low-cost, readily available fertilizers can be used. Various coating materials were tested, including paraffin, oils, resins, and plastics. Because of excessive cost, testing was not extensive. No really satisfactory coating procedure using these materials was developed.

Starting in 1961 elemental sulfur was tested as the coating material. Sulfur as a coating material has the advantages of low cost and supplying of a secondary plant nutrient. In the earliest laboratory tests, several coating procedures were studied, of which two were found to be suitable. These were (1) dipping of individual fertilizer prills or granules in molten sulfur, and (2) dissolving the sulfur in a suitable solvent, such as carbon disulfide, which was then sprayed onto the fertilizer particles.

The sulfur coatings were promising in providing slow release of the nitrogen from urea. However, the dipping procedure applied sulfur approximately equal to the weight of the urea. The lighter coatings, 5% to 8% of sulfur, applied by dissolving sulfur in carbon disulfide and spraying it on the prills of urea were effective in inhibiting nitrification. In 85 days in soil, only 45% to 65% of the urea nitrogen was nitrified. The surface of some of the samples had been glazed by treatment with direct flame. Moisture was readily absorbed by all of the materials.

Laboratory work on the coating procedure was continued in an effort to develop a more practical method. Sulfur coatings were applied by atomizing a spray of molten sulfur with a jet of hot air. Figure 1 shows some early laboratory equipment used for coating. Good control was obtained and the coatings appeared to be uniform. The heavier coatings gave good resistance to dissolution of the nitrogen by contact with water as measured by an arbitrary laboratory procedure.

BENCH-SCALE TESTS OF SULFUR-COATING—BATCHWISE PROCEDURE

Equipment and Procedure

After several trials, a bench-scale procedure for spray coating with molten sulfur was developed. This method involved atomizing a stream of molten sulfur with a jet of hot air which spread the stream of sulfur onto a moving bed of the granules being coated. This was a batchwise procedure using 200 grams of fertilizer in each batch in a rotary drum. Good control was obtained and coatings appeared to be uniform.

Fluidized bed-type laboratory apparatus was tested in sulfur coating. The particles remained free flowing and did not agglomerate, but dissolution rates were quite high. This type of procedure was abandoned and work continued in the laboratory with a rolling bed of granules in a rotary drum.

Laboratory Evaluation of Products

Laboratory evaluation procedures were developed to compare the characteristics of products made by different procedures. Changes in these procedures evolved as the small-scale work progressed and later moved to pilot-plant scale. For coated urea, the coating was taken as that portion insoluble in water. This was determined by crushing a 10-gram sample and letting it stand in 175 ml of water at room temperature for 24 hours. The latest procedure used a high-speed kitchen blender (Waring) for crushing and

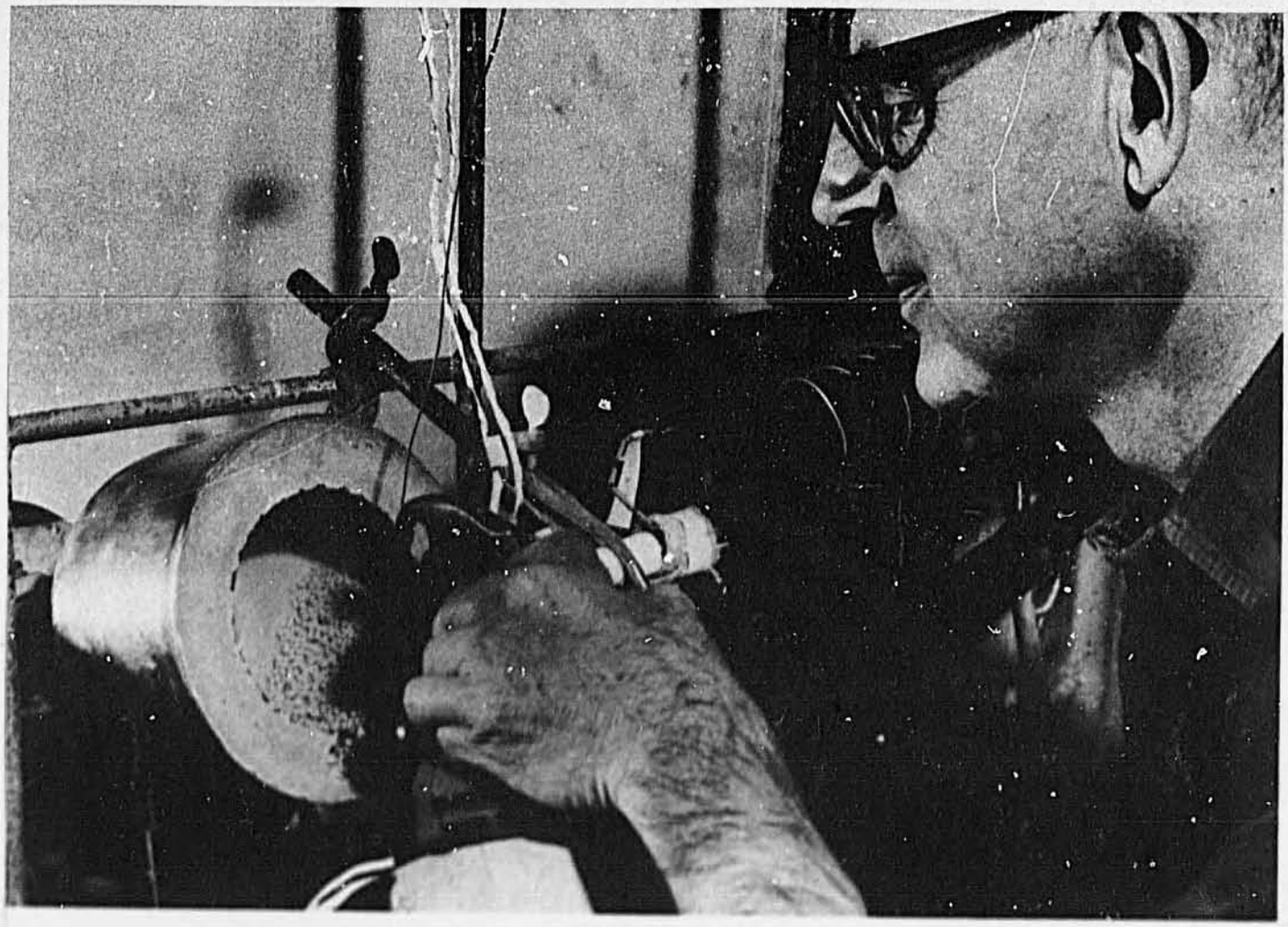


Figure 1. In early TVA work, urea was coated in a miniature rotating drum by spraying molten sulfur with a hand-held sprayer

dissolving the particles. The insolubles were filtered, washed, dried, and weighed to determine the coating weight. The dissolution period was decreased to 2 hours by maintaining the sample in water at 150°F (65°C).

The relative effectiveness of the coating in controlling the rate of dissolution of the nitrogen was determined by immersion of a 50-gram sample in 250 ml of water for specified periods of time. The temperature of the water was 80°F (27°C) in the earliest tests. The temperature was later changed to 100°F (38°C). Time of exposure was 1, 5 or 7, 14, and 28 days. In some tests the products were tested for only 7 days. The amounts of substrate in solution were determined by the specific gravity (pycnometer) of the solution; calibration of specific gravity with chemical analysis was necessary. Later, an Abbe refractometer was used to determine the concentration of substrate in the solution. This speeded up the operation considerably and proved to be quite reliable. Data from two typical samples of coated urea are plotted in figure 2.

The effect of temperature of the water used in the dissolution tests on the rate of dissolution is shown in table 1. The data also are plotted in figure 3.

As was expected, the percentage of substrate dissolved in a given time period increased as the temperature of the water increased from 32° to 100°F (0° to 38°C). After these tests the routine procedure used water at 100°F (38°C).

Use of Plasticizer for Sulfur

In the early tests of spraying molten sulfur, the addition of a plasticizer improved the efficiency of the sulfur coating substantially. The plasticizer apparently decreased the minute cracks in the sulfur coating and made the sulfur adhere more tenaciously. In these tests, increase in the sulfur coating from 3% to 15% decreased the dissolution of urea from 57% to 24%. With plasticizer added, an increase in coating weight from 4% to 16% lowered dissolution from 43% to 6%. About 5% appeared to be the optimum content of plasticizer in the sulfur.

The incorporation of plasticizer in sulfur used for coating offered potential improvement in coating effectiveness. However, the product tended to cake excessively when plasticizer content was more than about 5% of the

Table 1. Effect of water temperature on substrate dissolution rate

Sample	Total coating wt %	Substrate dissolution, wt %, in water at 32.4°F (0°C) 7 days	14 days	Daily	81°F (27°C) 7 days	14 days	Daily	100°F (38°C) 7 days	14 days	Daily
1	23	8.4	12.2	0.5	12.8	17.3	0.6	17.6	21.3	0.5
2	23	8.0	13.4	0.8	14.1	17.8	0.5	18.1	21.1	0.4
3	23	6.0	11.9	0.8	12.2	16.1	0.6	17.2	20.2	0.4

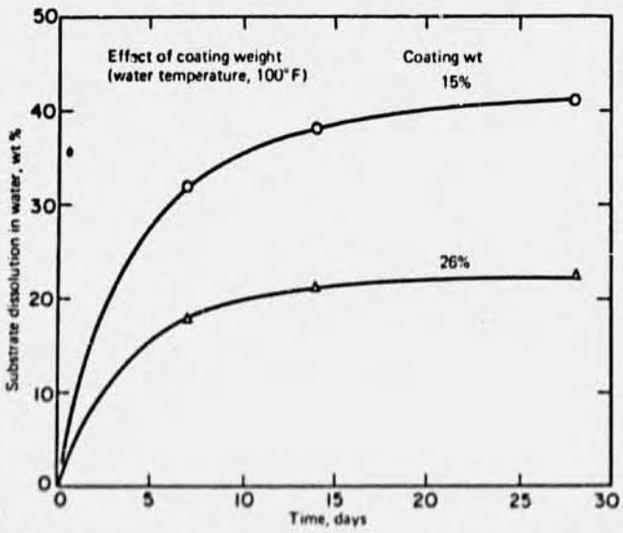


Figure 2. Effect of time on substrate dissolution in water at 100°F

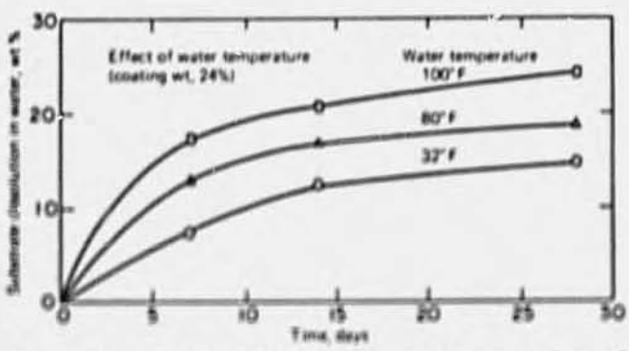


Figure 3. Substrate dissolution in water versus time of immersion of sulfur-coated spray-drum-granulated ureas (-6+10 mesh)

sulfur weight. Later, the effect of plasticizer was insignificant because of improved coating technique and use of better sealants. Study of plasticizers therefore was discontinued.

Coating with Sulfur Dissolved in Solvents

Since sulfur dissolved in carbon disulfide and sprayed onto the granules gave considerably better results than spraying of molten sulfur, tests of other solvents were made. Use of liquid ammonia appeared to have better economic prospects because of lower cost and prospects for effective recovery. Tests with ammonia as the solvent gave quite promising results—actually better than with carbon disulfide as the solvent. A 5.6% coating of sulfur applied in the ammonia solvent resulted in dissolution of only 16% of the urea in 30 minutes exposure to water. Approximately the same coating weight applied as a carbon disulfide solution gave dissolution of 27% in the same time of exposure to water.

Because of the complexity and probable difficulty in recovery of solvent, this procedure was not used in subsequent test work.

Use of Sealant on Sulfur Coating

All of the test work on spray coating with molten sulfur showed that the effectiveness of the coatings was limited because of the presence of cracks, pores, and crystalline interstices in the shells. Very heavy coatings of 30% to 40% decreased occurrence of these, but such heavy coatings would be costly and would drastically decrease plant food content of the fertilizer being coated. Tests were initiated, therefore, in the use of various materials to seal the microscopic fissures in the sulfur coating.

The earliest tests utilized boiled linseed oil sprayed onto the granules after the sulfur coating had been applied. This treatment was not very effective. Later tests were made using SAE 30 petroleum oil of the nondetergent type. The treatment with this type of oil was very effective in decreasing dissolution in water, as an undercoating, top coating, or combination of the two. It was possible to decrease the sulfur coating substantially by applying 4% to 7% of the oil. The effectiveness of the oil treatment on various sizes of granules or prills is shown in figure 4.

In other tests, very good resistance to dissolution was obtained by applying 3% oil undercoating followed by a 23% coating of molten sulfur and finally applying 5% additional sulfur as a solution spray. Although very good slow-release properties were obtained, the complexity of the multiple coating procedure did not appear to be practical for a commercial operation.

In continuing tests, use of SAE 30 motor oil as a topcoating on the sulfur gave as good dissolution results as

oil followed by split applications of sulfur. Data are shown in figure 5. About 3% of oil was optimum on the larger (30-10-0) granules, while about 5% was needed for the smaller urea prills.

Although the granules and prills coated with sulfur and then with topcoating of SAE 30 oil gave satisfactorily low dissolution in water, when they were imbedded in soil, capillary action removed the oil and caused rapid dissolution. Therefore, several other sealants were tested in the laboratory. These included SAE 60 oil containing 6% of tallow oil, PRP (petrolatum, resin, parrafin mix), SAE 30 oil followed by petrolatum, and petrolatum alone. In simulated soil exposure (powdered vermiculite) in a humidity chamber, only the PRP and refined petrolatum were effective.

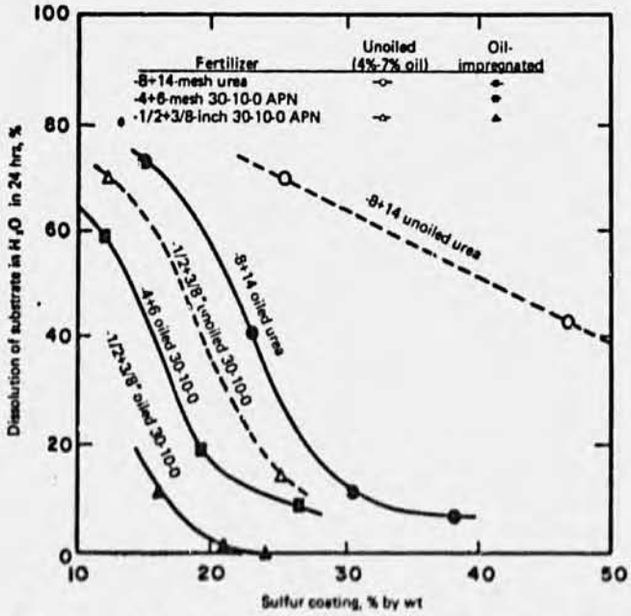


Figure 4. Effects of proportion of sulfur coating on dissolution rate of oiled and unoiled fertilizer granules

In exploratory tests using either a pan granulator or rotary drum in batchwise tests, coating effectiveness was not good using molten sulfur sprays and crude petrolatum sealant. Also, the 30-10-0 ammonium phosphate nitrate being coated was of smaller particle size than had been used in the laboratory tests. Laboratory tests were then made using the same size granules and crude petrolatum. The crude petrolatum sealant was found to be the primary cause of the poor quality of the coating as is shown in figure 6. The refined petrolatum used successfully in previous tests was much more expensive than the crude material. Therefore, less expensive sealants that would be effective were sought.

Tests were conducted to evaluate 11 much less expensive types of petroleum derivatives. An 18% coating of sulfur was applied to -8+14 mesh urea prills, and the various sealants were applied at 3% by weight of the coated urea. Only 3 of the 11 sealants proved to be effective in tests for 24 hours and 10 days in water at 100°F (38°C). Soft wax was the best of the sealants based on dissolution of the urea substrate (29%) during the 10-day exposure.

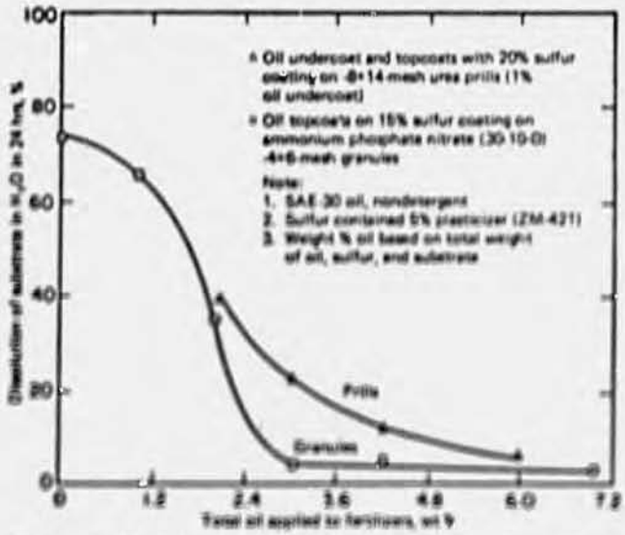


Figure 5. Effect of oil topcoat on dissolution rate of sulfur-coated urea and 30-10-0 ammonium phosphate nitrate

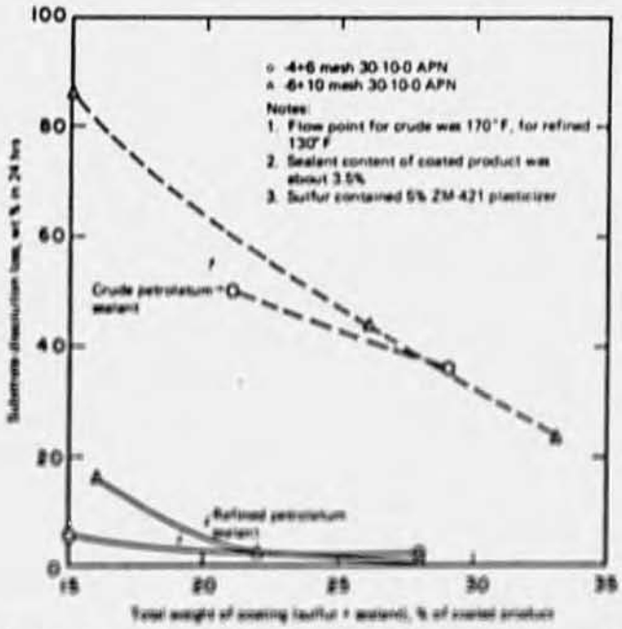


Figure 6. Effects of grade of petrolatum and size of granules on substrate dissolution losses from sulfur-coated 30-10-0 granules in water immersion tests

Sulfur coatings were consistently ineffective as controlled-release fertilizer under greenhouse conditions when the fertilizer was mixed with the soil. It was at first believed that soil capillary forces removed the oil from the sealant. A more effective sealant appeared to be necessary so the following types were tested:

Stearic acid	Shellmax 800 (microcrystalline wax)
Paraffin	Sperm 90 (sperm oil plus sperm wax)
Slack wax	Shellmax 800 with a surfactant additive
Asphalts	

All of the above materials were applied as sealants on SCU and the products were evaluated by standard dissolution tests. Only the microcrystalline Shellmax 800 was satisfactory. It was adopted for use in later test work.

The microcrystalline wax imparted considerable tackiness to the surface of the granules. This was countered by dusting a conditioner onto the surface of the granules after they had been cooled sufficiently (100°-105°F) to prevent embedding of the conditioner in the wax. About 1.5% to 2% of kaolin clay, diatomaceous earth, or vermiculite was satisfactory.

Tests of Dissolution in Soil

SCU granules continued to show poor controlled-release properties in the soil although low dissolution was obtained in water at 100°F (38°C). Tests were made in the laboratory in which samples of coated urea were exposed in the soil at 100°F (38°C) to simulate greenhouse conditions. The granules were completely buried in the Davidson soil (pH 5.8). Coating weights including sealant were 35% to 43% by weight. The soil layer was 3 inches deep and the granules were buried 2 inches deep. The soil surface was open to the atmosphere so that normal evaporation of moisture could take place. Water was added daily to replace that which had evaporated. Conventional water immersion tests at 100°F (38°C) were conducted over the same period for comparison. Data from these tests are shown in table 2.

The data in table 2 show that the simulated greenhouse soil exposure tests were much more severe than the water-dissolution tests.

Use of microbicides--In the course of coating evaluation, other products were subjected to soil burial tests. In general, dissolution rates in the soil were much higher than in water, and in some cases samples with low dissolution rates in water failed completely in the soil in a few days. This suggested that soil microorganisms were destroying the coating when soil temperature and moisture conditions were favorable. Soil tests with steam-sterilized soil under otherwise identical conditions did not show the same deterioration, thus confirming the theory of microbial attack.

Soil chemists pointed out that wax sealant is more subject to microbial attack than the sulfur coating. To combat this, a number of known microbicides were added to the wax sealant. The microbicide content of the product was in the range of 0.5% to 2% by weight. The addition of microbicides was easily accomplished. Additives soluble in wax were dissolved in the wax prior to sealing, whereas insoluble ones were dusted on the tumbling granules. The standard soil test procedure was followed. One-gram samples of the coated urea were buried in 250 grams of soil in which the water:soil ratio of 1:3 was maintained by replacing the evaporated water each day. The average daily evaporation of water was 10 grams. Among the several materials tested, pentachlorophenol and coal tar gave the most consistent good results. Data showing the marked effectiveness of these two microbicides at the 0.5% level are plotted in figure 7.

Tests of sealants in water and soil exposure--Laboratory work as evaluated by water dissolution tests

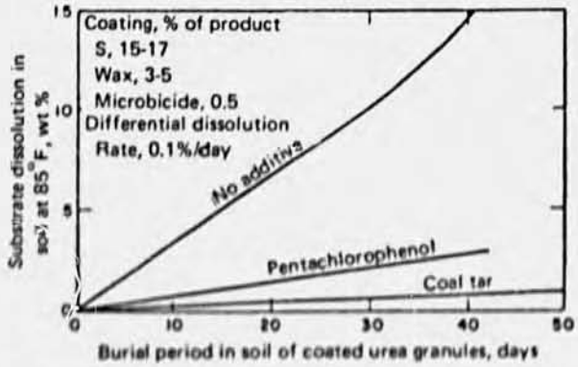


Figure 7. Effect of microbicides on substrate dissolution in soil at 85°F (30°C)

Table 2. Substrate dissolution of sulfur-coated urea granules[a] in water and wetted soil at 100°F (38°C)

	Coating, wt %			Substrate dissolution, %							
		Soft wax		5 days	6 days	2 weeks		4 weeks		8 weeks	
Test No.	S	sealant	Total	H_2O	soil	H_2O	Soil	H_2O	Soil	H_2O	Soil
1	29.9	5.0	34.9	14.3	35.9	35.9	52.8	36.7	–	44.0	96.3
2	32.8	5.0	37.8	7.1	16.4	12.4	29.6	21.1	–	30.1	68.6
3	38.7	4.0	42.7	1.0	5.1	1.7	10.6	2.0	–	5.8	52.9

[a]Urea mesh size, 4+10. Water content of soil ranged from 70% to 100% saturation.

showed that Shellmax 800 was fully equal to soft wax as a sealant for SCU. To obtain additional data, the two sealants were tested under the same conditions in water and soil tests at 100°F (38°C). The substrate was -6+8 mesh urea granules and the total coating weight in each case was about 24% (18% sulfur, 5% sealant, and 1% vermiculite conditioner). The coatings and sealants were applied batchwise on heated granules in a 6-inch (15-cm) rotary drum. Samples from each test were buried in soil (pH 5.8) or immersed in water, and dissolution losses were determined after 7, 21, and 42 days. Free evaporation of moisture from the soil was allowed, and the soil was resaturated daily. The data are plotted in figure 8.

The two sealants were about equally effective in the water-immersion tests over the period of 42 days; in this time, dissolution in water was only 0.7%/day. In the soil tests, however, there was a large difference in effectiveness of the two sealants. At the end of 3 weeks, the soft-wax-coated product was essentially all dissolved, whereas the Shellmax-800-coated product had lost only 38% by weight. These data indicated that a more severe test than water immersion at 100°F (38°C) might be needed to fully evaluate coatings.

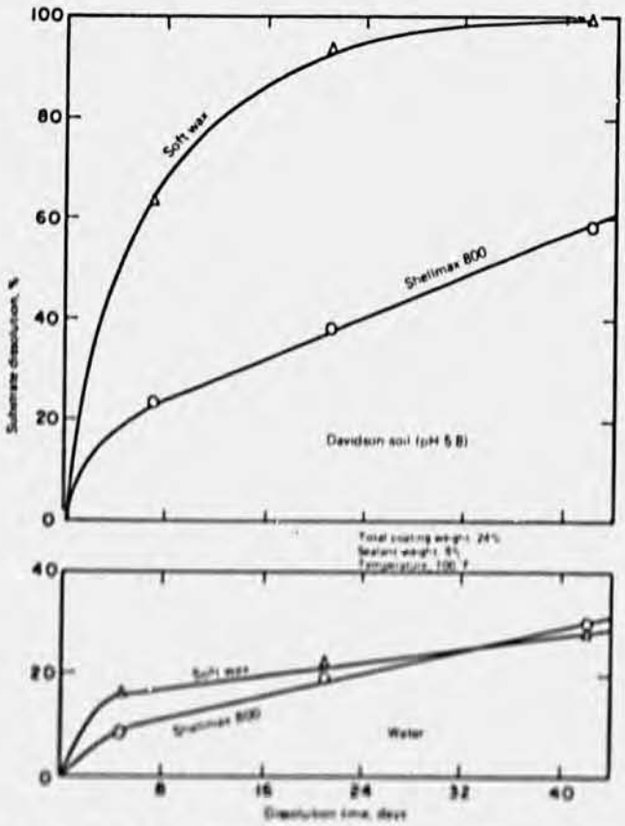


Figure 8. Substrate dissolution of sulfur-coated urea granules sealed with soft wax or Shellmax 800

Effect of Particle Size and Shape of Substrate

In laboratory tests of spray coating with molten sulfur, the pronounced advantage of use of substrate of larger particle size was quite evident. The much lower surface area of larger granules or prills allows better coating with a given amount of sulfur.

