AGGLOMERATION OF SUPERPHOSPHATE IN A FLUIDIZED BED

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TABLE OF CONTENTS

INTRODUCTION 1

LITERATURE SURVEY 4

MATERIALS AND METHODS 15

Materials 15

Equipment 15

Experimental Procedure. 23

EXPERIMENTAL RESULTS 26

CONCLUSION 33

ACKNOWLEDGMENTS 39

BIBLIOGRAPHY 40

INTRODUCTION

A progressive trend in the use of commercial fertilizers in this country is a shift from pulverized mixtures to granular mixtures which are less likely to cake or stick in the drill or spreader (2$\frac{1}{4}$). The granulation mixtures are made by processes which transform pulverized mixtures into small grains or pellets.

In 1954, 80 of some 1300 fertilizer mixing plants in the United States were equipped to granulate mixtures. Currently the number has increased to 150 or more. Most of the large manufacturers as well as many small ones have installed granulating equipment. The initial outlay for these plants is from $50,000 to $300,000 or higher depending upon the size of the installation and the process used.

The importance of granular mixed fertilizers is shown in the latest survey of fertilizer consumption in the United States (24). The survey reports the first separate figures on granular mixed products and covers the fiscal year July 1954 through June 1955. In that year 1,331,524 tons or about 9% of all fertilizer mixtures marketed in the continental United States were in the granular form.

Research with plant nutrient materials in granular form in the U. S. Dept. of Agriculture was begun in 1922. The research program was undertaken by the fertilizer section, now within the Agricultural Research Service, in an effort to overcome two problems that were troublesome to the farmers and the

fertilizer industry: 1. caking of pulverized fertilizers during storage and 2. their unsatisfactory drillability. Granulating research began with single plant nutrient fertilizers. The early results were encouraging. By 1930 the researchers introduced a granulating technique that could be recommended for producing mixed fertilizers. In 1936, one chemical company put granular mixed fertilizers into production in the United States.

The current fast mounting popularity of granular mixtures stems mainly from technological advances since 1950, and particularly improvement in the methods for combining ammonia--the most economical source of fertilizer nitrogen--with superphosphates in the mixtures.

Use of granular fertilizers has advanced much faster in some European countries than in the United States. For example, granular fertilizer constitutes about 90% of all the mixed fertilizers in the United Kingdom. This extensive adoption of granular fertilizers is attributed to the fact that soils, climate and crops are less varied than those in the United States and fertilizer manufacturers can meet customer requirements with fewer formulations and grades.

The advantages of granular superphosphate over fine superphosphate are, that

(1) Higher yields are obtained with granular than with finely divided superphosphate in the pot and field tests of cereal crops

(2) Less caking results in storage; thus granulation makes possible the satisfactory farm use of many high analysis

fertilizers that otherwise could not be used.

(3) Blending with other granular fertilizer materials produces granular multinutrient grades.

(4) Free flowing of superphosphate is obtained by granulation.

(5) It is easier to handle and apply, and does not blow out wastefully in windy weather.

(6) It results in the saving in space.

The only disadvantage of granular superphosphate is the cost. Now with improved processes, many granular products are priced a dollar per ton higher than pulverized materials.

The purpose of this research was to investigate the feasibility of performing the agglomeration in a fluidized bed, with a view to reducing the cost of the process.

The usual practice for agglomeration of superphosphate is to use either a rotary dryer or a pan mixer, and no work has been done on granulation of the material in a fluidized bed. If such a scheme could be developed, better control of particle size and more uniform composition and particle shape could result in economies. The process would have to compete with established methods, of course, but it is probable that the simplicity of the equipment and the better control possible would make this feasible.

Agglomeration in a fluidized bed would depend on how vigorously the particles are moving and how closely one could control plasticity of the particles to form strong granules (21). If granulation could be achieved, advantage could be taken of

the size classification that occurs in a fluidized bed. As a further step of modifying the process, it might be possible also to incorporate a drying zone into the same vessel.

LITERATURE SURVEY

The first process to be used commercially for producing granular superphosphate was the Oberphos process (23), in which phosphate dust and sulphuric acid were injected into a rotary autoclave and reacted under pressure. The process is not extensively used in this country.