Effect of particle size of the urea on coating efficiency was studied further. The three particle size ranges were -6+16, -6+10, and -6+8 mesh. These fractions were obtained by screening granular urea that had been produced in a pilot plant using a pan granulator. The total coating weight applied was about 21% in all tests. Dissolution rates of substrate in water at 100°F are given in table 3. The data indicated that decreasing the particle size range from -6+8 to -6+16 mesh increased the dissolution rate in water only slightly. It appeared that coating of granules screened in the -6+16 mesh range would be practical.

Additional batchwise tests were made to study the effect of particle size of urea on coating effectiveness. Batches of 12-15 lbs (5.5-7 kg) of commercial prills "as received" (2% -6+8 mesh, 90% -8+16 mesh, 8% -16 mesh) or screened to -6+10, and -10 mesh (9% -16 mesh) fractions were coated in

Table 3. Substrate dissolution rates of sulfur-coated urea granules[a] in water at 100°F (38°C)

Test[b] No.	Granular urea size, mesh	Total coating[c] wt %	Substrate dissolution in water at 100°F (38°C) wt %/day — Length of test in days: 1	5
1	-6+16	19.8	4.2	1.0
2	-6+16	23.1	1.3	0.2
Average		*21.5*	*2.7*	*0.7*
3	-6+10	18.7	3.8	1.0
4		20.4	2.9	1.2
5		21.9	2.8	1.4
Average		*20.3*	*3.2*	*1.2*
6	-6+8	19.8	2.7	0.6
7		20.6	1.1	0.5
8		22.2	1.9	0.1
Average		*20.9*	*1.9*	*0.4*

[a]Pan-granulated urea from pilot plant.
[b]Operating conditions: 15 to 20 psig air, 315° to 320°F; sulfur 295° to 300°F, bed 150° to 200°F; sulfur spray 15 to 20 gpm; and 8-inch diameter drum.
[c]Total coating including sulfur, Shellmax 800 (5%) and vermiculite (1%).

the 22-inch (56-cm) pan granulator. A coating of about 16% sulfur, 3% wax (Shellmax 800), 0.5% microbicide (coal tar oil) and 1% conditioner was applied in all tests. The prills were heated to 150°F (66°C) and the molten sulfur was applied at 290°F (143°C). Wax was applied at 190°F (88°C) and the product was cooled to 110°F (43°C) prior to conditioning.

The 24-hour dissolution rates for the -6+10 mesh fraction, the material as received, and the -10 mesh fractions averaged about 3.5%, 16%, and 42%, respectively. These data showed that with this coating weight (16% sulfur, 20.5% total), the proportion of defective coatings increased with decrease in particle size of the urea. However, the differential rate (daily dissolution rate after the first 24 hours) was low (0.3% to 1.1%) in all cases. These data indicated that commercial prilled urea could be coated satisfactorily, particularly if the finer material is removed by screening.

Other tests were made in the laboratory to determine the effect of particle size of urea on dissolution rate. The two materials tested were fertilizer grade spray-drum-granulated urea (-6+10 mesh) and forestry size (-3+6 mesh). The tests also included the effect of content of wax on dissolution rate. Conditions of operation and results of the dissolution tests are shown in figure 9.

The curves show the marked effect of particle size on dissolution rate. Under the conditions tested, the forestry size SCU had extremely low rates. The fertilizer size SCU

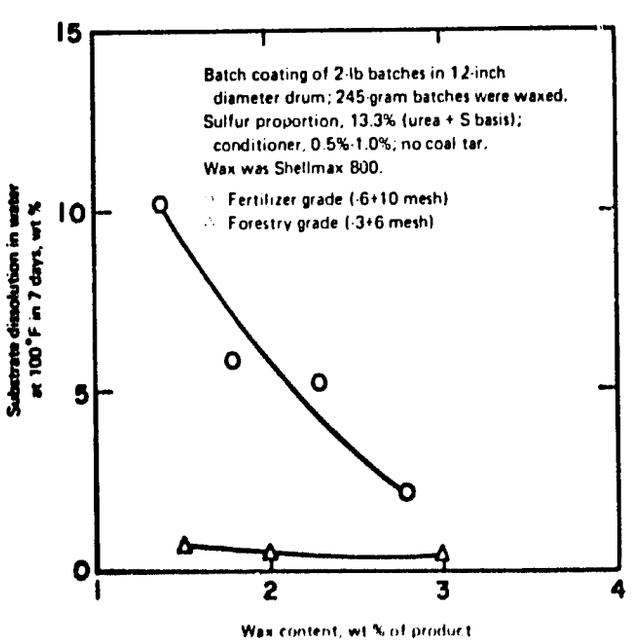


Figure 9. Effect of sealant proportion on dissolution rate of sulfur-coated commercial granular urea of two sizes (laboratory batch coating tests)

had considerably higher dissolution rates but they were still well within the desired range. The percentage of wax used had very little effect on dissolution of the large forestry size granules. Higher proportions of wax (2% or 3%) did lower dissolution of the regular commercial fertilizer size urea.

Variables that affect coating effectiveness include shape as well as size of the granules and smoothness of the surface. Tests were made in the laboratory using granular urea that was irregular in shape and with another batch in which the particles were smooth. The quantity of sulfur and wax added was about 35% in one test and 31% in the other. The batch tests were conducted in a 22-inch (55-cm) pan granulator. Data are given in the following tabulation.

Test No.	Description	Granules/gram	Sulfur plus sealant[a] wt %	Substrate dissolution in water at 100°F (38°C) in 24 hrs wt %
1	Irregular	130	34.9	27.1
2	Smooth	80	31.3	3.7

[a]5% wax.

The data show that irregularly shaped particles are very difficult to coat effectively. Even with a higher percentage of coating, the dissolution rate for the irregular granules was about seven times that for the smooth granules.

Study of Process Variables

Important variables in the coating procedure were studied to define the optimum range of conditions. These were amount of sealant used, sulfur temperature, air temperature, and temperature of bed of granules being coated. The sulfur temperature was found to be the most important of these variables. A range of 290° to 340°F (143° to 170°C) proved to be best. The best range of temperature for the bed of granules being coated was 130° to 155°F (54° to 70°C). The highest range of temperature of the atomizing air tested was best; this was 300° to 370°F (150° to 190°C). Two percent of sealant was selected since larger amounts did not give sufficiently lower dissolution to justify the added cost.

Since temperature of the sulfur being sprayed was found to be the most important variable studied, tests were made in which this temperature was varied over the range of 265° to 345°F (130° to 174°C) and closely controlled. Other variables were held in the range shown to be best in previous tests. Results of these tests are shown in figure 10.

The curve shows that the optimum temperature for the sulfur was in the range of 315° to 330°F (157° to 166°C). The less effective coatings at lower and higher temperatures apparently resulted from the nonuniformity of coating thickness. At temperatures higher than this range, the sulfur

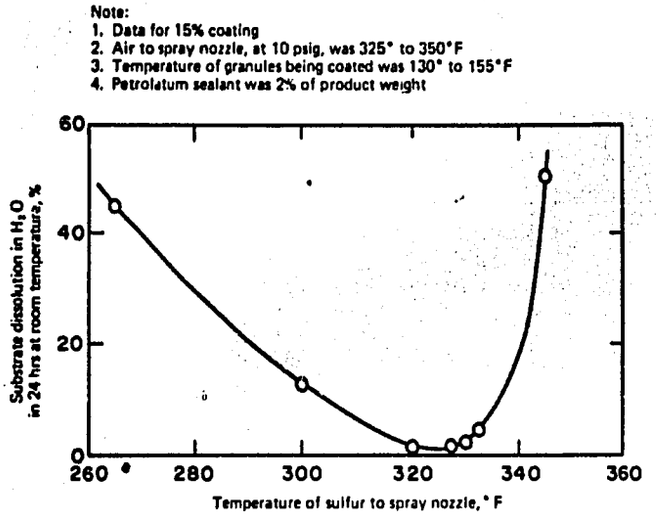


Figure 10. Moisture resistance of sulfur coatings as affected by various temperatures of sulfur to the spray nozzle and evaluated by dissolution rates of the substrate

likely was too viscous for good spraying; at temperatures lower than this range, the sulfur probably did not have enough plasticity to spread uniformly. The 315° to 330°F (157° to 166°C) range of temperature for the sulfur was used in subsequent test work.

Several tests were made to study further the effect of heating the bed of granules prior to applying the sulfur coating. Normally, the bed temperature had been about 80°F (27°C) at the start and 100° to 140°F (38° to 60°C) at the end. In the tests of preheating, the bed was maintained at 150° to 195°F (68° to 90°C). The molten sulfur and atomizing air temperatures were held in the range of 300° to 320°F (150° to 160°C). Data are given in the tabulation below.

Test No.	Temp. of tumbling bed being coated with sulfur, °F	Total coating[a] wt %	Substrate dissolution in water at 100°F (38°C), wt % 24 hrs	5 days	Daily rate between 1 & 5 days
1	180-190 (82-88°C)	20.3	4.0	9.4	1.3
2	160-180 (70-82°C)	22.3	2.9	3.8	0.3
3	150-170 (65-76°C)	23.2	4.2	9.3	1.3
4	180-195 (82-91°C)	25.5	5.6	6.7	0.3
Avg.	*175 (80°C)*	*22.8*	*4.2*	*7.3*	*0.8*

[a]Sulfur, Shellmax 800 sealant (5%) and vermiculite conditioner (1%).

The average product had a 23% total coating and a differential dissolution rate in water of only 0.8%/day. Preheating of the urea prior to coating was found to be quite beneficial. This procedure was used in subsequent work.

Miscellaneous Tests

Tests were made to determine the effect that temperature changes in handling and storage would have on dissolution characteristics of sulfur-coated fertilizers. Ammonium phosphate nitrate of 30-10-0 grade was used as the substrate. In these tests, portions of the coated samples were cooled to 32°F (0°C) and others were heated to 120°F (50°C). These materials were allowed to return to room temperature and then were subjected to the standard dissolution test. Other portions were subjected to the dissolution test without prior heating or cooling. Results of the tests showed, in most cases, that the coatings were effective on samples that were neither cooled nor heated and on samples that were cooled. The coatings were no longer effective on samples that were heated. The phase changes of ammonium nitrate probably caused the ineffective coatings after heating. When urea was used as the substrate, the effect was much less pronounced. The best procedure found to prevent serious deterioration of the coating at higher temperatures was to use sealant as a top coat at the 3% level.

In early greenhouse tests, sulfur coatings on fertilizer particles eliminated toxicity to seeds and root systems when the fertilizer and seed were placed together in the row. Later, tests were conducted to determine whether lighter coatings than are necessary for controlled release would be effective in preventing damage. Granular urea (-6+14 mesh) was used with sulfur coatings of 8%-16% by weight. One group of tests was with sulfur coatings unsealed and the other with 3% of soft wax as sealant. The coated fertilizers were applied in contact with 10 corn seeds in Mountview soil. After 7 days, the toxic effect of the fertilizers was evaluated by observation of seedling emergence and root systems. None of the unsealed coatings were effective in preventing damage. The 8% sealed coating was not effective, but the 12% and 16% sealed coatings completely eliminated any toxicity as evidenced by 100% seedling emergence and well developed root systems.

Several improvements were made in the batchwise laboratory coating procedure. These included use of Shellmax 800 as sealant, use of well-rounded -6+8 mesh urea granules instead of -6+10 mesh, and use of a higher preheat temperature of 150°-200°F (60°-94°C). With these improvements, coatings of about 18% gave dissolution rates about as low as with 30% to 35% coatings in the early tests. Dissolution rates were determined in water at 100°F for 1 and 5 days. Data for the tests with improved process conditions over a range of coating weights are shown in table 4.

The products with 21% and 24% coating showed release rates that were too low. Total coating weight of 17.5% was indicated to be adequate for satisfactorily controlled release.

Table 4. Substrate dissolution losses from sulfur-coated urea granules[a] in water at 100°F (38°C)

Test No.	Total coating,[b] wt %	Substrate dissolution, % 1 day	5 days	Daily rate between 1 & 5 days
1	10.7	25.5	87.5	15.5
2	13.7	8.6	16.5	2.0
3	15.8	15.6	32.0	4.1
Average	*13.4*	*16.6*	*45.3*	*7.2*
4	17.0	9.2	13.0	0.9
5	17.0	2.4	6.5	1.0
6	17.2	1.0	3.5	0.6
7	17.3	10.2	20.0	2.5
8	18.1	1.3	5.5	1.0
9	18.9	4.6	4.5	0.0
Average	*17.5*	*4.7*	*9.0*	*1.0*
10	20.3	1.0	0.5	0.0
11	21.0	0.8	2.5	0.4
12	21.4	2.0	4.0	0.5
13	21.6	3.2	6.5	0.8
14	22.4	1.1	1.0	0.0
15	22.6	0.9	1.0	0.0
Average	*21.5*	*1.5*	*2.5*	*0.3*
16	23.7	0.3	0.5	0.0
17	23.8	0.3	0.0	0.0
18	24.4	0.6	0.5	0.0
19	24.5	0.2	1.5	0.3
Average	*24.1*	*0.3*	*0.5*	*0.1*

[a]Coating applied to urea granules (-6+8 mesh) at 130°-190°F.
[b]Total coating of sulfur plus Shellmax 800 sealant (5%) and vermiculite conditioner (1%).

Coating of Larger Batches

In most of the early laboratory work, 100- to 200-gram batches of urea granules were coated with sulfur in a 6-inch (15-cm) diameter rotary drum. These amounts of products were sufficient for standard water-dissolution and greenhouse tests. However, larger amounts were needed for additional agronomic testing including field tests. Early attempts to move to a larger scale of operation were not very successful in that the products had erratic dissolution characteristics, and semicontinuous test products required heavy coatings of 30% to 40%. In later tests, a successful coating procedure for larger batches was developed using a 22-inch (56-cm) pan granulator.

The pan was 6 inches (15 cm) deep and was operated at 22 rpm with a slope of 75 degrees. It was equipped with a 3-inch (8-cm) retaining ring and 12 flights. A single pneumatic nozzle (Spraycо M 125) was used for atomization of the molten sulfur. About 2.2 lbs (0.9 kg) of sulfur was applied to 10 lbs (4.5 kg) of urea in a 10-minute period. The temperature of molten sulfur was held at about 280°F (138°C) while the wax temperature was 200°F (93°C). The substrate urea temperature was varied from 130°-175°F (72°-80°C). Atomizing air was held at 10°F above the temperature of the molten sulfur. Good results were obtained when the substrate (urea) temperature was 150°-170°F (66°-77°C). Under the favorable conditions, total coating weights of 19% and 23% (5% wax) gave dissolution rates in water at 100°F (38°C) of only 3% in 24 hours and 5% in 5 days. The coated products were equal in effectiveness to those prepared in the smaller equipment. The larger amounts of products allowed more thorough agronomic testing. With additional experience, lighter sulfur coatings were applied with good results; a total coating of 16.8% gave only 3.6% dissolution in 1 day and 6.0% in 3 days.

DEVELOPMENT OF A CONTINUOUS COATING PROCEDURE

After considerable experience with batchwise procedure had established the effect of a large number of variables, studies were initiated using a simulated continuous coating procedure. First tests were in a 22-inch (56-cm) pan granulator, equipped with flights, that had been used satisfactorily in batch tests. Results were not promising because of obvious poor coating uniformity. There appeared to be considerable bypassing of material fed to the pan so that not all of the granules passed beneath the sprays of sulfur.

Because of poor results with the pan, its use was discontinued and a rotary drum was used in subsequent test work of a continuous process. The drum was 24 inches (60 cm) in diameter and 19 inches (48 cm) long. A single spray gun inside the drum was set to spray directly onto the rolling bed of granules. To obtain a good indication of the

number of sulfur spray nozzles that would be required in a continuous operation on a one-pass basis, the granules or prills were recirculated until they appeared to be well coated. Samples were taken after four to eight passes beneath the spray. Portions of the last four cycles were sealed with 5% of wax. Determinations were made of the dissolution rates; these data are plotted in figure 11.

The data indicate that for each cycle the sulfur deposition was about 4% by weight of the product. After eight cycles, the product had a 33% sulfur coating. After addition of 5% of sealant, the substrate dissolution in water at 100°F was only 0.8%/day after 5 days. With six or seven cycles, the 30% and 36% coatings had dissolution rates of about 2.5%/day after 5 days.

These tests indicated that coating by a continuous process would be practical.

Because of the apparent need to apply coatings of 30% to 35% in the simulated tests of a continuous procedure, an effort was made to establish technique for preparing suitable products with coating weight of 20% or less. Urea granules (-6+10 mesh) were fed (30 lbs/hr, 14 kg/hr) to the 24-inch (60-cm) drum and sulfur was sprayed onto the granules in two stages. Each stage consisted of a 7-inch (18-cm) long section of the drum with a spray nozzle. The stages were separated by a ring dam. The granules in the drum were heated with hot air prior to coating with sulfur. After 30 minutes of operation, the products were sampled and sealant (Shellmax 800) was applied. Data from the tests are shown in the following tabulation.

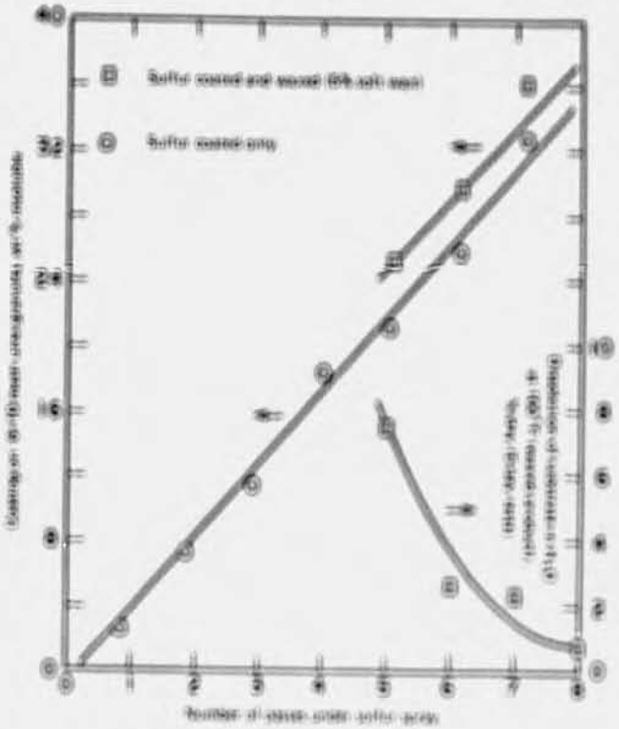


Figure 11. Dissolution rates of products made by multiple passes of a single batch under the sulfur spray by continuous recycle

Test No.	Average operating temp., °F: Feed to spray nozzle: Sulfur	Feed to spray nozzle: Air	Granule bed: 1st stage	Granule bed: 2d stage	Total coating wt % (including 5% sealant)*	Substrate dissolution in water at (100°F (38°C)), wt %: 24 hrs	Substrate dissolution in water at (100°F (38°C)), wt %: 5 days
1	275	290	140	120	17.3	13.7	20.6
2	285	303	150	130	27.9	2.0	8.9

*Shellmax 800

Although the temperature of the bed of granules was lower than desired, the lighter (17.3%) coating gave a substrate dissolution of only about 2%/day after the first day. These results, that showed considerable improvement over past exploratory tests of a continuous procedure, were quite promising as to prospects for lighter coatings being effective.

To obtain data at higher rates and with simplified continuous procedure, tests were made using the 24-inch (60-cm) drum with a feed rate of 100 lbs/hr (45 kg/hr) of urea. The sulfur was sprayed with a single nozzle. The feed materials were granular urea (-6+10 mesh) and unscreened prills (-6+30 mesh). The urea was preheated in the first 8 inches (18 cm) of the drum with hot air to temperatures of 130°-140°F (54°-60°C). The heater capacity limited preheating to temperatures lower than the desired 150°F (65°C). The sulfur and atomizing air were held at 285°F (140°C). The preheat section of the drum had a 8-inch (18-cm) retaining ring; there was no retaining ring at the discharge end of the drum. The single spray nozzle was positioned to spray vertically onto the bed at a point midway between the center and upper edge of the bed. This was the most active area of the bed. After good continuous operation was achieved, 300-gram samples were taken for waxing (3% or 5% of Shellmax 800) and conditioning. Conditions and results of the tests are shown in table 5.

The larger granules gave particularly promising results. A satisfactory product was made with a 20% coating (16% S, 3% wax, and 1% conditioner). The unscreened prills also were coated satisfactorily. With about 27% total coating (23% S, 3% wax, and 1% conditioner), the dissolution rates were 18% the first day but only 0.8%/day after that.

Dissolution as high as 18% to 20% the first day is not objectionable as long as the daily rate afterwards is no more than about 1%.

These test results showed that a continuous coating procedure was fully feasible at a reasonable rate of production. The promising results prompted plans for a complete pilot plant for continuous operating procedure.

Table 5. Water dissolutions of sulfur-coated urea obtained by continuous operation[a]

Test No.	Urea[b] substrates	Coating, wt % Sulfur	Hard wax	Conditioner	Total	Substrate dissolution in water at 100°F (38°C), wt % 24 hrs	5 days	Per day after 1st day
1	Granules	12.0	3.0	1.0	16.0	12.0	20.3	2.1
2		16.0	3.0	1.0	20.0	7.5	10.8	0.8
3		19.1	3.0	1.0	23.1	4.7	9.2	1.1
4		22.0	3.0	1.0	26.0	5.6	8.8	0.8
5		23.1	3.0	1.0	27.1	2.0	4.5	0.6
6		23.7	3.0	1.0	27.7	2.8	4.5	0.4
7	Prills	20.4	5.0	1.0	26.4	17.7	23.1	1.3
8		21.2	5.0	1.0	27.2	19.1	22.2	0.8
9		26.5	5.0	1.0	32.5	12.6	14.8	0.6
10		27.4	5.0	1.0	33.4	12.0	14.1	0.5

[a]Urea feed rate of 100 lbs/hr (45 kg/hr).
[b]

	Screen analysis, % 16	16:8	8:10	10:12	12:14	14:16	16
Granules	0	69	30	1	–	–	–
Prills	0	2	13	31	29	17	8

THE SMALL PILOT PLANT

Description of Pilot Plant and Operating Procedure

To develop the coating process further, a pilot plant was designed and constructed to coat fertilizer granules or prills continuously with sulfur, wax, and conditioner. The design production rate for the initial pilot plant was 150-300 lbs/hr (70-140 kg/hr). The unit was put into operation in September 1968. A schematic flow diagram with typical operating temperatures is shown in figure 12 and a photograph of the pilot plant is shown in figure 13.

The initial pilot plant consisted of three rotating drums operated in series. The third drum later was eliminated by using the second drum for dual functions. The first drum was 3 feet (0.9 m) in diameter and 8 feet (1.2 m) long. The drum was divided into three sections by partitioning rings and was powered by a variable speed drive unit giving drum speeds of 2 to 15 rpm. The fertilizer substrate was preheated with hot air to about 160°F in the first section, coated with molten sulfur 300°-310°F (150°-155°C) in the second, and treated with a mixture of molten wax and microbicide in the third section. Temperature of the wax microbicide mixture was 200°-220°F (95°-105°C). The drum was lined with felt for insulation and to avoid slippage of the bed. Felt strips ½-inch thick were installed in the sulfur coating section to obtain better rolling action. Later, 1-inch triangular flights were installed and still later transverse rings about 1-inch high were used. (Advantages for internal structure that in later tests gave very marked improvement will be discussed later.)

A single Spray-co nozzle was used in the initial tests. Later, two or three nozzles were used for higher production rates. The nozzles were positioned to direct the spray onto the most active area of the bed which was just below the upper edge. Hot air at 300°F (150°C) was used to atomize the spray of molten sulfur. The molten wax plus microbicide was dripped onto the bed in the third section of the drum. Good coating was obtained with a very short retention time—about 3 minutes—in the coating section.

The second drum was 2 feet (0.6 m) in diameter and 3 feet (0.9 m) long. This drum tumbled the granules slowly while they were being cooled to about 105°F (60°C) and then conditioned with clay or diatomaceous earth. Cooling prior to conditioning was necessary to prevent the

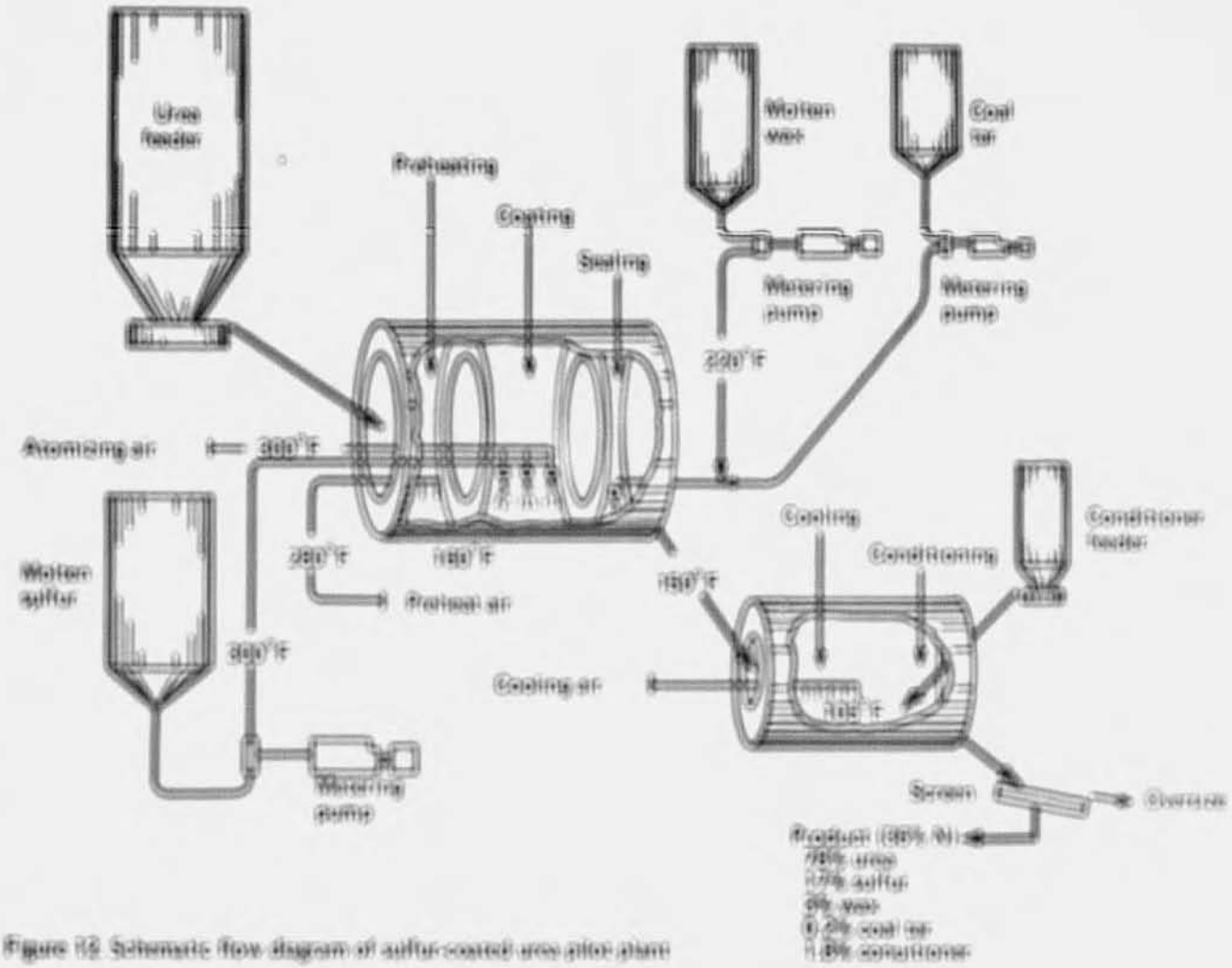


Figure 12. Schematic flow diagram of sulfur-coated urea pilot plant

conditioner from embedding into the hot wax and thus becoming ineffective.

Positive-displacement, remote-head diaphragm pumps were used for metering the liquid streams of sulfur, wax, and coal tar. Disk-type feeders were used for controlling the feed rate of solids. The nozzles used for spraying sulfur were of the pneumatic (air atomized) type in all of the early work.

Temperature recorder-controllers were provided for the heated liquid streams and for hot air at 300°F (±20°C) to the spray nozzles and preheat section.

In the early phases of pilot-plant operation, difficulties were encountered with freezing of sulfur in the small feed line. It was found that complete steam jacketing and heavy insulation of the sulfur lines alleviated the freezing problem. Pump stoppage was virtually eliminated by installing 100-mesh stainless steel screens in the intake to keep trash out of the check valves. A similar strainer in the sulfur header minimized plugging of the small orifices in the spray nozzles.

Both the coating drum and the cooler-conditioner drum were adequately vented with a duct-blower system to prevent escape of sulfur mist, wax-coal tar fumes, and conditioner to the operating area.

Feed Materials

A number of substrate feed materials were used during the course of operation of the small pilot plant. These included urea of the air-prilled, spray-drum-granulated, pan-granulated, and pug-mill granulated types and granular potassium chloride of the flotation crystallized and recrystallized types. Urea in the various forms was the predominant substrate used. The spray-drum-granulated and pan-granulated types of urea were used in most of the test work because of the larger, more favorable granule size.

The elemental sulfur used in the pilot-plant work was commercial bright, run-of-mine sulfur. Plasticizers (extenders) were not used because it was found in latest bench-scale tests that they offered no significant advantage

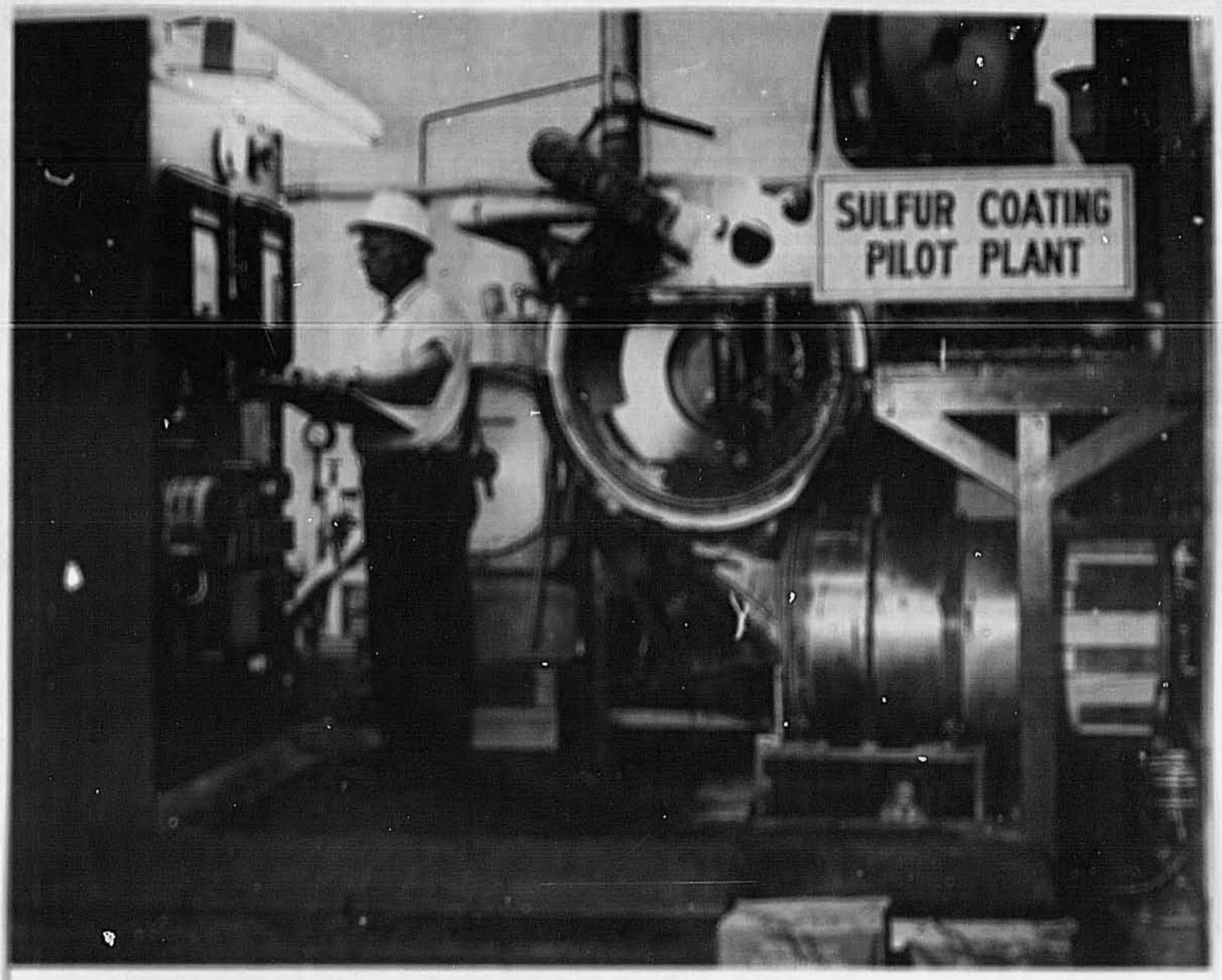


Figure 13. Sulfur coating pilot plant

over unplasticized sulfur after a better coating technique was developed.

The sealant used in the pilot-plant work was a microcrystalline wax (believed to be commercially available) that has an oil content (ASTM) of about 8% to 10% and a melting point of 167°F (75°C). The wax (Sheltmax 800) is a byproduct of petroleum refining. Originally about 0.5% coal tar was added with the wax, but later about 0.25% was found to be adequate. In later work, the microbicide was omitted.

Diatomaceous earth was used almost exclusively as the conditioner in the pilot-plant work because it was readily available. Others found to be effective in benchscale tests include kaolin clay, talc, and vermiculite.

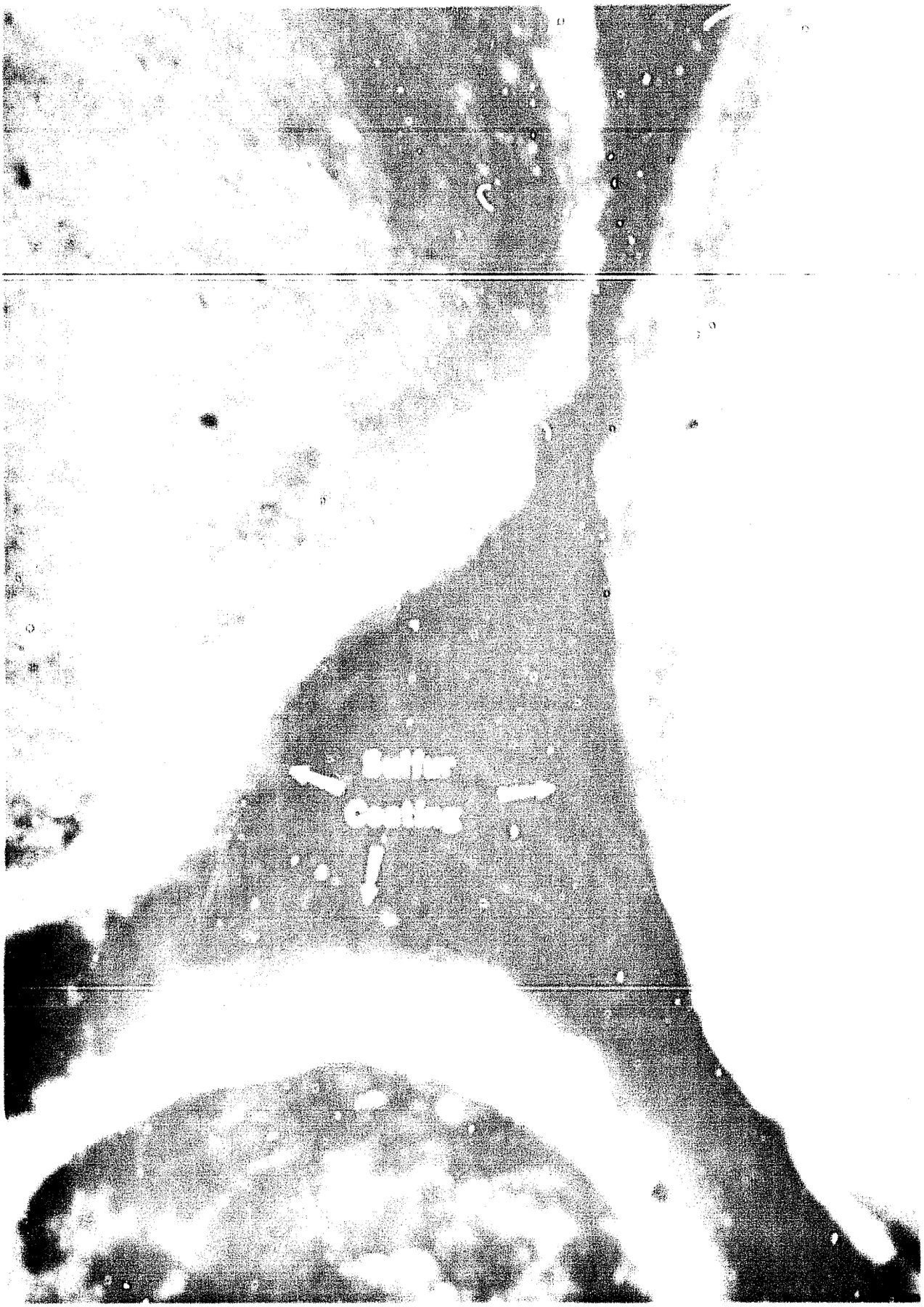
Product Characteristics

The sulfur coating process, as has been described, provides a three-component, laminated coating consisting of first a layer of elemental sulfur, then a thin film of microcrystalline wax, and finally a layer of conditioner. A cross-sectional photomicrograph of typical coated granules is shown in figure 14. The rather uniform outer coating can be readily observed.

The range of contents of the coating components follows: sulfur, 10% to 25%; wax, 2% to 3%; microbicide, 0.25% to 0.50%; and conditioner, 1.5% to 2%. The range of content of the various coating components gave a nitrogen content for the coated urea prills or granules of 37% to 38%. This is still a quite high-analysis fertilizer.

Operating Conditions for the Pilot Plant

In the initial pilot plant studies, only a single sulfur spray nozzle was used. It was apparent that the product quality did not approach that of small batchwise coating. It was believed that the very short retention time under one spray did not allow full and uniform coverage of the


Buffer
Coating

granules with sulfur. A second spray nozzle was added and later a third one. With each addition of a nozzle, improvement in uniformity and quality of the coating were obtained. The number of sprays was not increased above three because of space limitations in the drum.

The pilot plant drum was sized to provide an overall retention time of about 6 minutes at a 300 lbs/hr (140 kg/hr) rate of production; this was provided by a loading (by volume) percentage of 2.5 at a bed depth of 2 inches (5 cm). This bed depth and retention time was regulated by adjustment in depth of the retention ring.

Temperatures of the various feed streams that were found to be best in the small scale work were used in the pilot plant. The various temperature levels used in virtually all of the pilot plant work were as follows:

Material	Temperature range, °F
Substrate	155-165 (70°-74°C)
Sulfur	295-300 (146°-150°C)
Preheat air	280-300 (138°-150°C)
Atomizing air	300-305 (150°-153°C)
Wax (plus coal tar)	200-220 (93°-104°C)
Coal tar	ambient
Product cooler discharge	90-100 (32°-38°C)

Effects of Principal Process Variables

Coating weight (thickness)—The rate at which the soluble substrate diffuses through the coating is generally a function of the thickness of the coating. The nature of the sulfur as deposited on the granules is also very important. With standardized conditions in the coating process, the weight of the coating generally can be correlated with rate of dissolution. The variation in dissolution rate with coating weight for typical tests is shown in figure 15. The substrate was spray-drum-granulated urea.

The average initial 7-day dissolution rate increased from only 6% with a 30% total coating to over 60% for a 12% total coating. The average daily rate (between 7 and 14 days) varied only from about 0.3%/day to 1.4%/day.

Substrate particle size and shape—Because of the spherical geometry involved, the surface to volume ratio of small particles is much greater than that of large particles. As a result, for a given coating weight, the coatings on small particles are much thinner and less effective (higher dissolution rate) than those on larger particles. For this reason, granules or prills of the largest practical size were used in most of the pilot plant work. Spray-drum-granulated and pan-granulated urea were particularly good because of the larger, more uniform size. The effect of particle size of urea on dissolution rate (with other conditions constant) is shown graphically in figure 16. Although the coating weight was quite high (27% to 30%), the markedly slower dissolution for the larger particles is readily evident.

In addition to the particle size, the quality of the granules or prills is quite important. Spherical particles with smooth surface are best suited to accept a good coating.

Sulfur spray conditions—Several variables were studied to evaluate the effect of spraying conditions on quality of

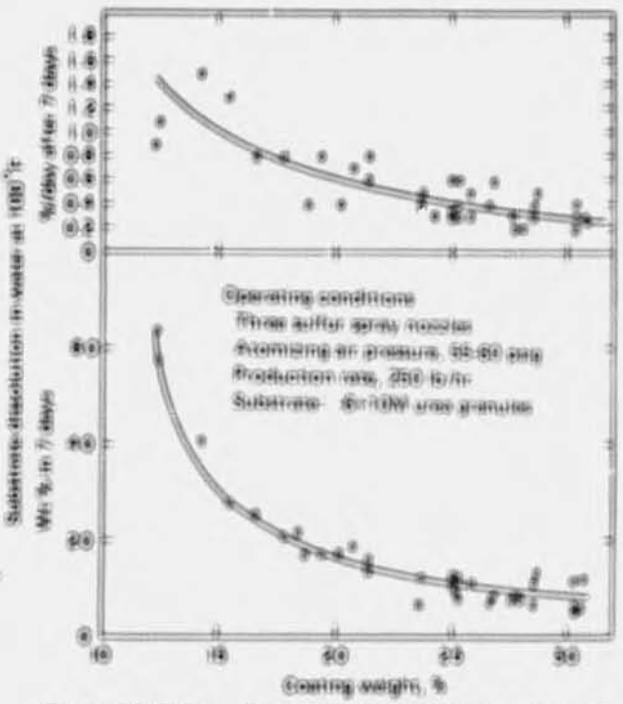


Figure 15. Effect of coating weight on dissolution rates

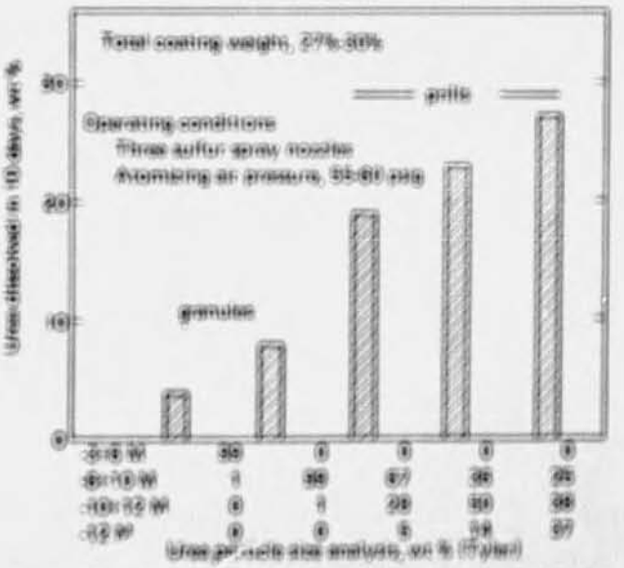


Figure 16. Effect of substrate particle size on dissolution rate

the coating. Variables studied included atomizing air pressure, nozzle type, distance from the nozzles to the bed, spray nozzle discharge rate [lb sulfur/(hr)(nozzle)] and production rate.

The effect of atomizing air pressure (pressure drop across air jets) on coating quality is shown in table 6. With the urea prills as substrate, coating quality improved at all coating weights as the atomizing air pressure was increased from 30 to 60 psig. As air pressure was increased, dissolution decreased from the range 39% to 70% to a range of 27% to 40%. With granules, the improvement in quality due to pressure increase from 60 to 80 psig was more pronounced; dissolution rates of the products decreased about two-thirds. Further increase in air pressure to 100 psig did not result in significant improvement.

Later tests with an air compressor providing nozzle pressures of 80 to 100 psig confirmed the fact that the maximum effect was achieved by increasing the pressure from 60 to 80 psig, with little further improvement at 100 psig. These data are shown in figure 17. Also shown is the effect of production rate on product quality; with a coating weight of 20%, decreasing the production rate from 200 lbs/hr to 150 lbs/hr resulted in a decrease in dissolution rate from 18% to 14%.

The pneumatic sulfur spray nozzles used in the test work described above were of the constricted external-mix type having diametrically opposed wing air jets and a central air jet concentric with the sulfur jet. A sketch of a cross-sectional view of the type nozzle is shown in figure 18. The volume of air going to each of the jets was governed by two internal replaceable orifices. The ratio of the air volumes to each jet had a significant effect on coating quality. The most effective configuration was a 0.06-in., (1.5-mm) wing jet with a 0.08-inch (1-mm) central jet. This configuration was used in most of the work with pneumatic nozzles.

Other spray system variables studied included number of spray nozzles used, height of nozzle discharge above the bed, cone or fan spray pattern, and production rate. The most effective coating was obtained with 2 or 3 nozzles, 5- to 6-inch distance of sprays above the bed, and low to moderate sulfur rates of up to 65 lbs of sulfur/nozzle/hour. There was very little performance difference between the

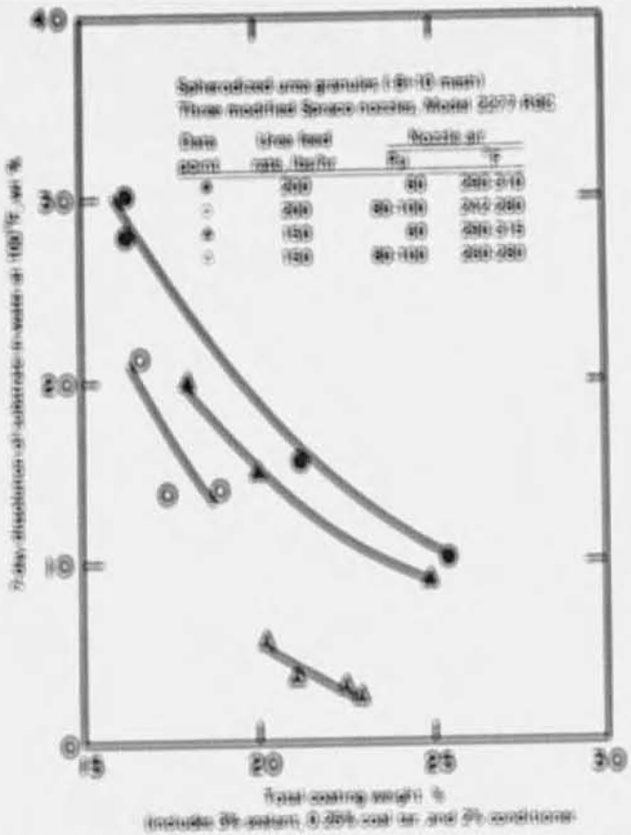


Figure 17. Correlation of dissolution rate with coating weight as affected by atomizing air pressure and urea feed rate

Table 6. Effect of atomizing air pressure on product dissolution rates

Average total coating, wt %	Substrate dissolution in water at 100°F (38°C), wt %[a]				
	Atomizing air pressure, psig[b]				
	30	45	60	80	100
Urea prills (95% -6+14 mesh)					
23	70	60	40		
28	57	42	31		
31	39	28	27		
Spherodized urea granules (99% -6+10 mesh)					
20			16	6	4
23			9	3	3

[a] 5 days for prills and 7 days for granules.
[b] Three spray nozzles.

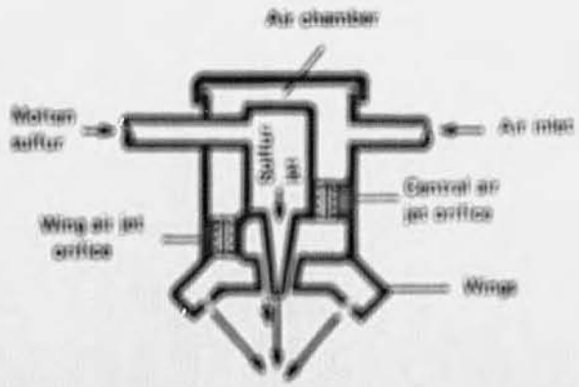


Figure 18. Schematic cross-sectional diagram of sulfur spray nozzle

cone-type and fan-type nozzles at these low to moderate production rates.

Coating of Granules or Prills Prior to Sulfur Coating

Patents have been obtained in the United States and United Kingdom by others on advantages of precoating particles with finely pulverized carbon black prior to sulfur coating. Tests were made in the laboratory to study the effect of precoating with about 1% of carbon black. In some tests 3% of wax sealant was applied and in others only 1%. The effect of carbon black at the two levels of wax content is shown in figure 19.

The results show that carbon black was highly effective with 1% of wax sealant, with 3% of wax as sealant there was no appreciable advantage for the precoat of carbon black. Use of the lower proportion of wax resulted in less tackiness of the product but also resulted in a significantly higher dissolution rate when the total coating was lowered.

Tests of Various Waxes

Nine different waxes from two suppliers were tested as sealant on SCU. Only two of these gave results as good as the microcrystalline wax (Shellmax 300) used in most of the pilot-plant test work. These were Gulf Petrolatum X-22182, and Texaco Petrolatum 1231. Although no sealant was found to be significantly superior to Shellmax 300, the two other effective waxes could provide alternative sources of supply. Both of the alternative types of wax gave effective protection in soil burial tests of 14 days duration.

The melting points and oil content of the three waxes are shown below:

Sealant designation	Melting point, °F	% oil in wax
Shellmax 300	167 (75 °C)	10-11
Texaco Petrolatum 1231	140 (70 °C)	9.7
Gulf Petrolatum X-22182	168 (75 °C)	7.0

Cost of the waxes is about the same. However, in the current (1974) petroleum shortage, these petroleum waxes have proven to be difficult to obtain in large quantities under contract.

Tests of Lighter Coatings

Results of agronomic testing indicated that the coatings did not need to provide as slow rate of dissolution as had been sought in the early work. Therefore, tests were made in the small pilot plant with coatings (total of sulfur, wax, and microbicide) of 10% to 26%. Normal conditions of operation were used. These were:

Use of 3 sprays
60 psig atomizing air pressure
160°F (70°C) substrate preheat

In one series the production rate was 200 lbs/hr (90 kg/hr) and in the other was 400 lbs/hr (180 kg/hr). Results are plotted in figure 20.