Ross and co-workers of the Bureau of Agricultural Chemistry and Engineering began studies on the granulation of fertilizers (4) and fertilizer mixtures as far back as 1931 (10). Of the several methods of granulation which they studied, that of tumbling disintegrated material in a rotary cylinder with the addition of heat or water, or both, was considered the best for ordinary superphosphate. A process in which superphosphate was granulated by spraying water on it in a rotary cylinder was patented by the Davison Chemical Company; the granulated superphosphate was subsequently dried in a rotary drier to accomplish accelerated curing and to harden the granules. This process is reported to be in commercial operation.

A similar process consists of spraying water on a mixture of superphosphate and basic slag, both finely ground, in a rotary cylinder; granulation and neutralization of the free acid is accomplished. The processes for granulating superphosphate were studied by the Tennessee Valley Authority in the following

particulars. (i) Flaking of green superphosphate by means of rolls. (ii) Spraying fluid superphosphate from a centrifugal mixing device. (iii) Spraying fluid superphosphate from a mixing nozzle.

Pan granulators (13) are in use in several countries for granulating superphosphates and mixed fertilizers. Relative to the search for improvement of processing equipment, there is considerable interest in the United States regarding the suitability of the pan for ammoniating and granulating high analysis fertilizers.

Current processing practice in the United States differs from that in most countries in that it often includes the use of sulphuric acid or phosphoric acid in conjunction with a quantity of ammoniating solution in excess of that necessary to ammoniate the superphosphate present in the mixture. The quantity of the solution phase present in the original formula is sometimes in excess of that required for agglomeration, and requires recycling of the product fines to dry the slurry and bring the mixture into an agglomerated state.

Technical literature on the operation and efficiency of pan granulators for mixed fertilizer production is sparse, but there appears to be sufficient information available to justify a rather broad evaluation of this equipment for ammoniating and agglomerating high-analysis fertilizers as they are formulated in the United States (10).

The two broad classes of pan granulators are the horizontal pan which is primarily a batch mixer, and the inclined pan which

is used for continuous granulating operations.

Another agglomerating device consists of a slightly inclined rotating cylinder (9). This is frequently used for continuous agglomeration of mixtures. Such drums vary in size, but a typical one, inclined at 2° from the horizontal and rotating at 8 rev/min, is 6.5 ft. in diameter and 18 ft. long. The operator, stationed at the discharge end, controls the continuous addition of water to the material on the basis of the appearance of the moist agglomerates as they approach the lower end of the cylinder. A buildup of 'skin' or mud is maintained on the smooth wall of the cylinder to reduce slippage of material and promote the rolling and tumbling action necessary for the formation of the agglomerates. In another procedure the rotary cylinder is used merely to compact agglomerates which are formed in a previous step of the process.

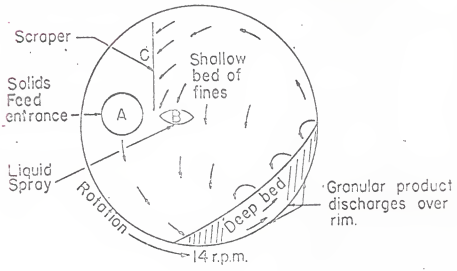
About 10 years ago, the West German Associates of Delo (Engineers) Ltd., London, England, are reported to have developed the Loesche inclined pan for granulating superphosphate and mixed fertilizers (13). The pan gained considerable commercial use for granulating fertilizers in West Germany. Similar units of various sizes have been used either for commercial production or experimentation in various countries.

Reppet (13) in Germany used an inclined pan for acidulating finely divided phosphate rock with either sulphuric acid or phosphoric acid and granulating the product superphosphate in one operation. For example, ground phosphate rock was fed continuously to the central area of the pan and sprayed with

phosphoric acid containing 43% P_2O_5 (or sulphuric acid of the required strength) at a rate sufficient to form triple superphosphate (or superphosphate). As the granular product flowed over a baffle into a channel at the periphery of the pan, it received a 2% coating of rock dust which facilitated curing the product in storage. Granulated mixed fertilizer was made in the same equipment by spraying the mixture with aqueous ammonia.