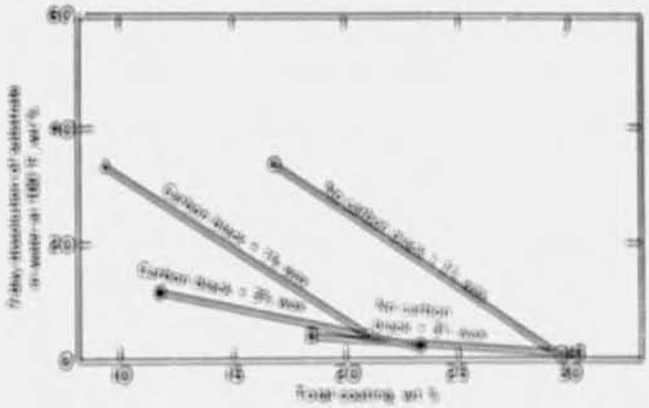


Figure 19. Effect of coating weight on dissolution rates of sulfur-coated spheroidized urea with and without carbon black precoat

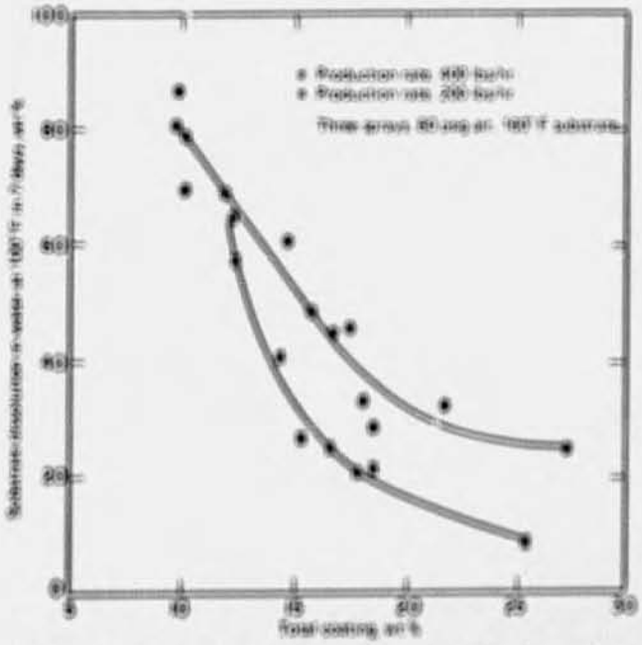


Figure 20. Effect of coating weight and production rate on dissolution rate of sulfur-coated commercial granular urea of regular size (-6+16 mesh)

The 7-day dissolution rates were lower for the materials made at the lower production rate. For example, with 20% coating weight, the low production rate gave material with about 16% dissolution in 7 days. The higher rate of production resulted in a 7-day dissolution of about 32%. With 15% coating weight, the dissolution rates were 32% and 52% respectively. With the 15% coating weight, the dissolution rate (7- to 14-day period) for products made at the two rates of production was about 1.2%/day. As the total coating weight was increased to 25%, the daily dissolution rate decreased to 0.9% for the high production rate and 0.1% for the low production rate. These latter rates are likely lower than would be desirable for most agronomic uses.

Design Data for Larger Pilot Plant

Several tests were made in the small pilot plant to obtain information for use in design and operation of a larger 1-2 tons/hr (0.9-1.8 mt) pilot plant. The effects of the following variables were investigated.

Fan (NE3M)[1] versus cone (R9C)[2] sulfur spray nozzles
Distance from spray nozzles to substrate bed, 5-9 inches (13-23 cm)
Number of spray nozzles, one-three
Sulfur feed rate, 12 to 190 lbs (6 to 86 kg)/hr/nozzle
Urea feed rate, 140 to 660 lbs/hr (64 to 300 kg/hr)

Spray-drum-granulated urea was fed in all of the tests. Conditions of temperatures throughout the process, drum speed, etc., were those found to be best in previous tests. Results are plotted in figure 21 to show the relationships between coating weight and 7-day dissolution rates as affected by the interactions of other variables.

The data show that the most effective coatings (lowest dissolution rate for a given coating weight) were attained with:

Multiple spray nozzles (two or three) versus one nozzle
Five- to six-inches spray distance versus nine inches
Low to moderate sulfur spray rates up to about 70 lbs (27 kg)/hr/nozzle.

Use of Pressure Type ("Hydraulic Nozzles)

Pressure, or "hydraulic," nozzles that depend only upon fluid velocity for atomization have several advantages over the air-atomized "pneumatic" type. These include simplification of the spray header and, most important, greatly decreased formation of sulfur mist and dust in the coating drum. The decreased mist and dust would make dust and fume control, to avoid pollution and environmental problems, much easier. Also, any explosion hazard due to

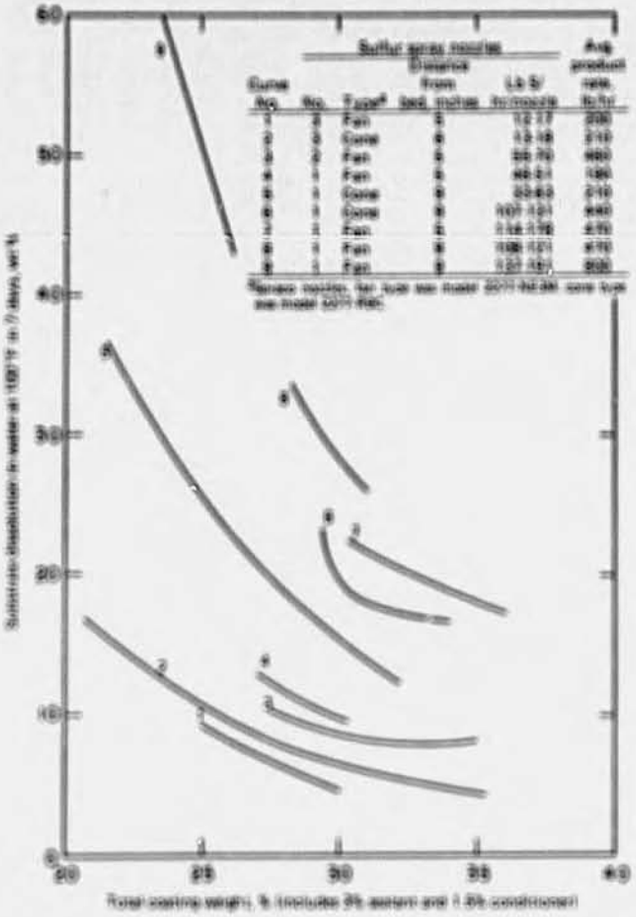


Figure 21. Substrate dissolution rate versus coating weight for sulfur-coated spherodized ureas made under various operating conditions

explosive mixture of sulfur mist and air in the coating drum would be essentially eliminated.

Two hydraulic nozzles were installed in the coating drum of the small pilot plant. These were Spray-Dry nozzles[3] (type SX, No. 74 orifice, 0.0225-inch diameter) with a 70-degree hollow cone pattern. Pump difficulties limited the sulfur feed rate to a maximum of 75 lb (34 kg)/hr/nozzle. Maximum production rate was about 800 lbs/hr (360 kg/hr). The nozzles were positioned at 5½-7½ inches (14-19 cm) above the bed and directed toward the most active area just below the upper edge. Bed depth was about 3 inches (7.5 cm). Commercial spray-drum-granular urea was coated in these tests. Because the production rate exceeded capacity of waxing, cooling, and conditioning, these operations were carried out in the laboratory on representative samples. Results of the tests at 450-1,000 lbs/hr (205-455 kg/hr) rate of production are plotted in

[1] Spraying Systems Company, Bellwood, Illinois.
[2] Ibid.
[3] Ibid.

figure 22. Also included are results of tests with R9C[4] air-atomizing nozzles for comparison.

The hydraulic nozzles yielded products with dissolution rates as low as were made using the air-atomizing nozzles. The height above the bed had little effect. These initial results were quite promising.

In further tests of hydraulic type (pressure atomizing) nozzles several types were tested. However, only those of the type used in spray drying (SX) gave good results. The latest work was with SX-80 (0.017-inch orifice), SX-74 (0.023-inch orifice), or SX-69 (0.029-inch orifice) nozzles.

A new high-pressure diaphragm pump[5] was installed in the pilot-plant system for these tests. It had a capacity of 180 lbs of sulfur/hr (82 kg/hr) at 700 psig back pressure. In initial tests, two SX-74 sprays were used; SX-80 sprays were tested also, but plugging of the very fine orifices prevented their use. Tests with the SX-74 nozzles were made using 700 psig sulfur pressure, 450-900 lbs/hr (205-410 kg/hr) production rate, drum speed of 15 rpm, bed temperature of 130°-140°F (54°-60°C), sulfur temperature of 300°F (150°C), and nozzle height of 5½-7½ inches (14-19 cm). The nozzles were tested at different angles but most tests were with the nozzles directed at the most active area of the bed near the top.

The data indicated that the pressure-type sprays give as good quality products as the air-atomizing nozzles at high sulfur feed rates of 50-90 lbs (23-41 kg)/hr/nozzle or 600-900 lbs/hr (273-410 kg) production rate. At 25% total coating weight, the 7-day dissolution rates were in the following order.

Air sprays, 900 lbs/hr (410 kg/hr) rate, 34% dissolution

Angled SX-74 sprays, midbed 700 lbs/hr (320 kg/hr) rate, 29% dissolution

Vertical SX-74 sprays, 600 lbs/hr (273 kg/hr) rate, 26% dissolution

Air sprays, 450 lbs/hr (204 kg/hr) rate, 22% dissolution

Angled SX-74 sprays, 600 lbs/hr (273 kg/hr) rate, 17% dissolution

Results with the vertical SX-74 nozzles appeared to be unaffected by production rates of 490-940 lbs/hr (225-430 kg/hr). The angled SX-74 nozzles at 600 lbs (275 kg) of product/hr gave better quality product than did air atomized nozzles at 450 lbs/hr (205 kg/hr).

The only major difficulty with the pressure (hydraulic) nozzles was plugging of the small orifices (0.017-0.029 inch) with impurities in the sulfur. Use of sprays with larger orifices was not practical because of the limitations in feed rate of the sulfur pump. Installation of very fine filters in the sulfur supply system essentially solved this problem.

The good results with hydraulic (pressure type) nozzles at higher rates of production, together with the greatly

[4] Spray Engineering Company, Burlington, Massachusetts.
[5] American Lewa, Inc., Framingham, Massachusetts.

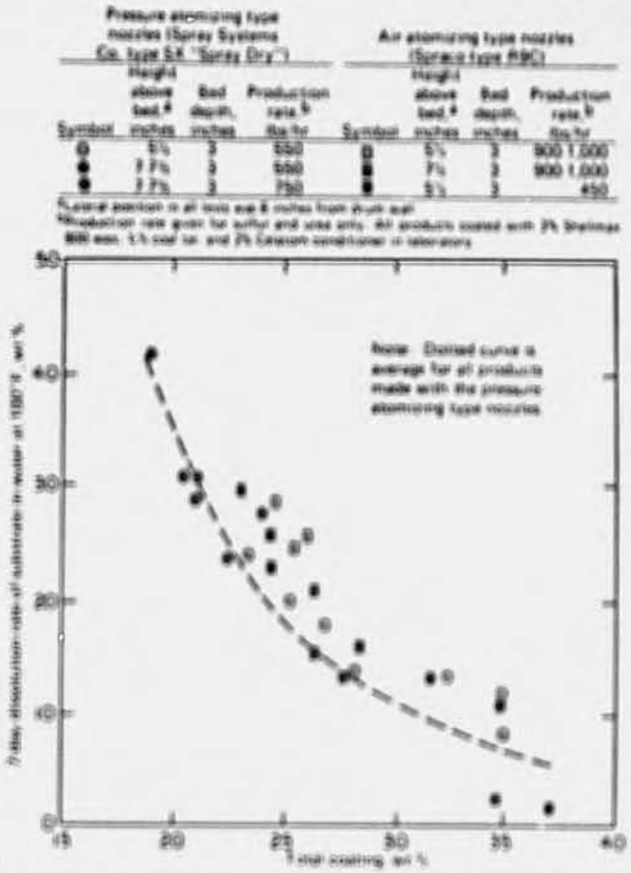


Figure 22. Sulfur coating of -6+10 mesh granular (Cominco) urea in small pilot plant—effects of coating weights and nozzle type and position on dissolution rate of product

decreased dust and mist in the coating drum, resulted in this type of spray nozzles being recommended.

Use of Flight System in Coating Drum

The coating drum of the small pilot plant was modified by installing a flight system. The purpose of the flight design was to provide a continuous, falling cascade of granules for spraying with sulfur. The principal of the flight arrangement is illustrated in figure 23. The flights were arranged so that they provided a uniform loading of granules to the pan-type deflector. Material rolling from the pan provided a continuous cascade of fast moving granules that could be sprayed by nozzles located as shown in figure 23.

The purpose of the flight-cascade design was to provide a high degree of particle-size homogeneity in the bed and falling cascade, thereby improving the uniformity of exposure to the sulfur sprays. This, in turn, permitted a faster movement of the substrate through the coating section without sacrificing product quality. Additionally, the scheme increased the area available for spraying per unit

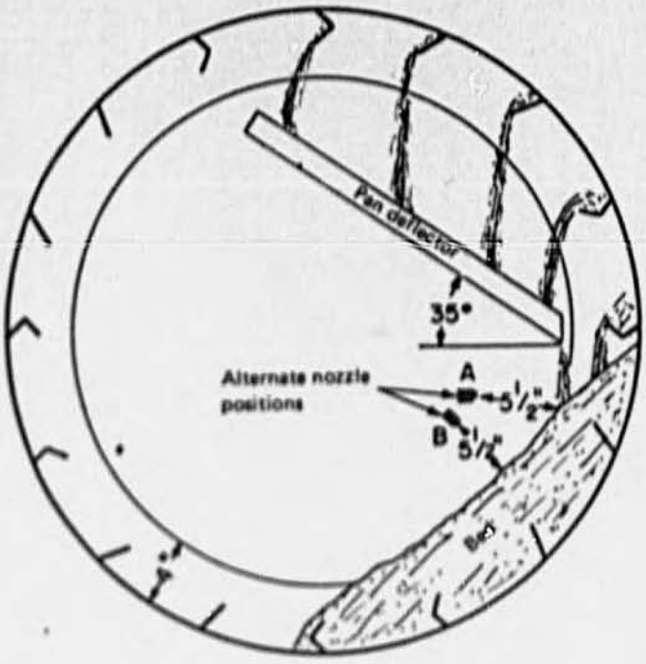


Figure 23. Flighted, cascading bed used in sulfur coating drum

of drum length. This would be important in designing a large-scale coating drum (more sprays could be used effectively).

Two pressure-atomizing (hydraulic) nozzles were used in the drum. They were hollow-cone nozzles of two types. In some tests, the SX-69[6] (0.029-inch orifice) nozzles were used and in others the two nozzles were of the SX-74[7] type (0.023-inch orifice).

Speed of the 3-ft (0.9-m) diameter drum was varied from 4 to 9 rpm in the tests. Samples were taken when sprays were positioned in the two locations A and B as shown in figure 23. Position A aimed the nozzles at the top of the bed where the curtain of granules flowed from the deflector onto the bed. In position B the nozzles were aimed toward the center of the bed. Other conditions were sulfur temperature, 304°-308°F (150°-153°C); sulfur supply pressure, 700 psig; bed temperature, 135°-145°F (57°-63°C); and production rates of 600-1,050 lbs/hr (270-480 kg/hr). It was obvious from observation of the bed action that good conditions for uniform coating were afforded. Since cooling capacity in the small pilot plant was not sufficient for these high rates, the waxing, cooling, and conditioning operations were carried out on representative samples in the laboratory. (The use of a microbicide had been discontinued prior to these tests on the basis of latest agronomic test results.)

Portions of the samples that were waxed and conditioned in the laboratory were submitted for dissolution tests. The results are plotted in figure 24 for comparison with previous results when using the standard R9C air-atomizing nozzles and when using the SX-74 pressure-atomizing nozzles. Best previous results were selected for the two types of nozzles when used in the drum before the flights were installed.

The data shown for the R9C nozzles were obtained with lower production rates of 200-450 lbs/hr (90-205 kg/hr). Lower production rates generally result in higher product quality. The production rates with the "cascade" flight arrangement were 1.3 to 4.5 times those used in the other tests.

In general, the dissolution rates of the products made with the cascade flight system installed in the coating drum were equal to or lower than without the cascade, regardless of production rates or number or type of spray nozzles used. For example, the dissolution rates of products made at a 600 lbs/hr (270 kg/hr) production rate with the cascade flight system and two pressure nozzles were about one-fourth lower than those of products made at a production rate of only 200 lbs/hr (90 kg/hr) with three air atomizing nozzles without the cascade arrangement in the coating drum. Also at 23% total coating weight and a 600

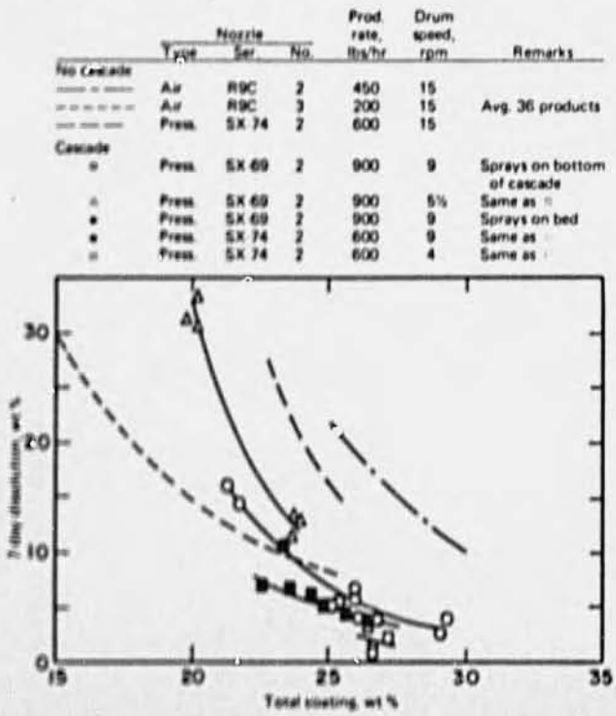


Figure 24. Effect of coating weight and operating conditions on product quality using pressure atomizing nozzles and a cascade flight arrangement

[6] Spraying Systems Company, Bellwood, Illinois.
[7] Ibid.

lbs/hr (270 kg/hr) production rate with the cascade, the products had a dissolution rate of 7% in 7 days as compared with 25% without the cascade. Results of the test with the nozzles aimed at the center of the bed were equal to those with the nozzles aimed at the junction of the cascade and the bed.

These results were considered to be very good, particularly in light of good quality products being obtained at the high production rate of 900 lbs/hr (410 kg/hr). This will be particularly important in determining the size of drum required for a plant-scale unit.

Difficulties were encountered due to stoppage of the very small orifices in the sulfur spray nozzles with carbon deposits. Use of a 400-mesh screen in the sulfur supply system appears to solve this problem.

THE LARGE PILOT PLANT

Laboratory tests and studies in the small pilot plant established the effect of most of the process variables. Best conditions of operation were delineated, and procedures for evaluation of the products were established. There was a need, however, to scale-up to a larger pilot plant to provide design data that would be needed for a commercial sized unit and to provide tonnage quantities of products for more widespread agronomic evaluation under actual crop conditions.

Description of the Pilot Plant

Construction of the nominal 1 ton/hr pilot plant was completed and initial test operation started in July 1971. An early flowsheet for this pilot plant is shown in figure 25.

The pilot plant consisted of a urea storage hopper, urea feeder, urea preheater, sulfur-coating drum, wax-coating drum, fluidized-bed cooler, conditioning drum, and auxiliary process equipment. The elevated urea feed hopper held about 3 tons. Urea flowed by gravity from this hopper into a volumetric feeder (set on weigh scales) that controlled the rate of feed to the preheater. The preheater was a rotary drum about 2 feet (0.6 m) in diameter and 6½ feet (2 m) long. Heat was provided by six 440-volt electircal resistance-type radiant heaters installed inside the rotating drum. The speed of rotation was variable. The preheated urea flowed by gravity to the coating drum (heart of the process) that was 4 feet (1.2 m) in diameter and 6 feet (1.8 m) long. A retaining ring about 3½ inches (9 cm) high was installed at the discharge end; the drum contained no flights and was insulated outside (flights were installed later).

Bright sulfur with carbon content of 500 ppm and ash content of about 55 ppm was used. The sulfur was stored in molten form in a railroad tank car on a spur track from where it was fed by air pressure to a scale-mounted feed tank at the pilot plant. The lines were completely steam-jacketed. A submerged diaphragm-type metering pump was used to regulate the rate of feed of sulfur to the spray header located in the coating drum. (A high-pressure, double-acting, piston-type pump was used to feed sulfur later when hydraulic-type nozzles were tested.)

Sulfur was supplied from the header to four or eight spray nozzles. Uniform flow to each nozzle was assured by fixed orifices 0.018 inch (0.5 mm) in diameter. This orifice system is imperative to obtain uniform rate of feed to each spray nozzle. The sulfur feed line and the header in the coating drum were fully steam-jacketed. The header was mounted so that essentially the full length of the bed of granules was covered, and sprays were directed to the most active area of the bed. Height of sprays above the bed also was adjustable. (A dual header was tested with higher production rates.)

The wax-coating drum was located at an upper platform level; therefore, the SCU was conveyed by bucket elevator to this drum. The drum was about 3 feet (0.9 m) in diameter and 2 feet (0.6 m) long. Melted wax was fed to this drum from a steam-heated and insulated tank with a small, submerged diaphragm-type pump. Coal tar microbicide was fed from a small container and metered with a small metering pump. The wax and coal tar mixed in a small pipe tee at the wax coating drum and were distributed by a "drip-tube" arrangement. Good coating with wax and coal tar was obtained. (In later test work, the use of microbicide was discontinued. Also, elimination of the wax to provide a sulfur-only coating was found to be practical.)

The coated and waxed material flowed by gravity through a rotary air lock valve into the fluidized-bed cooler that was located at an intermediate level. This cooler (Buttner type) had a bed area of 10 ft^2; it has been used to cool 2 tons/hr effectively. Air for fluidization and cooling was supplied by a centrifugal fan. The ductwork and damper arrangement allowed close control of airflow into the cooler. Fluidization was effected by airflow through a finely perforated plate or screen that covered the full cross-sectional area of the cooler. The air suspends the particles being cooled, above the screen in the cooler, giving the bed the appearance of a boiling fluid. The fluidizing principle allows excellent contact between the air and the

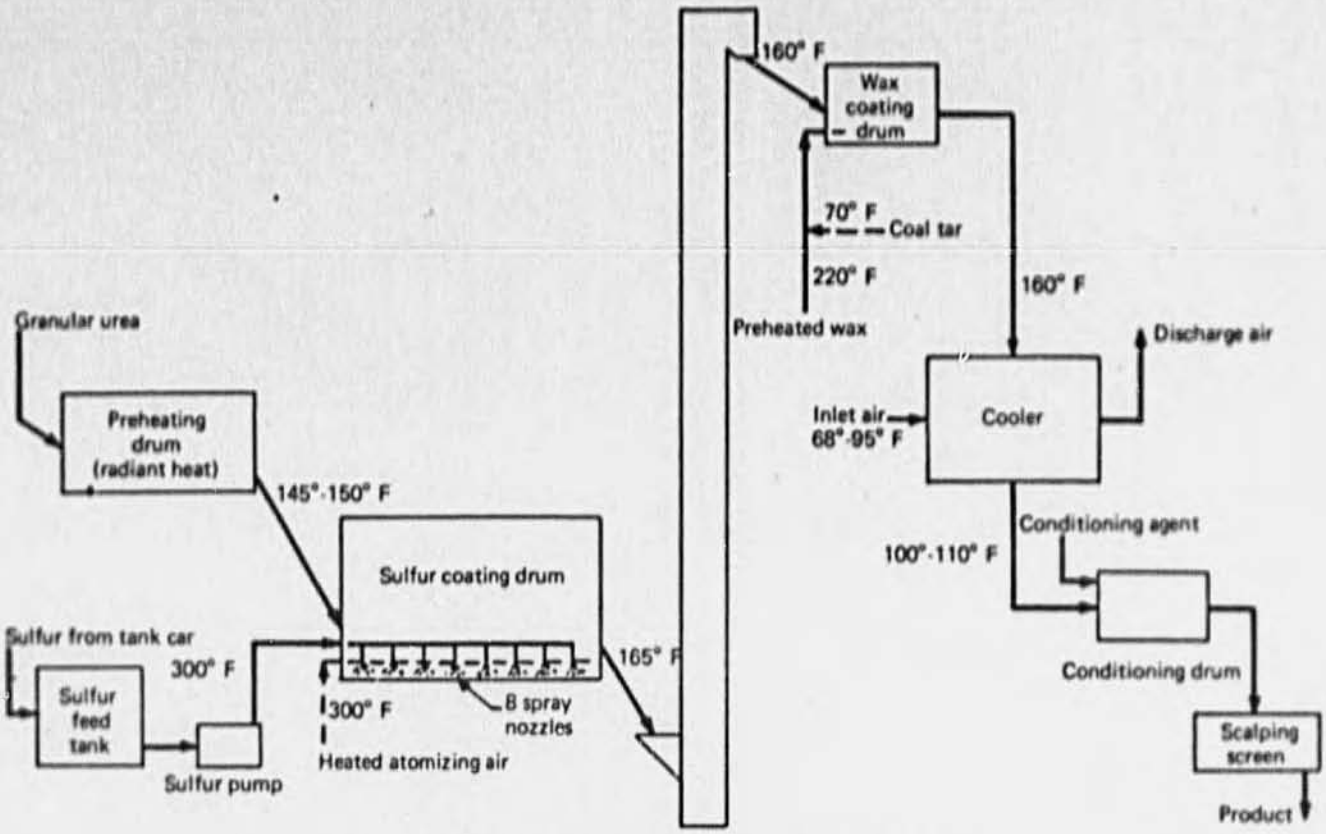


Figure 25. Basic flow sheet for sulfur-coated urea pilot plant

particles. This results in very good heat transfer from the particles to the air. As cooling proceeds, the wax solidifies, leaving only a slightly tacky surface that requires a conditioner to allow good handling and storage properties.

The fluidized bed unit was very efficient, giving discharge product temperatures within 5°F of the input air. Air from the cooler was vented to a stack. The cooled product flowed by gravity through a rotary air lock valve to the conditioning drum at ground level.

The conditioning drum was about 3 feet (0.9 m) in diameter and 3 feet (0.9 m) iong and had a smooth shell. Diatomaceous earth conditioner was metered to the drum with a vibrating screw-type feeder. Kaolinite was much less effective as a conditioner. Conditioning is required to counteract stickiness caused by residual oils in the wax. The conditioned product discharged onto a belt conveyor that fed a small 4-mesh, oscillating scalping screen. Only a very small amount of oversize (about 0.3%) was removed. Product from the screen was conveyed to a bagging hopper.

Bucket elevators were used to fill the urea feed hopper and to elevate the sulfur-coated material to the waxing drum. All rotary equipment except the conditioning drum was driven with electric motors through variable speed drive units. The conditioning drum had a fixed speed drive of 12 rpm. Instrumentation was provided throughout the pilot plant to obtain needed process data.

A photograph of the pilot plant is shown in figure 26.

Operation of the Pilot Plant Using Pneumatic Spray Nozzles

Typical operating temperatures are shown in figure 25. Urea was preheated to a temeprature of 140°-150°F (63°-66°C) before being fed to the coating drum. Molten sulfur was fed at a temperature of about 300°-310°F (150°-155°C) and air for atomization of the sulfur was heated to 300°F (150°C). The eight orifices in the header (one for each pneumatic nozzle) ensured an equal flow rate from each nozzle. The sulfur-coated material leaving the coating drum was at a temperature of about 165°F (74°C) and cooled to about 160°F (71°C) as it was elevated to the waxing drum. Wax was melted and heated to about 220°F (105°C) and then mixed with coal tar that was at ambient temperature of about 70°F (21°C). The mixture of wax and coal tar oil dripped onto the bed of granules and coated them evenly. (Coal tar was omitted in later test work.) The waxed product was then cooled from 160°F (71°C) to 100°-110°F (38°-43°C). Inlet air temperature to the

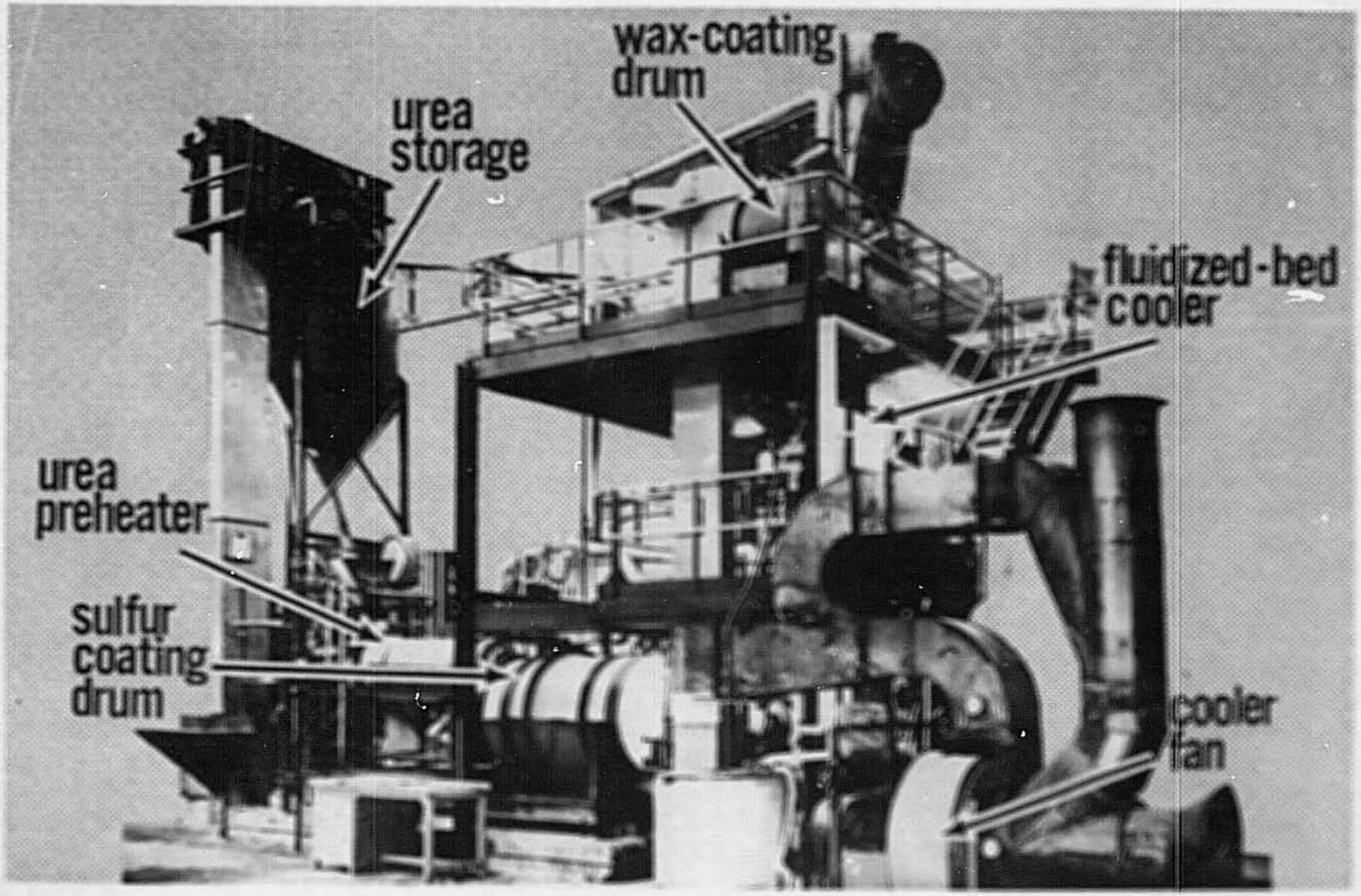


Figure 26. Pilot plant for sulfur coating urea

fluidized bed cooler ranged from 68° to 95°F (20°-35°C).

The pilot-plant system operated very well. No major changes in equipment were required. A change from pneumatic nozzles to the hydraulic type was made in later operation in order to test this simpler system. This will be discussed later. A very effective flight system was installed in the coating drum; this is discussed in a later section. A dual spray header was also tested. The preheater using electrical, radiant heat gave very good service at production rates of up to 2 tons/hr. Time cycling switches were used to vary the heat input. The smoothing of the surface of the granules that occurred in the preheater apparently was beneficial to the coating process. The spray header in the sulfur-coating drum gave very good service; the individual orifices were very effective in ensuring equal flow rate to each of the nozzles. The concentrations of sulfur mist and dust in the coating drum and exit gas were quite high when pneumatic nozzles were used (0.07 to 0.16 gram/m^3 of air/lb of sulfur sprayed). This would involve a difficult recovery problem and there possibly could be a potential explosion hazard (due to sulfur-air mixtures in the coating drum). These problems were essentially eliminated in later tests when hydraulic nozzles were used. The variable speed feature for the coating drum allowed tests over a range of 13-33 rpm. Best operation in the unflighted drum generally was at 24 to 26 rpm, which is about 65% of critical speed. (Critical speed is determined by the relationship $\frac{76.5}{\sqrt{D}}$ where D is diameter of the drum in feet.)

Waxing and treating with microbicide (when used) were easily accomplished in the small waxing drum. The fluid bed cooler was particularly effective and efficient. No difficulty was encountered with buildup on the screen or other parts of the unit. There was no appreciable agglomeration of particles as they passed through the cooler. Good cooling was obtained at rates as high as 2 tons/hr. Good coating of the product with conditioner was accomplished in the coating drum. The conditioner (diatomaceous earth) was applied uniformly. Usually, there was less than 0.3% of large (agglomerated) particles to be removed by the 4-mesh scalping screen.

Typical operating conditions in producing sulfur, wax, and microbicide coated urea are shown in table 7. These data are for operation at 1 ton/hr using the pneumatic-type sulfur spray nozzles. Temperatures are similar to those found to be best and shown in figure 25.

Products were evaluated by immersion of 50-gram samples in water at 100°F (38°C) for 7 or 14 days to determine dissolution rate.

Feed materials.—Most of the urea used in the large pilot plant was either commercial, spray-drum-granulated material of -6+10 mesh size or TVA granular urea -6+10 or -5+8 mesh size made in a pilot plant pan granulator. Commercial prilled urea was used in some tests; coating was effective on prills with wax and conditioner but not with sulfur alone. A heavier coating was required with prills (20% sulfur, 3%

Table 7. Operating conditions for sulfur coating (full coating)

Test No.	SCU-438	
Substrate, type	Granular urea (-6+9 mesh)	
Feed rate, lbs/hr	1,620	(740 kg/hr)
Preheater		
Substrate exit temperature, °F	135-137	(57°-58°C)
Retention time, min.	9	
Rpm	20	
Sulfur-coating drum		
Sulfur feed rate		
Per nozzle, lbs/hr	20.4	(9 kg/hr)
Total, lbs/hr	163	(74 kg/hr)
Sulfur feed temperature, °F	305	(145°C)
Spray nozzles		
Model	BOC	
Number	8	
Distance to bed, inches	4-3/4	
Sulfur flow control orifice size, inches	0.018	
Atomizing air		
Volume per nozzle, std. ft^3/min	5.5	
Temperature at nozzle tip, °F	294	(146°C)
Rpm	24	
Retention time, min.	6-1/2	
Temperature of SCU leaving, °F	164-166	(73°-74°C)
Total coatings, % by analysis	14 (avg.)	
Dissolution rates, %		
First week	24 (avg.)	
Second week	30	
Sulfur yield, %	87	
Wax-coating drum		
Temperature of entering coated material, °F	158-163	(70°-72°C)
Temperature of wax and coal tar mixture, °F	223	(106°C)
Rpm	14	
Retention time, min.	1/4	
Wax applied, % of total product	3.2	
Coal tar applied, % of total product	0.27	
Fluidized cooler temperatures		
Material entering, °F	155-159	(70°-72°C)
Cooling air entering, °F	86	(30°C)
Material leaving, °F	87	(31°C)
Air leaving, °F	91	(33°C)
Conditioning drum		
Rpm	12	
Retention time, min.	3.5	
Conditioning agent, % of total product	2.0	

was, and 2% conditioner). Larger forestry grade urea (-5+6 mesh) was used in a few tests. Typical screen analyses for these materials are shown in table 8. In the discussions that follow, commercial spray-drum- or TVA pan-granulated material was used unless otherwise noted.

The sulfur was bright sulfur that was purchased as a tank carload. Carbon content was about 500 ppm and ash content about 55 ppm. Several strainers were required in the sulfur supply system to avoid stoppages in the small orifices of the spray header and in the spray nozzles.

All of the was used was the microcrystalline was Shellmax 800. The coal tar oil when used was fluid enough at ambient conditions to allow easy pumping to the waxing drum. It mixed readily with the was in the small pipe tee provided for this purpose.

The conditioner used was of the diatomaceous earth type. It handled well without dusting despite its fine particle size.

Effect of Process Variables

Effect of coating weight and size and shape of urea.— Data from pilot-plant tests showing the dissolution rates of several products over a range of total coating weights are plotted in figure 27. Separate curves are shown for 7- and 14-day dissolution tests in water at 100°F (38°C). These data show how the dissolution rate increased with decreased coating weight. The total coating weight shown included 3% was, 0.25% coal tar, and 2% diatomaceous earth conditioner.

Table 8. Typical size of various urea products used in pilot-plant work

Commercial spray-drum granulated urea	Tyler, mesh	Commercial air-prilled urea	Tyler, mesh
	+6 + 0		+8 + 3.8
	-6+7 + 11.0		-8+10 + 82.6
	-7+8 + 65.8		-10+12 + 89.5
	-8+9 + 20.5		-12+14 + 3.9
	-10+12 + 0.4		-14 + 0.6
TVA pan-granulated urea (pilot plant)	+6 + 14.0	Forestry size spray-drum-granulated urea	+3 + 0
	-6+7 + 63.7		-3+4 + 25.5
	-7+8 + 21.7		-4+5 + 66.4
	-8+9 + 0.3		-5+6 + 5.4
	-9+10 + 0.1		-6+7 + 0.7
	-10+12 + 0.1		-7+8 + 0.9
	-12 + 0.1		-8 + 1.1

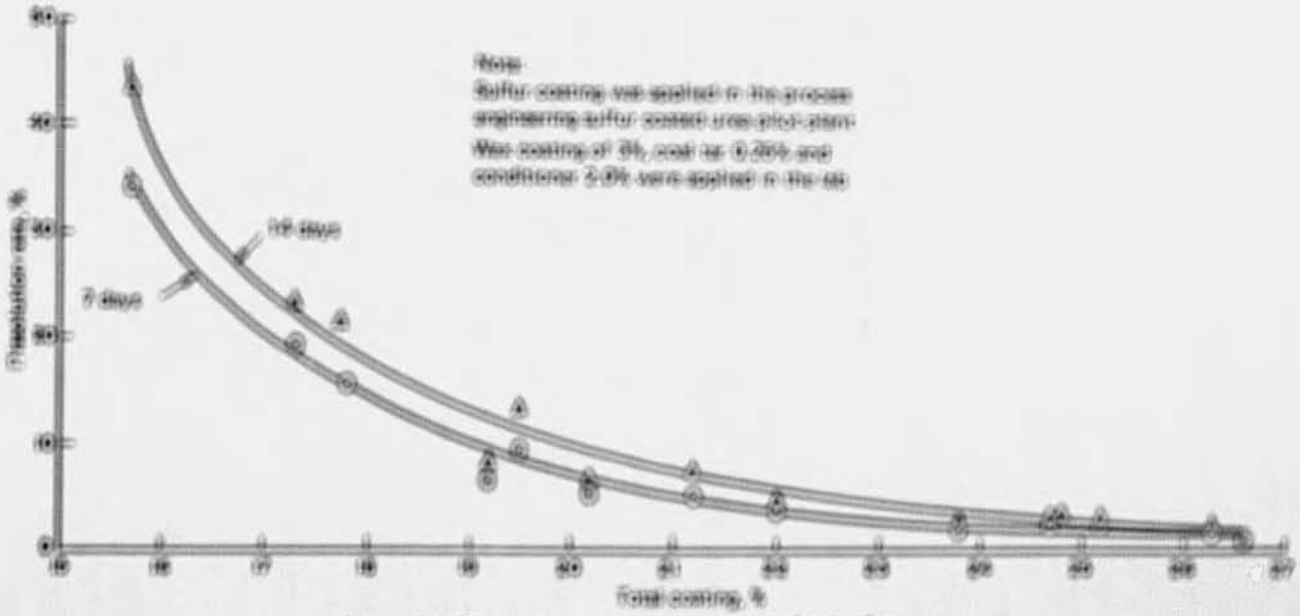


Figure 27. Dissolution rate in water at 100°F (38°C) versus total coating for spray drum-granulated urea (-6+10 mesh)

Data showing effect of total coating weight on 7- and 14-day dissolution rates are plotted in figure 28. Also shown is the effect of use of a sealant (3% wax, 0.25% coal tar) and no sealant. These data gave promise of suitable coating without use of a sealant.

As has been pointed out previously, the size and shape of urea being coated are very important. Less coating material is required for a good coating on large, smooth granules than on small granules with rough surfaces. This is

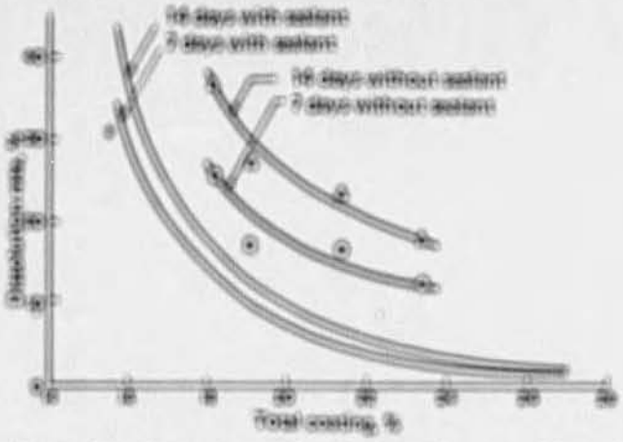


Figure 28. Comparison of sulfur-coated urea with and without wax sealant

shown graphically in figure 29. The large, closely sized spherical urea was coated satisfactorily with considerably less total coating than when the smaller irregularly shaped urea was used. This factor is very important in obtaining best results with a minimum coating weight.

Effect of drum speed.—The speed of rotation of the coating drum had a pronounced effect on quality of the coating. Figure 30 shows the effect of drum speed on the quality with other conditions kept constant. The total coating weight required to obtain a 7-day dissolution of 25% decreased from about 21% at 14 rpm (48% of critical speed) to only about 16.5% at 26-29 rpm (70%-76% of critical speed). The carryover of material in the drum became quite high at a drum speed of 29 rpm (75% of critical speed). This did not affect quality of the coating but did increase crust formation in the drum. Therefore, it is not practical to operate the drum at speeds higher than 65%-68% of critical.

At a drum speed of 26 rpm, dissolution rates averaged 25% in 7 days and 32% in 14 days with a 17% total coating, giving a product with about 39% nitrogen content.

Smoothing of surface of urea granules.—Tests were made to determine whether smoothing of the surface of granular urea by heat-polishing would improve its suitability for sulfur coating. Commercial spray-drum-granulated urea was heat-polished by passing the urea under sprays in the sulfur coating drum with only hot air at

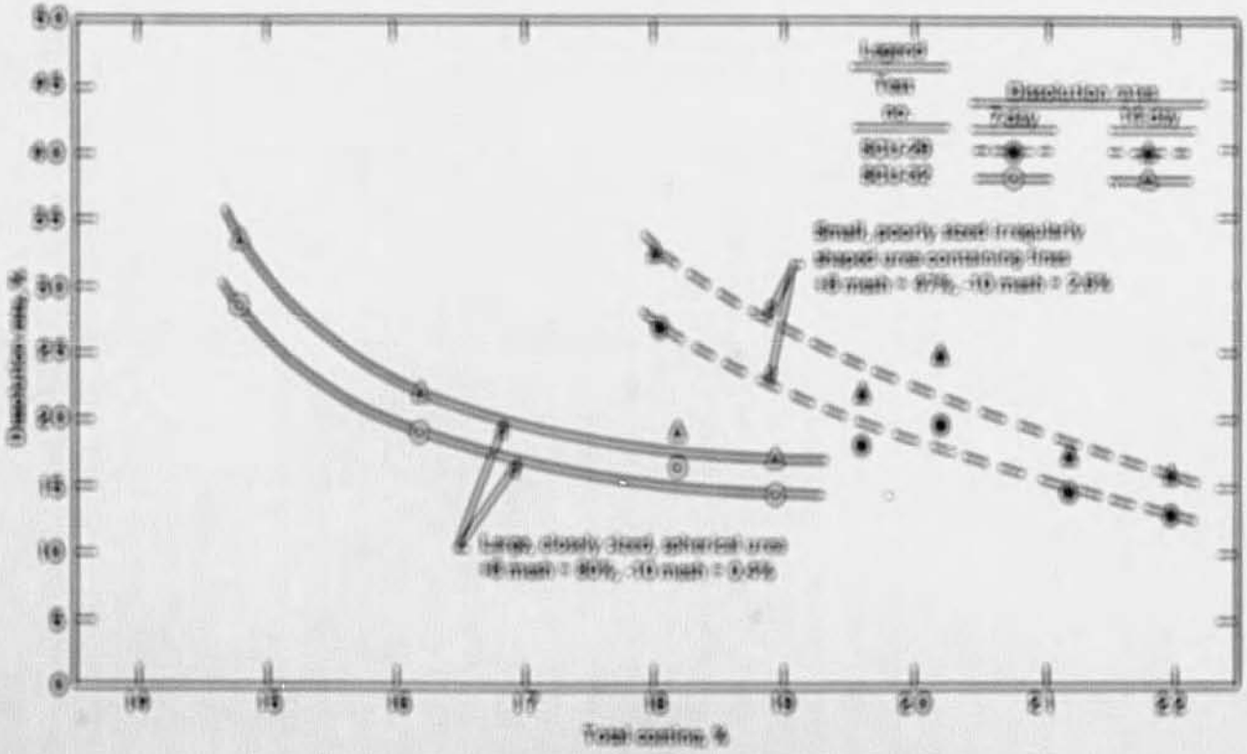


Figure 29. Effect of size and shape of urea on total coating requirements using four spray nozzles and a production rate of one-half ton/hour

300°-310°F (150°-155°C) being used. Observation of the granules after treatment showed that the surface was well polished. The polished urea was then used as feed to the full sulfur-coating process. Comparison of dissolution rate data with previous runs where the urea had not been polished showed no apparent advantage for the pre-polishing. Apparently the surface of the spray-drum-granulated urea was smooth enough, particularly after preheating, to not need any further smoothing.

This procedure might be helpful for granules that have a rough surface.

Temperature of material while coating and waxing — Temperature throughout the system is very important in coating effectively. The temperature of the urea leaving the preheating drum was varied from 100°-140°F (38°-71°C) while operating at a production rate of 0.5 ton/hr. Other conditions of operation were those considered to be best in previous tests. Seal coatings of 3% wax, 0.25% coal tar, and 2% conditioner were applied. Total coating weight was 13%. Figure 31 shows how the 7-day dissolution rate increased from 19% for material preheated to 140°F (71°C) to 37% for material heated to only 100°F (38°C).

The other curve of figure 31 shows the effect of temperature of waxing on dissolution rate. The optimum range was shown to be 153°-158°F (67°-69°C). The rate of dissolution increased rapidly as the temperature of waxing increased above 160°F (70°C). These data confirmed results of tests in the small pilot plant.

In the production of SCU without any seal coating, the temperature of the material leaving the sulfur-coating drum should approach but not exceed 202°F (94°C). Higher temperature appeared to cause the applied sulfur to change to a less desirable crystalline form. The quick cooling provided by the fluidized-bed cooler decreased but did not eliminate the detrimental effects of the high temperature. During the production of SCU with the seal coatings, the best results were obtained when the sulfur-coating temperature was held as high as possible without exceeding the above-mentioned 153°-158°F (67°-70°C) temperature for the product leaving the wax-coating drum. When the temperatures of the sulfur and atomizing air leaving the spray nozzles were between 295° and 310°F (147° and 155°C), the sulfur appeared to atomize well and bond tightly to the urea. This is the temperature range where sulfur has its lowest viscosity.

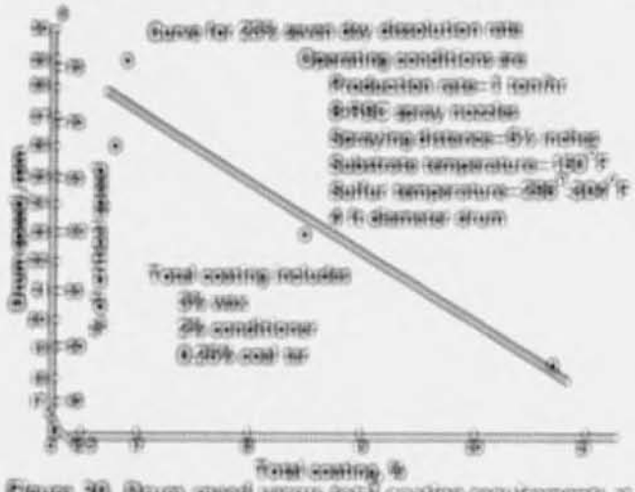


Figure 30. Drum speed versus total coating requirements at a constant sulfur-coated urea dissolution rate

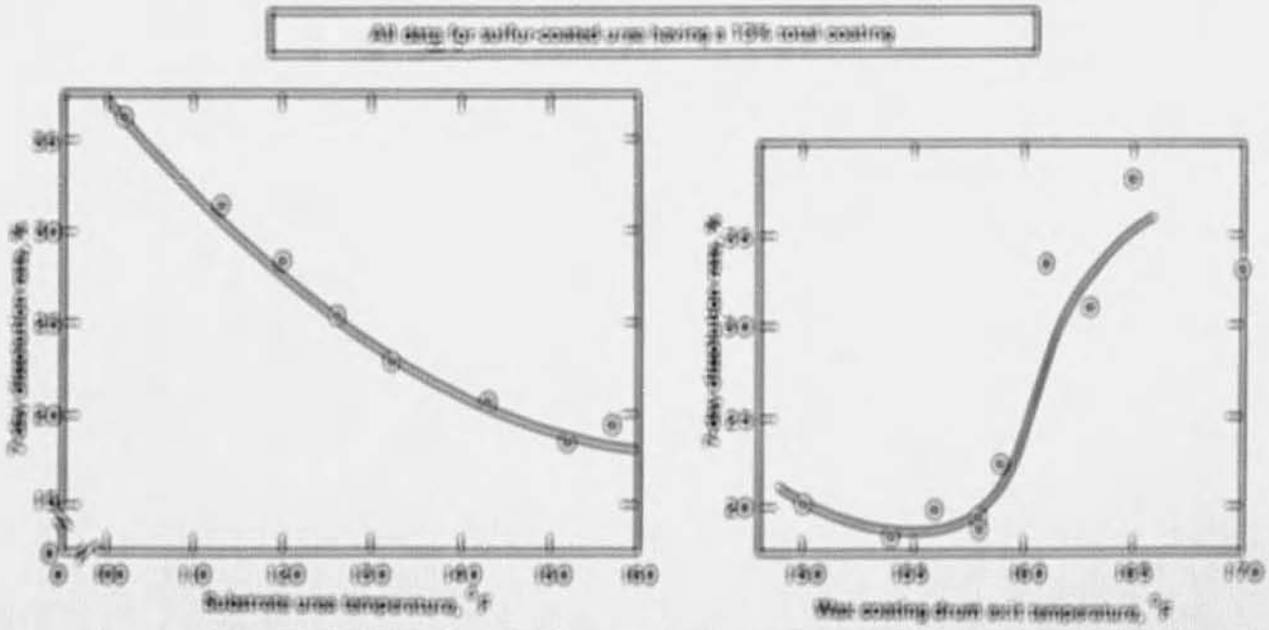


Figure 31. Relation of preheated urea temperature and wax-coating drum exit temperature with dissolution rate of sulfur-coated urea

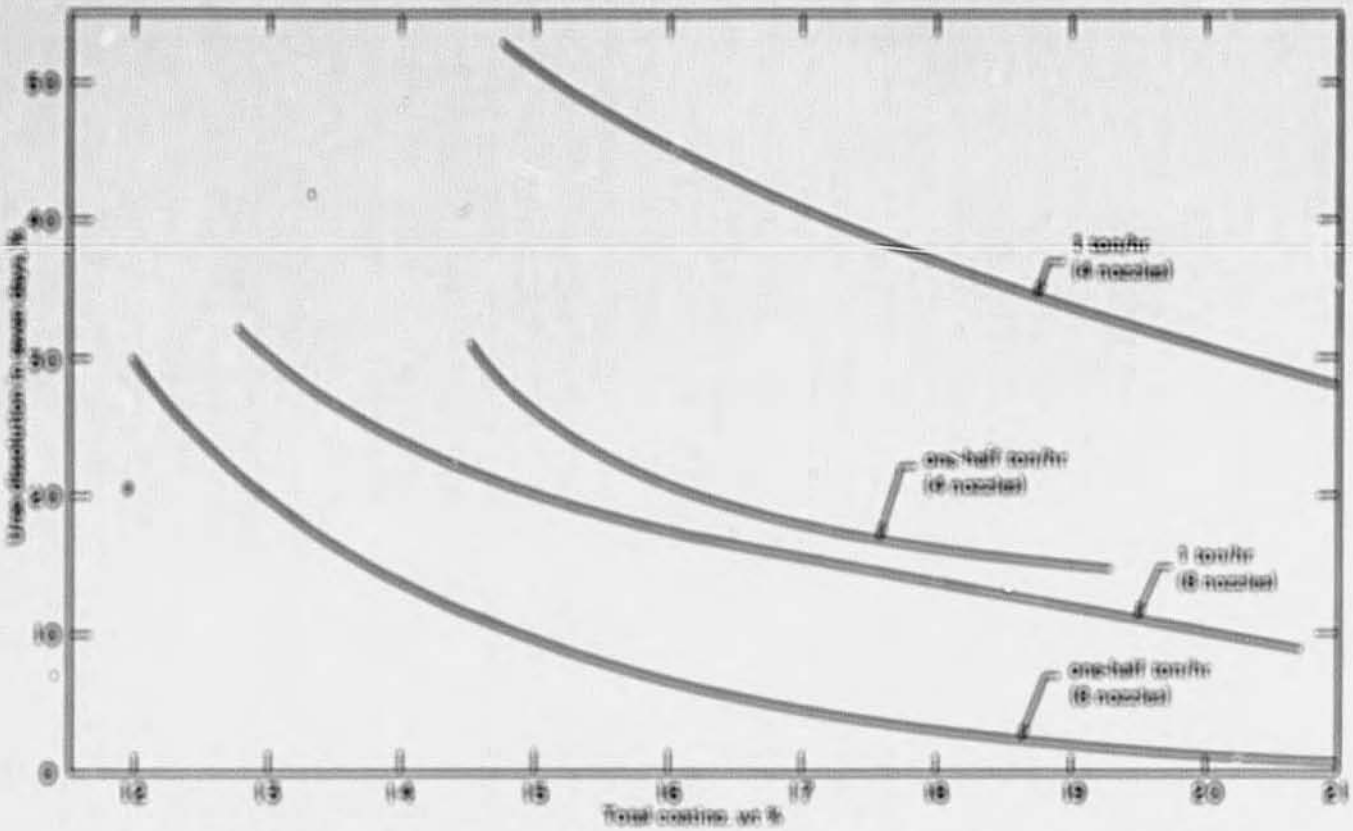


Figure 32. Effect of eight versus four sulfur spray nozzles on the total requirements for sulfur-coated urea at production rates of one-half and one ton/hour and operating under the best known conditions

Effect of production rate and number of spray nozzles used—Tests were made at 0.5 and 1 ton/hr using four and eight air-atomized spray nozzles of the RNC[a] type at each rate. Sulfur flow rates ranged from 10 to 40 lbs/nozzle/hr. As the ratio of nozzles to production rate was increased, the quality of the sulfur coating improved. Further, when the number of nozzles and the production rate were increased proportionally, a better quality coating was applied. These results are shown in figure 32. For example, the dissolution rate in water at 100°F (38°C) for material produced at the rate of 1 ton/hr with eight nozzles was 3% to 5% lower with the same total coating than for the material produced at the rate of 0.5 ton/hr with four nozzles.