Considerable industrial research has been carried out in Australia on the use of the inclined pan for granulating superphosphate to the extent of reducing the dust content of the product used for top dressing or direct application. While the pan gives variable results, owing to small differences in the character of the superphosphate feed, it is still being used to produce a coarse grade of superphosphate for direct application. In this operation run-of-pile superphosphate is passed over a 3/16 inch screen and the fines are fed to the pan. A water spray is directed on the fine material leaving the scraper indicated in Figure 1. The larger particles leave the pan with little or no wetting and no size enlargement, while the fines receive most of the water added. Thus the moisture required for granulation, depending on the fines content, is 2 to 3% less than that required for granulating the same material in the rotary cylinder. This illustrates the classifying or segregating action of the inclined pan which is highly desirable in granulating individual materials, but less desirable with mixtures in which homogeneity in nutrient content of the granular product is an important factor.

Figure 4. Diagram showing action of the rotating inclined pan.



Operating characteristics of the pan may be affected by altering the positions of the solid feeder and liquid spray nozzles; also, by modification in design or position of the scrapers, spreaders and deflectors within the pan or the height and shape of the rim. Aside from the classifying action peculiar to the inclined pan, the rolling and cascading motion imparted to the material being agglomerated essentially is the same as that obtained by the use of a rotary cylinder. The principles of agglomeration which involve the progressive gathering of the small particles into a larger interlocked structure are the same in either piece of equipment.

It is known that two of the principal factors concerned in agglomeration of moist particles are the average particle-size and the size distribution (10, 11, 12). The chemical composition of the material is less important, provided none of the constituents is soluble in the liquid (usually water) responsible for cohesion. The cohesive forces which are primarily due to surface tension will, however, depend upon the wetting characteristics (angle of contact) of the liquid with respect to the solid. The presence of soluble constituents may increase the surface tension and, if a drying stage is included, may form a crystal net work which could bind the insoluble particles together.

Newitt and Conway-Jones (19) calculated the weighted mean granule diameter of each sample and plotted these against the time of granulation. They found an approximately linear relation, the slope of which corresponded to the rate of granule

growth. A considerable variation of the rate of growth occurred for comparatively small changes in the moisture content. It appeared that there was a critical value of moisture content below which the rate of growth is negligible; for fine sand, this is approximately 62% (19).

Strong, uniform granules of fine sand are most easily made with a moisture content in the range of 64-69%. But with higher moisture contents, the rate of growth becomes excessive and the granules are weaker and tend to collapse under their own weight. If the charge is too dry the sand adheres to the surface of the drum and when dislodged it tends to slide rather than roll; in these circumstances granules are not formed.

It was found, however, that the moisture was not uniformly distributed and the material which had not granulated was drier than the average. It is, therefore, probable that to initiate granulation in fine sand, there must be nuclei with a moisture content of not less than 62%. Granules so formed may be considered to be approximately saturated and to have a slight excess of moisture at their surface. This allows a certain degree of 'surface-plasticity' which enables partial deformation to occur when two granules are in collision.

A granule containing less moisture is more rigid and with no excess moisture in the surface, would not, on collision with another granule be able to coalesce. Since direct cohesion of two such surface-dry granules can not take place the rate of growth will be slow.

A granule with a moisture content above 62% can coalesce

with a smaller granule, and the action of tumbling in the drum soon kneads such pairs into a nearly spherical shape.

The rate of granule growth depends upon the ability of granules to deform and coalesce upon impact with one another. Thus, a fundamental variable is the crushing strength of the granules, and it was shown that among other factors, this is inversely proportional to the average particle size. It would, therefore, be expected that the finer materials would produce stronger granules and have a slower rate of growth (19).

Mixed sand and silt proved easy to granulate, as there is little tendency for it to stick to the drum (19). The mean granule diameter was shown to increase with time at rates which depended upon the initial moisture content (14). These relations were not linear, but tended to flatten out at higher granulation times. This was accompanied by a considerable increase in strength, which these granules attained during the granulation process (2).