A main problem in design of a commercial-size unit would be to arrange for the largest practical number of spray nozzles.

Location of sulfur spray nozzles—Eight sulfur spray nozzles were used in most tests. The tests indicated that best results were obtained when the spray nozzles were 4½-5½ inches (12-14 cm) from the cascading bed of solids and positioned directly over the top end of the elongated eye in the bed. Positioned at this point, the spray nozzles spray directly downward on what appeared to be the fastest moving portion of the bed. Essentially all granules passed under this point on every cycle of material. However, the granules moved in multiple layers so that each granule did not receive a sulfur application on each cycle, but the probability of each particle receiving an equal amount of sulfur coating was maximized. When the sulfur nozzles were moved farther than 5½ inches (14 cm) from the bed, some improvement in coating quality was obtained. However, dusting became heavier in the sulfur-coating drum, thus, increasing the sulfur explosion hazard and increasing sulfur loss in the offgas.

The urea bed depth in the sulfur-coating drum—The bed depth was varied by using various retaining rings in the drum, varying from 2 to 3-3/8 inches (4%-5.5% drum loading), with no appreciable effect.

Volume of atomizing air/nozzle—This was checked between 3 and 5 scfm/nozzle at sulfur flow rates of 15 lbs (6.8 kg)/hr/nozzle while using Spray Engineering RNC nozzles with 0.060-inch-diameter wing tip orifices and 0.040-inch-diameter center tip orifices. (Some TVA modifications were also incorporated in these nozzles.) However, at higher sulfur-to-atomizing air ratios than those tested, the quality of atomization is certain to decrease and this is no doubt one of the reasons for a decrease in product

[a] Ibid.

quality with an increase in production rate with the same number of nozzles.

Wax requirement—Pilot-plant results showed that waxing at a 2% level gave essentially the same dissolution rates as coating with 3% wax, provided there was an additional 1% sulfur added to the total coating. The lower proportion of wax resulted in a less sticky product.

Specifications for the Coatings

Based on the results of a number of tests in the pilot plant of waxed and conditioned products, curves were structured (Figure 33) to show total coating required for specific 7-day dissolution rates. The corresponding nitrogen contents and sulfur contents of the products are also plotted in the upper curves. These curves are based on use of good quality spray-drum-granulated urea, 3% wax, and 2% conditioner. Production rate was 1 ton/hr using eight spray nozzles of the air atomized type. For instance, if a 7-day dissolution of 20% is desired, a total coating weight of about 15% would be needed. Nitrogen content of the product would be 39.5% and sulfur content 9.5%. A product having a 30% dissolution rate in 7 days could be produced with a 13% total coating, resulting in a product having a nitrogen content of 40% and a sulfur content of 8%. SCU without any seal coating required an 18% coating of sulfur to obtain a dissolution rate of 30% in 7 days if produced at 0.5 ton/hr with eight spray nozzles.

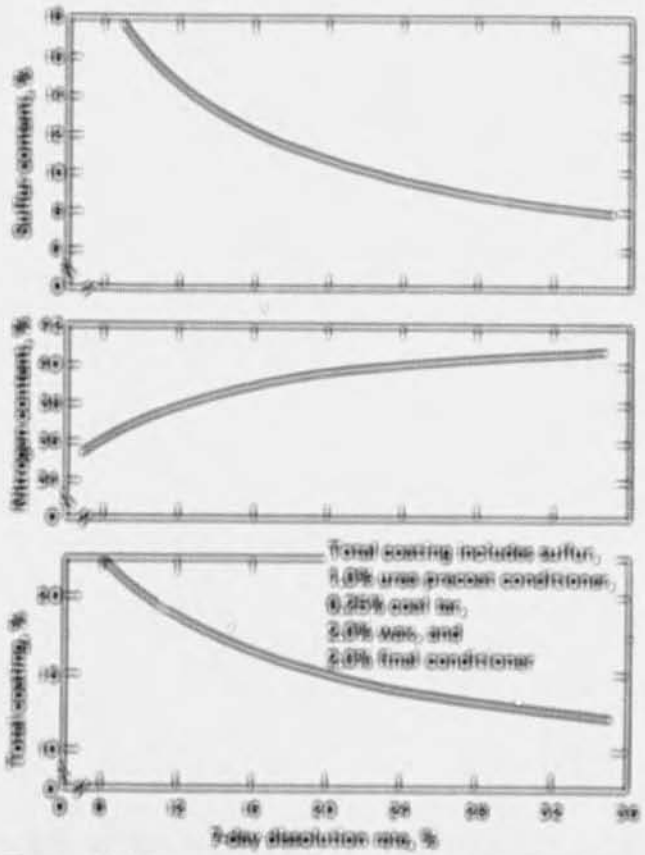


Figure 33. Nitrogen and sulfur content of sulfur-coated urea based on the total coating requirement to obtain a given 7-day dissolution rate with production at 1 ton/hour under the best known conditions using air atomizing nozzles

Use of Dual Spray Headers in the Coating Drum for Production of 2 Tons/Hr

Alterations were made to the pilot plant to allow production rates as high as 2 tons/hr with seal coatings of 2% wax, 0.25% coal tar, and 2% conditioner. A second bank of resistance heating elements was installed in the preheating drum, its retaining ring was lowered, and the speed was increased from 20 to 35 rpm. The fluid bed cooler cooled the 2 tons/hr very effectively. Mechanically, the 2 tons/hr rate was handled effectively by all pilot-plant equipment. However, dissolution results were not very satisfactory. At the 2-ton rate and seven nozzles used on a single header, a total coating of 22% gave a 35% dissolution in 7 days.

A second sulfur spray header with eight nozzles was constructed and installed in the coating drum parallel to the other one. Nozzles on both headers were 8 inches (20 cm) apart, but the nozzles were staggered so that nozzles on the two headers were 4 inches (10 cm) apart lengthwise along the axis of the drum. Both headers were installed so that their orientation relative to the drum and to each other could be easily adjusted for maximum effectiveness of the sprays.

Type RSC nozzles of the pneumatic atomizing type were used on each header. The second header was designed like the first with a 0.018-inch orifice for each spray nozzle to ensure uniform flow.

At a production rate of 2 tons/hr, the dual header gave much better quality than with the single header. However, with two headers at 2 tons/hr, results were not as good as they were for a single header at 1 ton/hr. At a production rate of 1 ton/hr, the two-header system gave results about the same as for a single header. However, the dual header system using pneumatic (air atomizing) nozzles could not be operated for long periods. Extremely heavy sulfur mist occurred, resulting in uncontrollable sulfur buildups on the drum and header. Control of sulfur dust and vapor would be very difficult, and cleaning of inner structures of the drum would be required. A considerably larger amount of agglomerates was formed in the sulfur-coating drum with the two headers. Although these results were discouraging, it was believed that use of hydraulic nozzles (no atomizing air) would give better results with the tandum header. There is much less dust and vapor to contend with.

Urea Coated with Sulfur Only (Pneumatic Spraying)

A few previous results have been given for exploratory tests of the production of a controlled-release product by coating with sulfur only. These results had indicated that prospects might be good for simplifying the process by eliminating equipment for waxing and conditioning of the product. There also was indication that cooling might not be required or that cooling to a lesser degree would be sufficient.

In later pilot plant test work, good results were obtained by eliminating the addition of 2% wax, 0.25% coal tar, and 2% conditioner that were added as seal coatings.

The processing temperature at which the sulfur is applied is the most critical variable in the process when coating with sulfur only. Pilot-plant tests showed that the sulfur should be applied at a temperature close to but not exceeding 204°F (96°C). In the pilot plant, the temperature of material leaving the sulfur coating was controlled at 195°-200°F (91°-94°C).

Substantially better results were obtained when coating with sulfur only (using pneumatic nozzles) if the production rate was decreased to 0.5 ton/hr instead of the usual 1 ton/hr. A product with 21% coating of sulfur gave a dissolution rate of 30% in 7 days when produced at the 0.5-ton rate. When produced at 1 ton/hr, a 28% coating was required to give the same dissolution rate. In general, a sulfur coating about 50% higher than for waxed product was required. However, the total coating was increased by only 4½ percentage points.

Quickly solidifying sulfur from temperatures above 318°F (160°C) greatly increases the probability of formation of the desired amorphous sulfur. However, spraying sulfur at temperatures higher than 318°F (160°C) is very difficult because of the sulfur's high viscosity at these temperatures. Best results were obtained when the sulfur was sprayed at temperatures of 275°-318°F (135°-160°C).

Figure 34 shows how increased temperature up to about 195°F (90°C) was beneficial. The 7-day dissolution rate with material heated to 178°F (80°C) was 40%; with urea preheated to 193°F (90°C), the dissolution rate decreased

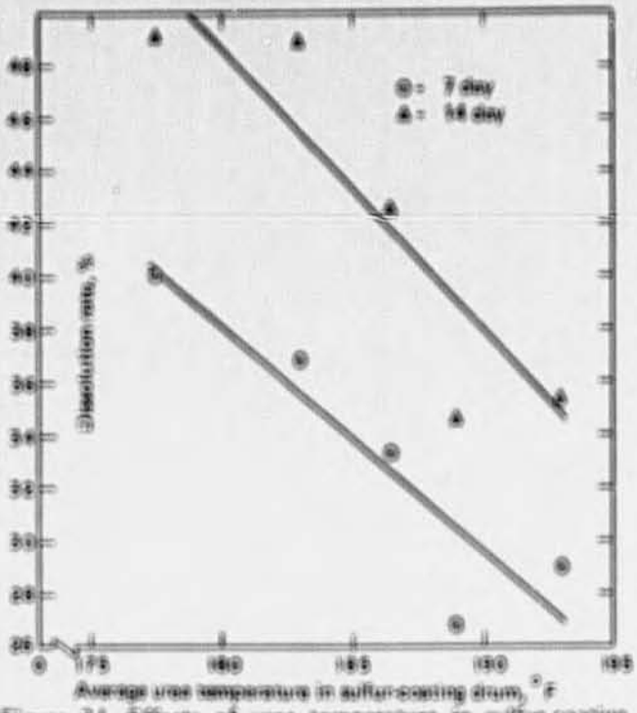


Figure 34. Effects of urea temperature in sulfur-coating drum

to about 28%. The 14-day dissolution results showed the same pattern.

Aside from the advantages of process simplification when only sulfur is used as the coating, the product has better storage and handling properties since stickiness of the wax is eliminated. Indications are that the proper type of wax will be difficult or impossible to obtain in the quantity required for a commercial-scale unit, so it may be necessary to eliminate the use of wax. Agronomic tests of urea coated with sulfur only are under way.

Discontinuation of Use of Microbicide

As results from additional agronomic tests were obtained, it became apparent that a microbicide was not required in the seal coating. Therefore, the microbicide was omitted in the latest test work.

USE OF HYDRAULIC SPRAYING SYSTEM

After tests in the small pilot-plant had shown much promise, a high-pressure, hydraulic spraying system was installed in the large pilot plant to replace the system using pneumatic nozzles.

The sulfur flowed from a weigh tank through a 400-mesh screen to remove impurities in the sulfur that would plug the very small orifices in the hydraulic spray nozzles. The sulfur then flowed through a 2-micron filter for further cleaning. Particular care in removing solids is required when using the pneumatic system because of the very small openings in the spray nozzles. The sulfur was pumped by a double

acting piston pump[9] that was pneumatically driven and was capable of developing pressure up to 2,800 psig at rates as high as 1 gpm. A pulse eliminator tank was located downstream from the sulfur pump. Air trapped in the top of this tank provided a constant pressure on the sulfur spray nozzles, and this maintained a constant flow during changes in direction of stroke of the pump. Flow rate of sulfur was regulated by combinations of the operating pressure and size and number of spray nozzles installed on the spray header. A sketch of a hydraulic spray nozzle assembly is shown in figure 35.

The spray header for the hydraulic system was much simpler than that used with the pneumatic system. No flow control orifices were needed. The header was further simplified, since compressed air supply and its heating were not required. The hydraulic nozzles tested had nozzle tip openings equivalent to orifices of 0.011 and 0.013 inch in diameter. Pressures ranged from 450 to 2,800 psig. Best operation with nozzles of the above size was at 750 to 1,500 psig.

From the standpoint of operation, the hydraulic system had several advantages over the pneumatic system. Sulfur mist and dust formation in the drum were greatly decreased. Dust loadings for the pneumatic system ranged from 0.07 to 0.16 gram/m^3 of air/lb of sulfur sprayed. With the hydraulic system, these values were only 0.003 to 0.008. This greatly simplifies the cleanup of air discharged from the drum and essentially eliminates the potential hazard of explosions (due to air-sulfur explosive mixture) in the sulfur-coating drum. The minimum explosible concentration for sulfur is 35 grams/m^3 in air. Heaviest concentration in the pilot plant with hydraulic nozzles was only 3.2 grams/m^3 of air—well below the explosible range. This is extremely important in design and operation of a commercial-scale unit. Buildup of solids on the drum and header are essentially eliminated and the amount of agglomerates is decreased. The process temperatures for effective sulfur coating are not so critical as with the pneumatic spray system.

In initial tests using a single header with eight nozzles, an increase in the amount of sulfur applied was required for effective coating as compared with experience with air atomized nozzles. A 22% total coating gave a product with dissolution rate of 35% in 7 days when the production rate was 1 ton/hr. This represents an increase of 4 to 5 percentage points in the sulfur coating over that required when using pneumatic nozzles. In later tests using a different type of spray nozzles (T250017-SS Uni Jet)[10] and with additional operating experience, much better results were obtained. At a production rate of 0.75 ton/hr, a total coating of 24% gave a 7-day dissolution rate of only

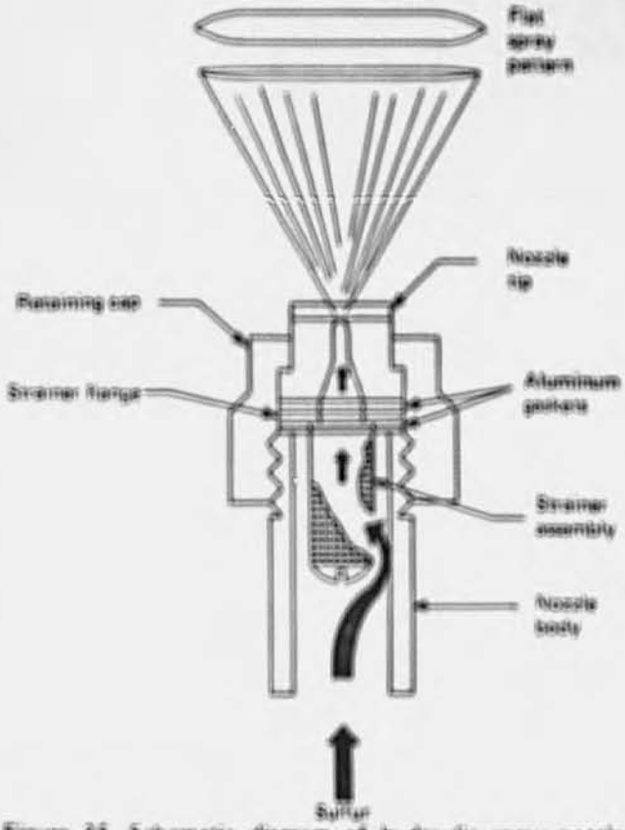


Figure 35. Schematic diagram of hydraulic spray nozzle assembly

7%. At a 1-ton/hr rate under the same conditions, a total coating of 22% gave a 7-day dissolution rate of 16%. At a production rate of 1.5 tons/hr, a 7-day dissolution rate of 31% was obtained with total coating weight of 21%.

Temperatures throughout the pilot-plant system for use of pneumatic and hydraulic spray systems in preparation of sulfur, wax, and conditioner coatings are shown in the flow diagram of figure 36.

The somewhat simplified pilot-plant system and heat temperatures throughout the process using pneumatic and hydraulic nozzles for preparation of coatings of sulfur only are shown in figure 37. The heat temperatures of preheating and of material in the sulfur-coating drum are considerably lower when using the hydraulic spray system.

A further improvement in performance of the hydraulic spraying system due to use of a flight system in the sulfur-coating drum is described in the next section.

Tests of a Flight System With Falling Curtain in the Coating Drum

The sulfur-coating drum in the large pilot plant was modified in a manner similar to the flights and cascade arrangement that gave very good results in the small unit. The greater amount of room in the larger drum allowed

[9] Graco "Bulldog" pump.
[10] Spraying Systems Company, Bellwood, Illinois.

more flexibility in the arrangement. Twenty-four flights were installed at 15-degree intervals parallel to the axis of the drum. The flights are 3 inches (7.5 cm) in depth and are canted 15 degrees forward to increase their lifting capacity. The retaining ring in the drum was increased to a depth of 5 inches (13 cm). As the drum rotates the granules discharge from the flights onto a sloped collection or deflection pan in the upper part of the drum. The pan is 20½ inches (52 cm) wide and extends essentially the full length of the drum. It is adjustable in slope and in relative position in the drum. The granules cascade down the collection pan, and as they discharge from it they strike a vertical deflector plate. This arrangement causes the granules to form a thin vertical curtain of fast moving granules. Spraying of sulfur into this fast moving curtain of granules is the key to this system. A sketch of this arrangement is shown in figure 38.

Operation of the modified sulfur-coating drum has been very good. The slope of the collection pan was adjusted to 35 degrees. With this slope and a drum rotational speed of 13 rpm, a curtain 17 inches (43 cm) in height and 3/8- to ½-inch (1- to 1.2-cm) thick was formed. At this drum speed the flights discharged completely as they passed over the collection pan. Eight spray nozzles of the hydraulic type were used. The spray header was adjustable to allow spraying into the curtain at right angles to it, or onto the bed at the point where the curtain joins it. In the first tests the sprays were located 5 inches (13 cm) from the curtain and were directed into it at right angles, and at a point about 2 inches (5 cm) above the junction of the curtain and the bed (position A of figure 37).

Observation of the interior of the drum showed that a fast moving, very active curtain of granules was formed. Samples taken from the discharge of the drum indicated very uniform coating. Results of tests with the sprays directed into the curtain (position A) were better than when directed onto the junction of the curtain and the bed (position B).

Tests with full coating of sulfur, wax, and conditioner — Spray-drum-granulated urea was used. In the initial tests, the full coating of sulfur, wax, and conditioner was applied. With the spray nozzles set for position "A," a total coating of 20% gave a 7-day dissolution rate in water of 15% at a production rate of 1 ton/hr. With essentially the same conditions except that the spray nozzles were directed to position "B," a total coating of 20% gave a 7-day dissolution rate of 27%. In figure 39 the results with these two positions are compared and these are also compared with previous results using hydraulic and pneumatic nozzles before the internal changes were made in the drum.

Of particular interest in these tests is the fact that better results were obtained with the hydraulic nozzles than in previous tests using pneumatic nozzles. This was the first time that better results were obtained in the large pilot plant with hydraulic nozzles under any conditions.

Before the flight system was installed in the coating drum, high rotational speeds were required to provide an

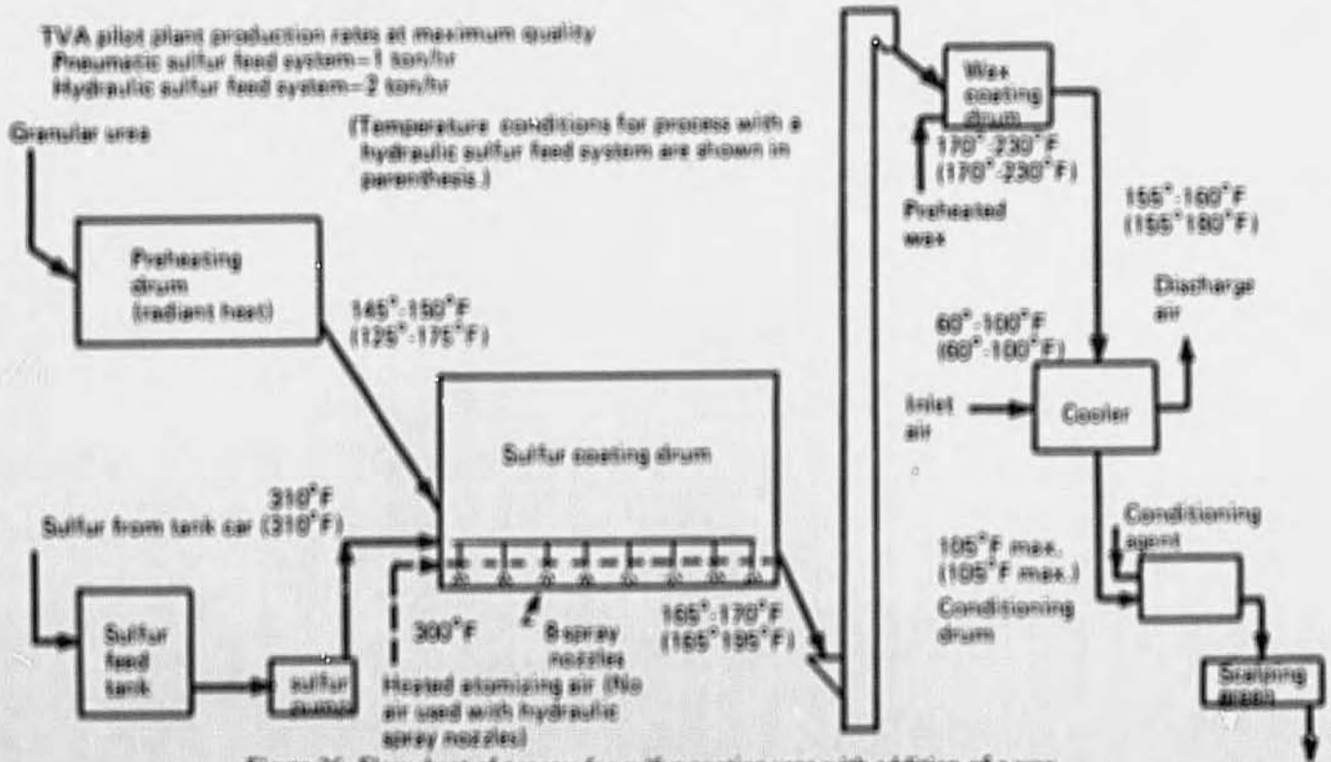


Figure 36. Flow sheet of process for sulfur coating urea with addition of a wax sealant using either pneumatic or hydraulic sulfur spraying system

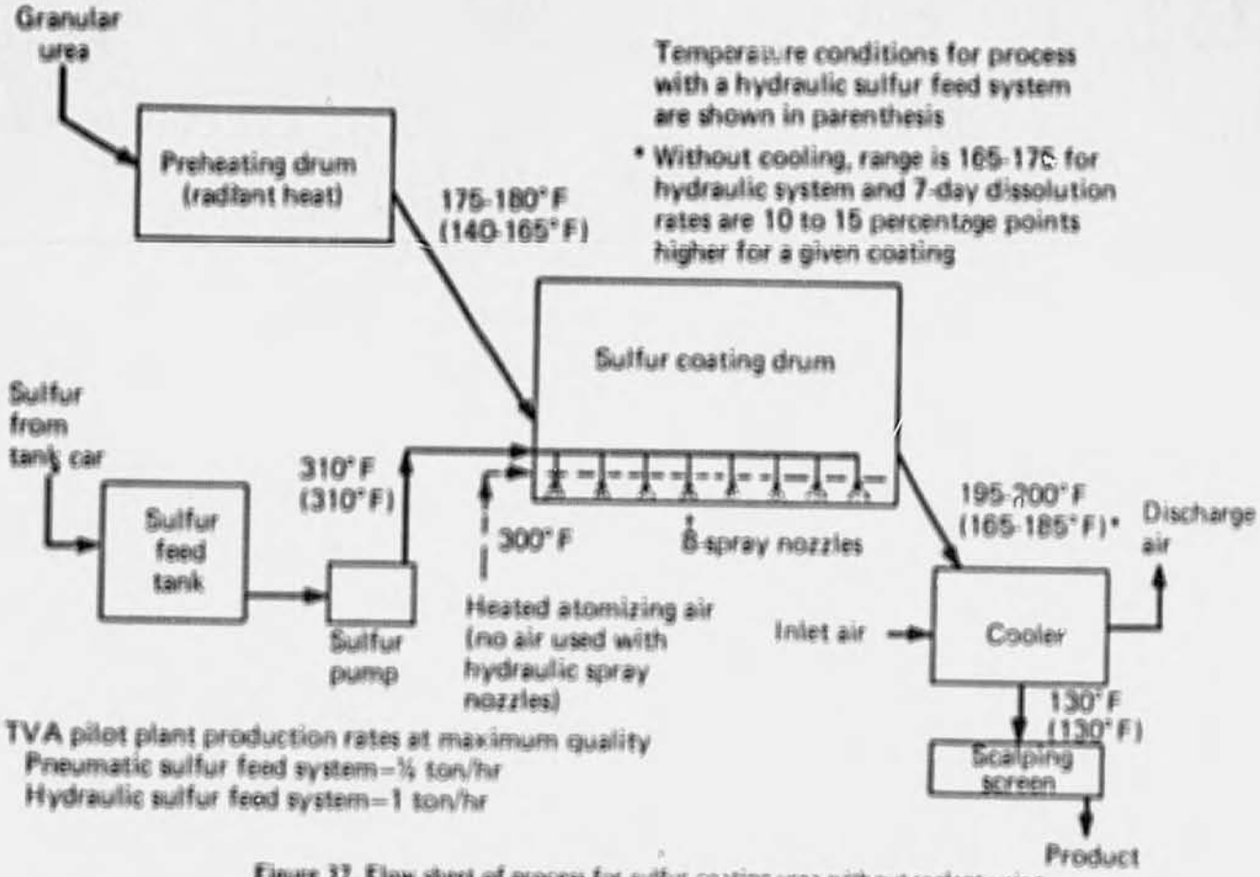


Figure 37. Flow sheet of process for sulfur coating urea without sealant using either the pneumatic or hydraulic sulfur spraying system

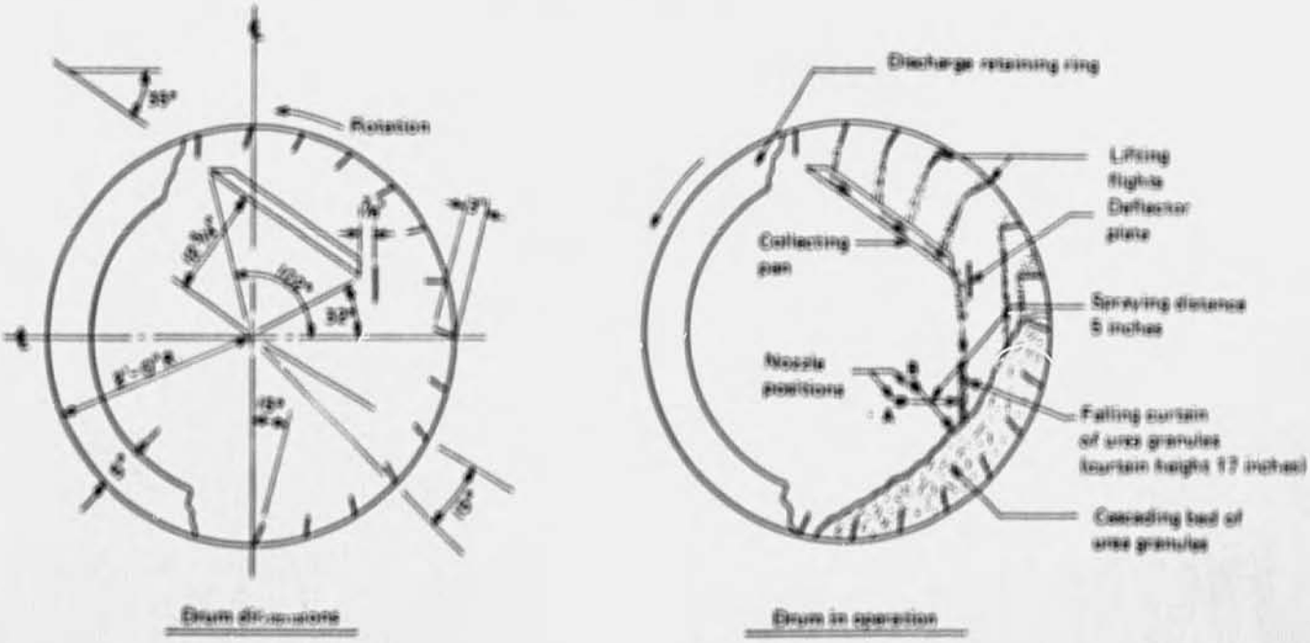


Figure 38. The sulfur-coating drum with changes to provide falling curtain

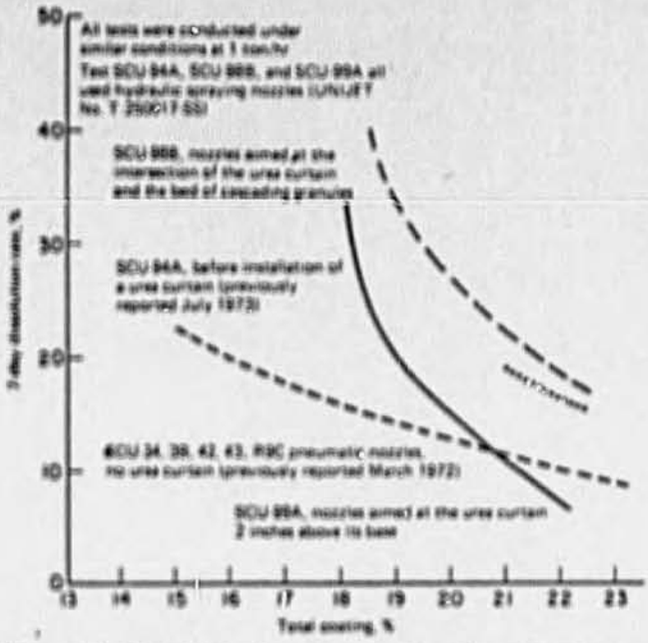


Figure 39. The effect of a urea curtain and nozzle orientation on the quality of sulfur-coated urea made using hydraulic atomizing nozzles

active bed for spraying with sulfur. With speeds below 21 rpm (55% of critical speed) there was segregation of the bed by particle size. Therefore, speeds of 22 to 26 rpm (55% to 65% of critical speed) were used. The flights and curtain arrangement work well at low rotational speeds; the granules in the falling curtain do not appear to be segregated by granule size. When the sulfur was sprayed directly into the falling curtain, a change from 13 rpm to 18 rpm did not give any improvement. At a production rate of 1 ton/hr, a total coating of 20% gave a 7-day dissolution of 15% at both drum speeds. When the sulfur was sprayed onto the junction of the curtain and rolling bed, better results were obtained at 18 rpm than at 13 rpm.

These tests showed definitely that spraying into the falling curtain at right angles was the best arrangement.

Effective sulfur coating in the drum with the flights and curtain system using hydraulic nozzles is not affected as much by increased rate of production as with the plain drum and pneumatic nozzles. Tests were made at 1 and 1½ tons/hr in which eight nozzles of the hydraulic type (Uni Jet No. T-250017 SS)[11] were used. There was no significant difference between the 1 and 1½ tons/hr rates. The 7-day dissolution rates were 24% and 26% respectively with total coating weight of 20%.

Tests were made at a still higher rate of production of 2 tons/hr using the same type of hydraulic spray nozzles spraying into the curtain. The results were very good. A total coating of 19% (14% sulfur) gave a 7-day dissolution rate in water of 24%. Complete data for this test are given in table 9. In another test at 2 tons/hr with 19% total coating (14% sulfur) the dissolution rate was only 18% in 7 days. These are by far the best results ever obtained at a production rate of 2 tons/hr.

The hydraulic (pressure type) nozzles inherently are not subject to less efficient spraying at higher rates. In fact, better atomization of the sulfur would be expected as the sulfur rate is increased through a given sized nozzle. Combination of this factor with the effective curtain arrangement should greatly facilitate design of a large scale unit.

Tests with coating of sulfur only--It is possible that wax of the type used successfully in pilot plant work will not be available for purchase in sufficient quantities for use in commercial sized sulfur coating units. Therefore, much more emphasis in the latest work was placed on coating with sulfur only.

In early work before the flights and falling curtain arrangement was installed in the coating drum, considerably more sulfur was required to give satisfactory controlled release properties for products with unwaxed coating. The heavier coatings appeared to give good early agronomic response but not in later periods. This was believed to be due to the thicker sulfur coatings that were too resistant to give good results in the latter part of the growing season.

Because of the good experience in coating in the modified coating drum, tests were made in preparation of products with a coating of sulfur only. It was hoped that the flights and falling curtain would allow effective coating at considerably lower coating weights. The eight sprays of sulfur were directed into the falling curtain. At a production rate of 1 ton/hr a 20% coating of sulfur gave a dissolution rate in water of 24% in 7 days. Complete data for this test are given in table 9. Tests were also made at higher production rates of 2 and 4 tons/hr. Prior to the test at 4 tons/hr, a second header was installed in the drum to also spray onto the curtain. Data are shown in the tabulation below.

Production rate, tons/hr	Sulfur coating, %	Dissolution rate in 7 days in water at 100°F (38°C), %
1	20	24
2	22.5	30
4[a]	25	30

[a]Two spray headers with eight nozzles each were used.

Use of a Different Type of Sealant

In the latter stages of pilot plant work it was found that the microcrystalline wax such as Shellmax 800 used in most of the work likely would not be available for commercial use. Tests of other potential sealants showed that a 80/20 or 70/30 mixture of bright stock oil and molten

[11]*Ibid.*

polyethylene gave reasonably good results. This mixture was more difficult to apply and required a temperature of about 195°F (90°C) of the SCU being sealed. This required about 30° to 35° higher temperature of preheating.

Table 9. Operating data for sulfur-coated urea pilot plant

	Sulfur, wax, and conditioner coating	Sulfur-only coating
Test No.	SCU-102B	SCU-103
Production rate, tons/hr	2	1
Substrate type, spray-drum-granulated urea with 1% clay conditioner applied by the supplier		
Screen analysis, mesh %	+ 6 = 0.0	(Approx. same)
	-6 + 7 = 9.2	
	-7 + 8 = 64.9	
	-8 + 9 = 23.5	
	-9 + 10 = 1.6	
	-10 + 12 = 0.4	
	-12 = 0.4	
Feed rate, lbs/hr	3,240 (1,480 kg/hr)	1,620 (740 kg/hr)
Preheater		
Substrate exit temp., °F	145 (63°C)	149 (60°C)
Sulfur-coating drum		
Sulfur feed rate, lbs/hr		
Per nozzle	68 (31 kg/hr)	50 (23 kg/hr)
Total	544 (250 kg/hr)	400 (182 kg/hr)
Sulfur feed temp., °F	311 (155°C)	311 (155°C)
Spray nozzles	Spraying Systems Co.	Spraying Systems Co.
Model	T-650025	T-250017
Number	8	8
Distance to curtain, inches	5½ (14 cm)	5½ (14 cm)
Spray tip opening	0.013	0.011
Hydraulic pressure at tip, psig	780	870
Rpm	13	13
Retention time, min.	7.1	12.6
Retaining ring height, inches	5 (13 cm)	5 (13 cm)
Temp. of SCU leaving, °F	162 (72°C)	167 (75°C)
Total coating, %[a]	19	21
Sulfur coating, %	14	20
7-day dissolution rate, %	24	25
Wax-coating drum		
Temp. of entering coated material, °F	155 (69°C)	–
Temp. of wax and coal tar mixture, °F	218 (104°C)	–
Wax applied, % of total product	2.0	None
Fluidized cooler		
Temp. of material entering, °F	155 (69°C)	Material cooled
Temp. of cooling air entering, °F	107 (42°C)	over a three
Temp. of material leaving, °F	112 (45°C)	day period in
Temp. of air leaving, °F	120 (50°C)	1,000 lb piles
Conditioning drum		
Retention time, min.	1.5	–
Conditioning agent, % of total product	2.0	–

[a]Includes precoat of conditioner.

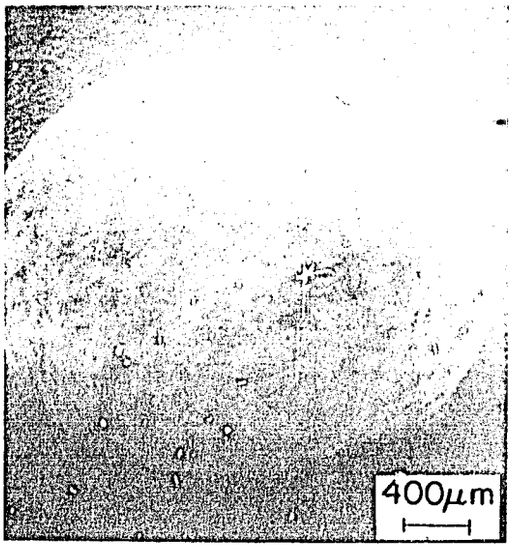
LABORATORY MICROSCOPY STUDIES TO EVALUATE SULFUR COATINGS

Laboratory studies were made using the scanning electron microscope (SEM) with supplemental evaluations by optical microscopy and x-ray diffraction techniques to evaluate sulfur coatings on products prepared in the pilot plants. These studies have been very helpful in studying details of the coatings applied and have helped in the selection of operating conditions to obtain the desired type of coating.

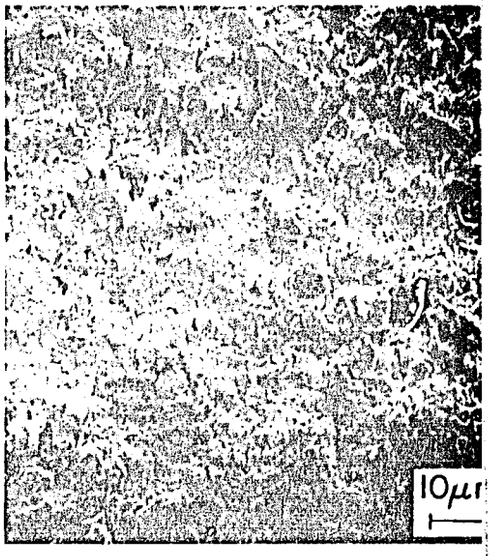
Figure 40 is a series of SEM photographs of urea granules coated under different conditions and with different materials. "A" is an enlargement of a waxed and conditioned SCU granule. The surface of the granule appears to be comparatively smooth. "B" shows surface detail of conditioner on a waxed SCU product.

"C" shows the urea-sulfur interface on an unconditioned urea granule. "D" shows a typical thin spot in the sulfur coating on a granule of urea. "E" shows wax and conditioner filling a thin spot in the sulfur coating. This

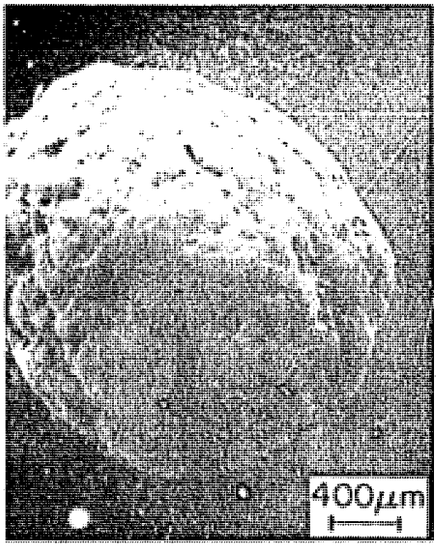
Figure 40. Photographs made with scanning electron microscope show granules of urea coated under different conditions and with different materials.



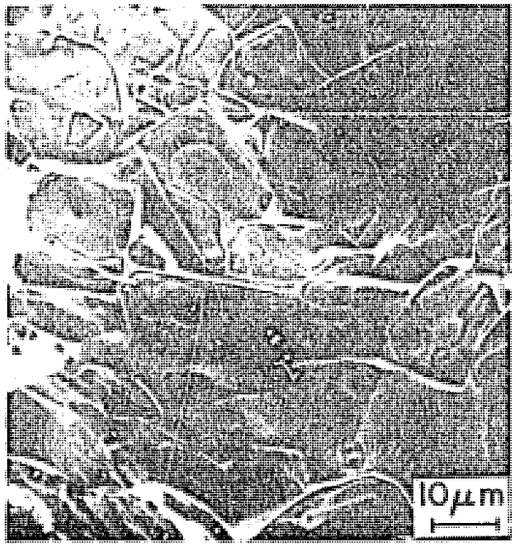
A—Waxed and conditioned sulfur-coated urea granule



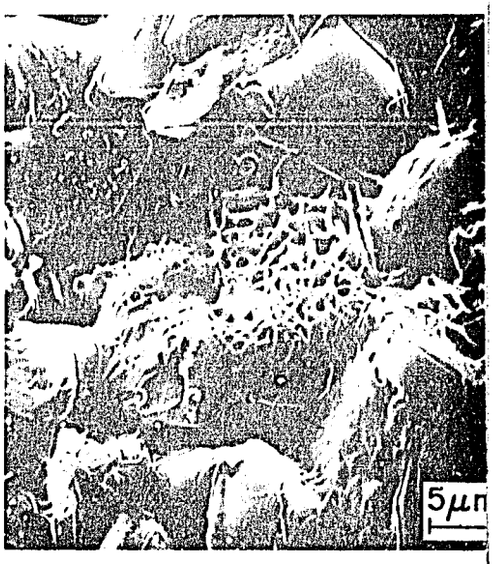
B—Surface detail of conditioner on a waxed SCU product



F—Sulfur coating on a sulfur-coated urea granule



G—Mosaic surface texture of sulfur allotropes



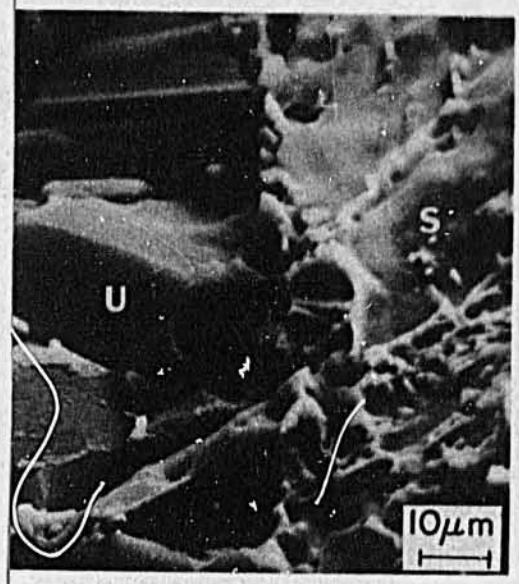
H—Poorly developed mosaic texture and intergranular pores on the exterior surface of the coating

illustrates how wax seals imperfections in the sulfur coating.

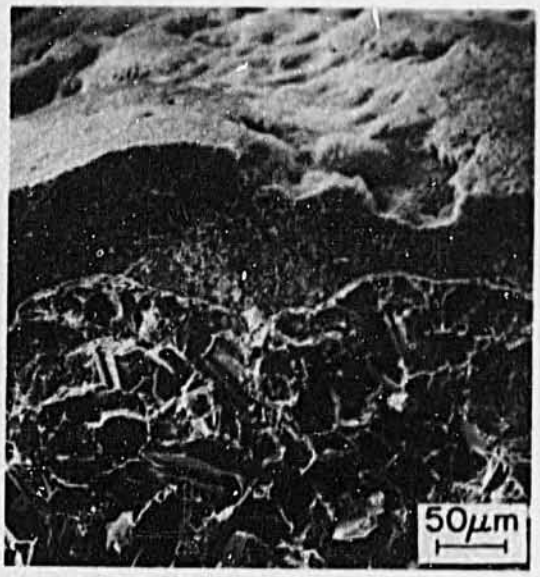
"F" shows a granule coated with sulfur only. The detail in "G" shows the desirable mosaic texture of a sulfur coating. This type texture is best, particularly for coatings of sulfur only. Temperature of the sulfur being applied and temperature of the substrate are important in obtaining this type of coating. "H" illustrates a poorly developed mosaic texture with intergranular pores on the exterior surface of the coating of sulfur.

These studies have also been helpful in illustrating the mechanism of release of urea from the coated granules. To observe the release of urea from SCU products, a number of granules from different samples were soaked in water for 10 minutes without agitation, filtered, washed with alcohol, and air dried overnight. They were then examined microscopically to observe the release of urea. "I" shows release of urea from a pinhole in a coating of sulfur only. "J" shows release of urea from cracks in an improperly cooled sulfur coating.

These studies have allowed a better understanding of the coating process and assisted in selection of operating conditions to obtain the desired quality of coatings. (See reference 4 for additional information.)

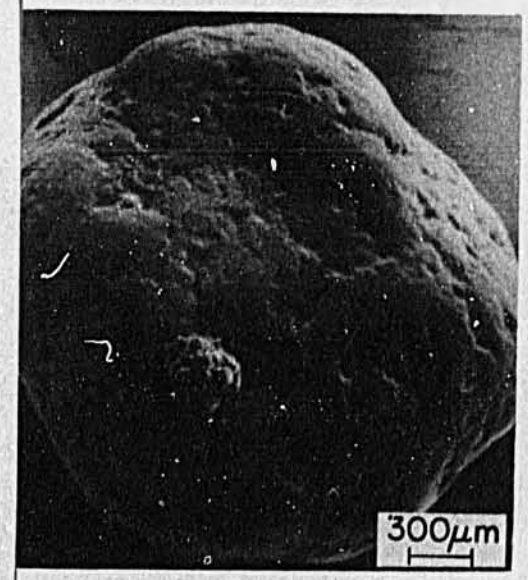


C—Urea-sulfur interface on unconditioned urea substrate (u = urea, s = sulfur)

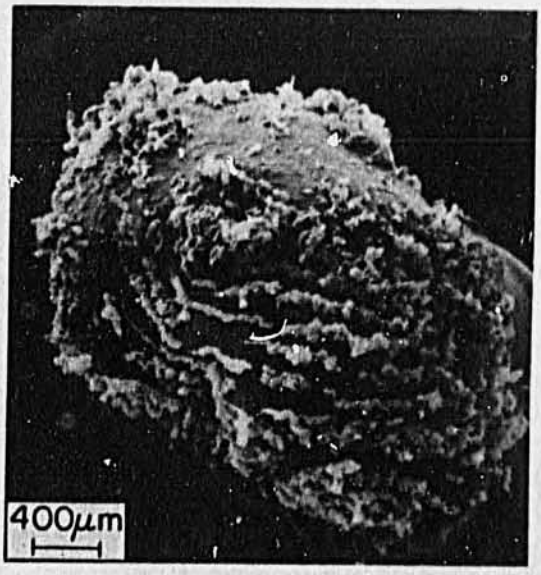


D—Thin spot in a typical sulfur coating

E—Wax and conditioner filling thin spot in sulfur coating (c = conditioner, w = wax, s = sulfur, u = urea)



I—Release of urea from a pinhole in a sulfur coating



J—Release of urea from cracks in improperly cooled sulfur coating

PHYSICAL PROPERTIES OF PRODUCTS

Two properties of the product that have a practical effect on its acceptability are its storage characteristics and its resistance to degradation by normal handling and distribution. Typical products were tested to evaluate these properties

Storage tests—Bagged storage tests were made covering periods up to 3 months. The material tested (coated, waxed and conditioned spray-drum-granulated urea) was stored in plastic-lined bags under a stack pressure of 3.5 psi (equivalent to that of the bottom bag in a stack of 20 bags). The tests were made at ambient temperatures. At the end of the period, there was a light bag set but no caking or lumping. However, dissolution rate increased in this period. Data are shown in table 10. One set of data is for a lightly coated product and the other for a heavily coated one.

The 14-day dissolution rate of the heavily coated material (Test 1) increased about twofold, from 2.6% to 5.4% in 3 months. This is a large percentage increase but hardly significant in terms of practical coating effectiveness. However, the 3-month increases in 14-day dissolution rates for the lightly coated product were significant from a practical standpoint; the rate increased from 33% to 48%. One-month increases in 14-day dissolution losses were considerably lower than the 3-month increases.

It should be emphasized that these results are the maximum that can be expected (bag at bottom of a 20-bag stack) and that average conditions would be less severe.

Additional tests were made to study the physical properties of SCU. Bagged storage tests were extended for 6 and 9 months. All products tested were made from commercial spray-drum-granulated urea (-6+10 mesh). Total coating weight including wax, coal tar, and conditioner was 18.5% to 20.5%. Storage pressure was from 0 to that equivalent to the bottom bag in a pile of 50-lb bags 20 bags high. No caking whatsoever occurred through the stack of bags in the 6- and 9-month storage periods.

Dissolution rates (7 days) did increase for products tested after storage for 6 and 9 months. The rate increased from 4.5% to 9.5% in 9 months under pressure; under no pressure the dissolution rate increased from 4.5% to 7.5%.

Conclusions from these tests are that the SCU products should withstand normal bagged storage quite well.

The coated product (-6+10 mesh spray-drum-granulated urea) was also tested to determine the effects of exposure to humid conditions as compared with similarly exposed uncoated urea. The total coatings ranged in weight from 16% to 21%, with dissolution in water at 100°F (38°C) of 4% to 21% of of the substrate in 7 days. The laboratory exposure tests were made at 100°F (38°C) and 90% relative humidity in a chamber with air circulated by a fan. The test fertilizers were exposed in open-top glass cylinders 7 cm in diameter by 20 cm deep. Moisture absorption was measured after exposure periods of 24 and 48 hours.

Moisture absorption of the coated materials was only 5% to 12% of that of similarly exposed uncoated urea. Moisture penetration was only about 25% of that of uncoated urea. All of the coated materials were free flowing, whereas the uncoated urea was hard caked. The protection against moisture absorption from the air would be a great advantage in handling and application of SCU.

Handling and application tests—Handling tests were made also of sulfur, wax, and conditioner coated products. Coated spray-drum urea granules were: (1) tumbled in a rotary drum for varying time periods, (2) dropped 35 feet onto a steel plate, or (3) fed several times through rotary plate or auger drill distributors, and (4) fed through a spinning plate-type broadcast spreader. The dissolution rates before and after treatment were measures of the effects. Results are given in table 11.

Tumbling for 5 or 12 minutes actually improved the coatings slightly. Dropping the material 35 feet onto a steel plate increased dissolution only slightly. The plate and screw drills caused measurable but not serious breakage of the coatings.

Spinner plate broadcast spreaders turn at high speed compared with a plate drill. Some are impact types in which the fertilizer flows directly into the path of rotating vertical blades which impact on the fertilizer granules to spread them. In another type, however, the fertilizer feeds into the center of the plate, where the speed is much less

Table 10. Change in dissolution rate of various sulfur-coated fertilizers during 3 months' bag storage

Test		Total	Substrate dissolution in 100°F water, wt %											
			As made			1-month storage[b]			3-month storage[b]			Difference in as made and 3 months		
No.	Coating	coating,[a] wt %	7 days	14 days	Daily	7 days	14 days	Daily	7 days	14 days	Daily	7 days	14 days	Daily
			Regular spray-drum-granulated urea (-6+10 mesh)											
1	Heavy	29.6	2.1	2.6	0.07	3.0	4.4	0.20	4.7	5.4	0.10	2.6	2.8	0.03
2	Light	16.1	27.5	32.7	0.74	33.8	41.4	1.09	40.0	47.6	1.09	12.5	14.9	0.35

[a]Coating includes 3% wax sealant, 0.25% coal tar microbicide, 2% conditioner, and sulfur.
[b]Three-pound bag subjected to 3.5 psi pressure.

than at the periphery; therefore, the impact is much less. In this type of spreader, the 7-day dissolution rate increased only 1 or 2 percentage points, even at maximum speed of 1,000 rpm.

Table 11. Effect of handling sulfur coated urea[a] on its dissolution rate

Type	Degree	Total coating, wt %	Substrate dissolution in water at 100°F, wt %		
			7 days	14 days	Daily
Simulated field application by drilling					
Control		25.3	3.6	4.4	0.1
Plate drill	1 pass	25.3	5.7	6.4	0.1
Plate drill	2 passes	25.3	6.4	7.4	0.1
Screw drill	1 pass	25.3	5.2	6.0	0.1
Screw drill	2 passes	25.3	5.7	6.2	0.1
Tumbling in rotary drum					
Control		25.3	2.7	4.0	0.2
Tumbling	5 min.	25.3	2.4	3.3	0.1
Tumbling	12 min.	25.3	2.3	3.0	0.1
Drop test					
Control		27.4	5.6	7.5	0.3
Drop	35 ft to steel	27.4	7.7	9.1	0.2
Spinning plate spreader, center feed					
450 rpm	Control	28.2	6.8	8.8	0.3
	1 pass	28.2	8.8	10.9	0.3
1,000 rpm	Control	27.8	7.8	9.7	0.3
	1 pass	27.8	9.3	11.6	0.3

[a]Granular urea, 6×10 mesh.

Additional tests were made to obtain more data on damage to the coatings when SCU was applied with fan-type broadcast applicators. The two commercial-type spreaders tested were of the fan type with the more common off-center feed onto impact-type, bladed spinner disks. Total coating weights on the products were 16% to 22% including 3% wax sealant, 0.25% coal tar, and 2% conditioner. Dissolution rates were measured before and after spreading.

The first spreader tested was a 8-ton, tractor-drawn unit in which the fertilizer from the hopper was carried by a drag-chain conveyor that discharged onto a single spinner disk 18 inches (46 cm) in diameter with six blades. Tests were made with spinner speeds of 500 to 615 rpm (usual in practice is 600 rpm). Total swath width was 50 to 60 feet (15 to 18 m).

The second spreader tested was a 6-ton dual spinner, self-powered truck unit. Feed was by drag chain conveyor, and the material fell into the path of spinning blades. Foam rubber protectors were attached to the backplate to soften the impact of granules. The spinner disks were 18 inches (46 cm) in diameter, each with six blades. The spreader was equipped with a variable speed hydraulic drive to the spinner disks. Speeds up to the maximum 1,000 rpm were tested.

A large polyethylene sheet was used to catch the material that had been broadcast. Representative samples were taken for dissolution tests. Data are shown in table 12.

The results with the single-spinner spreader indicate that the coatings were damaged with a spinner speed of 500 rpm. The 7-day dissolution rate increased by 6 percentage

Table 12. Effect of fan-type fertilizer applicators on dissolution rate of sulfur coated urea[a]

Test No.	Spinner speed, rpm	Product Total coating, wt %	Substrate dissolution in water at 100°F, wt %								
			Before spinner[b]			After spinner[c]			Difference		
			7 days	14 days	Daily	7 days	14 days	Daily	7 days	14 days	Daily
Single-spinner unit[d]											
ST-5	500	21.2	16.9	19.6	0.38	23.2	27.2	0.57	6.3	7.6	0.19
ST-6A	615	17.8	26.0	30.4	0.63	37.7	42.9	0.74	11.7	12.5	0.11
ST-6B	615	20.7	8.7	9.6	0.13	28.4	30.3	0.27	19.7	20.7	0.14
ST-6C	615	21.2	16.9	19.6	0.38	32.0	35.1	0.44	15.1	15.5	0.06
Twin-spinner unit[e]											
ST-7	500	21.9	21.1	27.1	0.85	23.6	29.0	0.77	2.5	1.9	-0.08
ST-8	600	22.6	21.1	27.1	0.85	22.9	28.2	0.76	1.8	1.1	-0.09
ST-9	700	21.6	20.0	24.4	0.63	20.1	26.6	0.93	0.1	2.2	0.30
ST-10	800	20.4	14.9	18.6	0.53	30.7	36.7	0.86	15.8	18.1	0.33
ST-11	900	21.8	18.2	23.3	0.73	32.1	37.5	0.77	13.9	14.2	0.04
ST-12	1,000	22.1	18.2	23.3	0.73	32.8	38.3	0.78	14.6	15.0	0.05

[a]Spherodized urea substrate (6×10 mesh). Coatings include 3% wax sealant, 0.5% coal tar, and 2% conditioner.
[b]Sample taken from drag-chain discharge to spinner.
[c]Ruffled sample from product collected on polyethylene ground sheet.
[d]John Blue Company, Huntsville, Alabama, Model 830.
[e]Fanco Company, Gainesville, Georgia.

Table 13. Effect on rate of dissolution of handling coated granules in elevator buckets

Test No.	Bucket condition	Product condition	Total coating, wt %	Dissolution rate in water at 100°F, wt %				Differences in dissolution rates, wt %	
				Before handling		After handling			
				7 days	14 days	7 days	14 days	7 days	14 days
1	Bare	Unsealed[a]	22.7	12.6	15.8	15.8	20.0	3.2	4.6
2	Bare	Unsealed[a]	27.8	5.0	6.2	5.5	6.3	0.5	0.1
3	Bare	Sealed	21.6	14.1	16.6	15.8	17.8	1.7	1.2
4	Bare	Sealed	30.2	3.5	4.6	3.8	4.2	0.3	-0.4
5	Coated	Sealed	23.9	13.4	16.6	14.2	17.1	0.8	0.5
6	Coated	Sealed	24.9	12.2	14.2	11.9	13.9	-0.3	-0.3

[a]Sealed with wax (3%) in the laboratory prior to dissolution analysis.

points. At 600 rpm (normal speed), the increase was by 11 to 20 percentage points. Padding of the backplate of the applicator likely would help.

With the twin-spinner unit, breakage of coating was low (dissolution rate increase 2 percentage points) at speeds up to and including 700 rpm, but increased sharply at 800 rpm. With speeds of 800 to 1,000 rpm, the increase in dissolution rate was 14 to 18 percentage points, which is excessive.

From these tests, it is concluded that field application of SCU with spinner-type spreaders of the usual design probably will not cause serious damage to the coatings if excessive disk speeds are not used and precautions are taken to cushion the backplate. If the backplate is suitably padded, disk speed up to 700 rpm may be acceptable, as with the dual-spinner unit tested. High-speed spinners (up to 2,400 rpm) such as are used for lime spreading must be avoided in spreading sulfur-coated fertilizers.

In later tests using spinner-type applicators, the urea products coated with sulfur only degraded quite severely with regard to increased dissolution. Special care would be needed, or a different type of applicator might be needed for application of the unsealed products.

Tests were also made to determine whether handling by a bucket elevator would seriously damage the hot product. The tests were made at two levels of coating weights. In one series of tests, the sulfur-coated product was handled in the elevator before the sealant was applied. This simulated the conditions planned in the demonstration scale pilot plant. In the other series of tests, wax-sealed product was handled. Another test variation was the use of bare metal buckets and of those coated with Teflon. The data are shown in table 13.

The difference in dissolution rates before and after handling was greatest with the unsealed product of low coating weight handled in bare metal buckets. Loss in 7- and 14-day dissolution rates was only 3.2% and 4.6% respectively. There were only small losses when sealed products were handled in either type of bucket or when the unsealed product with higher coating weight was handled in the bare buckets. These data indicated that use of a bucket elevator should be satisfactory in commercial units.

PLANT INVESTMENT AND PRODUCTION COST ESTIMATES

Estimates were made of the investment for production plant facilities for SCU at a rate of 300 tons/day. Also, estimates were made of the production cost for SCU, based on latest pilot-plant experience. Use of hydraulic spray nozzles in a coating drum equipped with flights and falling curtain was assumed. This is the most effective system tested in pilot-plant work. Separate estimates were made for:

1. Preparation of full coating of sulfur, wax, and conditioner
2. Preparation of coating of sulfur only

Based on latest pilot-plant data (2 tons/hr rate with hydraulic nozzles in flighted drum), a coating of 18% sulfur, 2% wax, and 2% conditioner was assumed for the full coating. A coating of 22% sulfur was assumed for the sulfur-only coating.

The investment estimates for the plant facilities were arrived at by estimating the cost of individual items of equipment and multiplying the total equipment cost by a factor of 3.5. Cost of bulk storage for 3 months' production was added. Location at an existing urea plant was assumed.

Results of the estimates are shown in tables 14 and 15. The estimated investment (including bulk storage) is substantially lower for coating with sulfur only—$2,000,000 versus $3,000,000. This results from the elimination of equipment for receiving, handling, and metering wax; eliminating equipment for storage, handling, and feeding conditioner; and decreasing cooler capacity to one-half that used for the full coating. Still further decreases in investment would result if equipment for preheating could be eliminated and cooling could be eliminated completely. These possibilities are being studied in the pilot plant.

The operating costs include the following items:

Depreciation (15-yr basis)
Interest (8% on one-half of plant investment)
Taxes and insurance (2% of plant investment)
Operating labor
Maintenance (5% of plant investment)
Analyses (20% of operating labor)
Overhead (100% of operating labor)
Handling (4% of other operating costs)
Interest on working capital (8% on one-half of working capital)
Product loss (0.5%)

The costs of raw materials were estimated from early 1978 delivered costs U.S. Gulf area.

Working capital was calculated as cost of 1 month's supply of raw materials plus 2 months' in-plant value of the coated product.

Return on investment was calculated as 20% of plant investment (including storage) plus working capital.

The data in table 14 for product with sulfur, wax, and conditioner coating show a raw materials cost (exclusive of urea) of $10.20/ton of product and operating costs of $10.80/ton for a production cost for the coating operation

Table 14. Estimated production cost plus return on investment for commercial plant (full coating of sulfur, wax, and conditioner)

Plant capacity—300 short tons/day
Product grade—37-0-0 14S
Estimated total capital investment including storage (existing plant site) $3,000,000

Raw materials	Tons/ton product	Cost, $/ton	Cost, $/ton product
Sulfur	0.14	34	4.75
Wax	0.02	213	4.25
Conditioner	0.02	60	1.20
Total raw materials cost (coating materials only)			10.20
Operating cost			10.80
Total production cost			21.00
Return on capital investment, 20%			9.80
Estimated production cost including 20% ROI			30.80
Straight urea price f.o.b. plant ($120/ton × 0.42)			98.00
Estimated sulfur coated price, f.o.b. plant in bulk			128.80
SCU price, f.o.b. plant, $/unit N			3.50
Straight urea price ($120/ton f.o.b. plant) $/unit N			2.60
Higher estimated price of SCU per unit N, %			34%
Estimated shipping cost in bulk, $10/ton, sales and administrative cost, $6/ton			16.00
Estimated delivered price for SCU			
$/ton in bulk			145.00
$/unit N			3.90
Delivered price for straight urea,			
$/ton in bulk			136.00
$/unit N			3.00
Higher estimated delivered price for SCU per unit N, %			30%

Table 15. Estimated production cost plus return on investment for commercial plant (coating with sulfur only)

Plant capacity—300 short tons/day
Product grade—37-0-0 22S
Total capital investment including storage (at existing plant site) $2,000,000

Raw materials	Tons/ton product	Cost, $/ton	Cost, $/ton product
Sulfur	0.22	34	7.50
Operating costs			8.80
Total production costs			16.30
Return on capital investment (20% on plant investment plus working capital)			7.90
Estimated cost plus ROI			24.20
Straight urea price f.o.b. plant ($120/ton × 0.78)			93.60
Estimated SCU price (f.o.b. plant)			117.80
Estimated SCU price (f.o.b. plant) $/unit N			3.30
Straight urea price (f.o.b. plant) $/unit N			2.60
Higher estimated price of SCU per unit N, %			27%
Estimated shipping cost in bulk $10/ton Sales and administrative cost, $6/ton			16.00
Estimated delivered price for SCU			
$/ton in bulk			134.00
$/unit N			3.70
Delivered price for straight urea			
$/ton in bulk			136.00
$/unit N			3.00
Higher estimated delivered price for SCU in bulk, per unit N, %			24%

of $21.00/ton. A 20% return on investment (plant facilities plus working capital) adds $9.80/ton for a total cost of the coating operation of $30.80/ton of coated product. With urea figured in at estimated f.o.b. plant price of $120/ton ($2.60/unit of nitrogen), the estimated f.o.b. price of SCU, waxed and conditioned, is about $129/ton or $3.50/unit of nitrogen. Thus, the estimated price for the waxed and conditioned SCU is about 34% higher than for straight urea.

The data in table 15 for product coated with sulfur only show raw materials cost (exclusive of urea) of $7.50/ton of product. Operating costs of $8.80/ton and 20% return on investment of $7.90/ton gives a total estimated cost of the coating operation of $24.20/ton. With urea figured in at f.o.b. plant price of $120/ton including return on investment ($2.60/unit of nitrogen) the estimated f.o.b. price of SCU (sulfur only) is about $118/ton or $3.30/unit of nitrogen. The estimated f.o.b. price per unit of N for the SCU is about 27% higher than for straight urea.

AGRONOMIC TESTING OF SULFUR-COATED UREA

Large quantities of SCU have been available from the pilot plants for use in widespread agronomic testing. Tests have been conducted on several crops in 38 U.S. states, 2 U.S. territories, and 29 other countries. All results have not been reported, but the SCU has shown significant advantages in several cropping situations. The general advantages include: decreased cost of application due to fewer applications, avoidance of burning of vegetation or damage to seedlings, and reduction of luxury consumption by plants. SCU also can lead to more efficient uptake by plants and smaller leaching losses, at least under some conditions.

Tests have been conducted on a large variety of crops under various conditions. Crops that have shown the best response to SCU, and where clear-cut advantages have been demonstrated, include rice (particularly under conditions of intermittent flooding), sugarcane, pineapples, forage, turf, and certain fruit and vegetable crops.

Rice

Agronomic test results have shown that perhaps the greatest potential for SCU is on rice grown under delayed or intermittent flooding conditions. Increased yields have been obtained in a large number of tests. The test work has been conducted in the United States, India, the Philippines, Peru, Korea, Pakistan, and Taiwan. These tests have involved each of the major water management practices used in rice growing, except deep water. These included delayed flooding as in U.S. practice (primarily Arkansas), intermittent flooding which is common in several countries where water is not continuously available (rainfed paddy), and continuous flooding in which rice is grown in fields that remain flooded until the crop matures.

The marked superiority of SCU under delayed or intermittent flooding results from its slow- or controlled-release property. Figure #1 shows one example. When conventional nitrogen fertilizers are applied under these conditions, the nitrogen is subject to nitrification by soil organisms during drained or unflooded periods. When flooded, the nitrate-N is denitrified to volatile forms (N_2, N_2O) which are lost to the atmosphere. Nitrate-N may also be lost by leaching. In addition to reducing N losses, savings in application cost can also be realized due to one prepaint application as compared with multiple applications of conventional soluble nitrogen fertilizers.

All factors considered, SCU appears to be a uniquely beneficial fertilizer for use on delayed or intermittently flooded rice. A whole new concept of rice fertilization may be realized through use of SCU.

Sugarcane and Pineapple

Crops such as sugarcane with long growing season (18 to 24 months) and pineapple (18 months) have shown very significant increases in yield and savings in labor costs through use of SCU. The savings in labor costs are realized because fewer applications are required than with conventional nitrogen fertilizers.

Fruits and Vegetables

An experiment with watermelons showed increased yield from SCU over both single and split applications of uncoated urea. This apparently resulted from the resistance of SCU to leaching. Most watermelons are grown in sandy soil either in high rainfall areas or under irrigation.

In experiments with staked tomatoes, an excellent yield increase over a single application of ammonium nitrate and significant increase over five applications were obtained. Elimination of four applications significantly decreases labor cost.

Very good results were obtained with SCU in a strawberry nursery on sandy soil. An increase in salable plants from 112,000/acre to 184,000/acre represented a very substantial increase in gross sales. The cost of fertilizer and application costs were also decreased.

SCU gave increased cranberry yield over split applications of urea, while there was no response to single

Figure 41. Sulfur-coated urea has been tested extensively in many countries. Its greatest potential seems to be on rice that is flooded intermittently. In the above photographs, from Peru, intermittently flooded rice fertilized with SCU (right) yielded nearly one-fourth more than that receiving uncoated urea (left).

applications of urea. The increased yield was due to increased number and size of berries.

Turf

SCU has proven to be an excellent fertilizer for turf grasses in several trials. When compared with a single application of soluble fertilizer, SCU gives less growth immediately following fertilization, but better growth toward the end of the season. This results in less burdensome clippings and a longer lasting turf color.

Forage

A number of experiments have evaluated SCU surface broadcast on established stands of forages. With Coastal bermudagrass, a single application of SCU in the spring resulted in more forage than single or multiple applications of ammonium nitrate. With tall fescue, SCU in a single application was markedly superior to three applications of urea or ammonium nitrate. An experiment with Timothy hay showed a very large yield increase for SCU over a single application of urea.

Other Row Crops

A large number of experiments have been conducted with corn, grain sorghum, small grains, cotton, soybeans, and potatoes. Generally, SCU has shown no advantage for fertilization of these crops. All of these crops need most of their nitrogen over a relatively short period of time; hence, slow- or controlled-release fertilizers normally are not beneficial. However, under conditions favoring leaching and/or volatilization of ammonia or from denitrification, SCU has shown an advantage over soluble sources for several of these crops.

Other Agronomic Advantages

SCU contains elemental sulfur that provides a source of this nutrient. Because of the nature of the coating, the sulfur is not as readily available the first year as in succeeding years.

There have been indications of substantially lower losses of nitrogen by leaching from the soil when SCU is used rather than conventional soluble sources of nitrogen. In addition to conserving more nitrogen for use by plants, this would tend to minimize any losses to streams that might occur in some situations.

Types of Sulfur-Coated Urea Used in Agronomic Tests

SCU with 7-day dissolution rates in water of 10%, 20%, and 30% were used in most of the agronomic tests. In general, for annual crops, best results were obtained with the 20% and 30% dissolution materials. Pilot-plant production, therefore, has been focused on the 20% to 30% material.

Recent tests indicated that microbicide is not required; therefore, it was eliminated in latest pilot-plant operation.

Agronomic tests also are being made of urea coated with sulfur only. First pilot-plant products made with a sulfur-only coating required considerably more sulfur than for waxed products to provide about the same dissolution rate. Latest pilot-plant work has given good results with only a moderately higher coating of sulfur. These latest products would be expected to offer better promise as a controlled-release fertilizer than earlier sulfur-only products. Agronomic tests using urea with some of the earlier rather heavy sulfur coatings indicated that early response was good but response in the latter part of the growing season was not very good. This was believed to be due to the heavier coating of sulfur that resisted dissolution too much, particularly in later periods of the tests.

Recent pilot-plant products have been made with the lighter (effective) coatings of sulfur only and these products are being evaluated in agronomic tests. If the waxing and conditioning treatments can be eliminated, the production process would be greatly simplified and cost of the product would be less.

SULFUR-COATED POTASSIUM CHLORIDE

Agronomists have shown an interest in potash with controlled-release properties. Therefore, tests were made in the small pilot plant to obtain data and to prepare quantities of sulfur-coated potassium chloride (KCl) for use in agronomic testing.

Crystalline KCl (92% -6+10 mesh) that was roughly cubical in shape was the only material available at the time; it was coarsely screened flotation concentrate. The coating procedures were essentially those used in coating urea. The production rate was about 200 lbs/hr (90 kg/hr). Representative results are summarized in the adjacent tabulation.

The dissolution rates were considered to be satisfactory for controlled-release properties. However, they were higher than are obtained with similarly coated urea of about the same particle size. The less effective coatings were attributed to the angular shape of the KCl particles. Additional test work is planned.

Granular KCl was also coated with sulfur and sealant in the large pilot plant at a rate of 1 ton/hr using four spray nozzles. The total coating was varied from 19% to 27% (15% to 23% S). The KCl granules were mostly cubical or elongated cubical in shape, but appearance of the coating was good. At the time of this writing dissolution tests were just being started.

Total coating wt %	K_2O content wt %	Substrate dissolution in water at 100°F (38°C), wt %		
		7 days	14 days	21 days
22.5	48.0	33.8	38.0	40.5
29.0	44.0	22.7	26.2	27.9
32.5	41.8	12.7	15.8	20.4

SULFUR-COATED POTASSIUM CHLORIDE PLUS UREA

Granular KCl that had been coated with concentrated (99%) urea solution in a pilot-plant pan granulator was coated with sulfur in the large SCU pilot plant.

A sulfur-only coating of 30% was applied at a production rate of 1 ton/hr. The product (22-0-11, 30S grade) had good appearance. The 7-day dissolution rate was 17% for the nitrogen and 36% for K_2O. This product is being tested agronomically. If agronomic tests are promising, there should be considerable interest in this type of product.

CONCLUSIONS

The approximately 12 years of laboratory, bench-scale, small pilot-plant, and large pilot-plant work by TVA on SCU has led to the development of a good and economical process for a controlled-release nitrogen fertilizer. Steady improvements in process and product have resulted in substantially decreased coating weights. A product with a coating of only 14% sulfur has suitable controlled-release properties when sealed with a coating of 2% wax and treated with 2% of conditioner such as diatomaceous earth. Latest work showed that a product coated with about 21% to 22% of sulfur only, had suitable controlled-release properties as measured by rate of dissolution in water. Agronomic tests are being made. Improved design of the internals of the sulfur-coating drum in later periods of the development work resulted in the improved coating technique.

Investment and production cost estimates were made for preparation of SCU. For a full coating of sulfur, wax, and conditioner, the f.o.b. price was estimated to be 32% to 34% higher per unit of nitrogen than for straight urea. With a somewhat heavier coating of sulfur only, the f.o.b. price was estimated to be 27% higher per unit of N than for urea. These costs are much lower than those of other controlled- or slow-release fertilizers now available that cost two to three times as much as conventional nitrogen fertilizers.

Very promising results have been obtained in agronomic tests of SCU conducted in 39 U.S. states, 2 territories, and 29 other countries. Perhaps the greatest potential is for fertilization of rice grown under delayed or intermittent flooding conditions. Other crops that showed particularly good potential were long-term crops such as sugarcane and pineapples. Promising results also were obtained in fertilization of forage, turf, and some fruits and vegetables. SCU would be very good for horticultural and other speciality uses.

Because of the great interest shown in SCU throughout the world, TVA plans to go a final step in development by constructing and operating a demonstration plant for production of about 10 tons/hr. This plant will have capability of producing waxed or unwaxed coatings. This operation will fully prove the process so that others could go to commercial-scale production with good confidence. Also, tonnage quantities of product will be available for onfarm testing and evaluation, and for use in bulk blending; this is an important step in market development.

Pilot plants for SCU are known to be in operation in at least three other countries.

Two patents have been issued to the Tennessee Valley Authority on sulfur coating and two others are pending.

Good results were obtained in the pilot plant in sulfur coating of granular KCl and in coating KCl that had been coated with urea in a pan granulator. Results of agronomic tests were promising.

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