An important property of a powder which indirectly affects its rate of granulation is the porosity or bulk-density to which it may easily be compacted. It has already been suggested that in order to make good granules, the pore space should be almost completely filled. The amount of liquid required therefore will depend upon the packing properties of the material.

Fraser (19) has shown that the porosity of a randomly-packed system of spherical particles depends upon the range and distribution of particle sizes. In particular he shows that the porosity of a random bed of uniform spheres will be reduced if

a proportion of smaller uniform spheres is introduced. The reduction will be most marked if approximately 25% of the smaller constituent is added. This is the largest quantity of smaller spheres that can be accommodated within the interstices between larger spheres.

The granule containing less moisture is more rigid and with no excess moisture on the surface will not, on collision with another molecule, be able to coalesce. Such 'surface dry' granules might, however, be able to hold individual grains if the latter were sufficiently moist to form a 'pendular' bond at the point of contact. Since direct cohesion of two such surface-dry granules can not take place the rate of growth will be slow. (14, 19, 22).

Six ideal properties of fertilizers have been drawn up by research scientists in the Agricultural Research Service. Some of these specifications can probably never fully be attained, though they can be approached. On the other hand, two of the more precisely stated goals are nearly met in granular mixtures now on the market (24, 17). These six properties are:

1. Particle size should be 10-14 mesh or 0.065 to 0.046 in. Most granular mixtures now sold contain some granules of these sizes but the range is wider. Too great a range of particle sizes carries certain disadvantages. A fine product may cake and thereby defeat one of the chief purposes of granulation, which is to avoid caking. A coarse granule may distribute nutrients unevenly among individual plants. For example, if coarse 5-mesh granules are applied at a rate of 150 pounds per acre,

crop rows 7 inches apart, only 11 granules will be placed per linear foot. If medium size 10-mesh granules are similarly applied, 154 granules will be placed per linear foot (3).

2. Particle shape should be spherical. Fertilizers consisting of smooth and perfectly shaped balls are at present impractical in farm use because of the high cost of producing them. Yet the rounder the granules, the better they will resist crumbling and packing. It is practical for the commercial mixtures to contain a large proportion of fairly well-rounded granules. This is achieved in one processing method by grinding the source materials fine, then moistening and rolling them in a rotating cylinder so that as they agglomerate they are smoothed and rounded.

3. Particle structures should be such that 95% of the particles are strong enough to resist crumbling and packing under a load equal to 100 lbs. per sq. in. Most of the granular products now marketed have adequate strength.

4. Homogeneity should be such that each granule is up to grade; that is, each contains its share of nutrients. It is found in a commercial fertilizer that the higher the nutrient content of the granular mixture, the less uniform is the nutrient distribution between granules.

5. Drillability of the product should be such that it will stay free from lumps and maintain its original drilling rate for one hour when exposed to 88% relative humidity at 76°F. A number of experimental and commercial granular products were recently tested for this degree of drillability at the Agricultural

Research Center. Most of the high analysis fertilizers passed this test even though some contained as much as 785 lbs. of ammonium nitrate per ton (17).

6. Fertilizing efficiency of the product should be such that the best possible delivery of nutrients to the growing plant is obtained. In experimental work some differences in certain situations have been observed. These differences involve chiefly relationships between the granule size of the mixture and the water solubility of the phosphorous, especially when fertilizer is used on neutral or alkaline soils (9).

Concern about the particle size of the raw materials used in the fertilizer granulation is evident in the recent literature. Standardization of the particle size of raw materials and the effects of particle size on granulation efficiency are subjects of current interest to the fertilizer industry (21,23).

Granulation efficiency of triple superphosphate during ammoniation was best when the initial material had a low proportion (5-15%) of coarse (6-20 mesh), and a relatively low proportion (10 to 20%) of fine (-65 mesh) particles. A higher proportion of coarse particles was detrimental to particle growth, and a higher proportion of fines made the material sensitive to slight variations in moisture content. A triple superphosphate with hard, discrete particles granulated more efficiently than one with soft, fragile particles and indicated greater response to changes in initial average particle size. Both the pattern of the size distribution and the average size of the raw materials are determining factors in granulating efficiency (24).

MATERIAL AND METHODS

Materials

The materials used in this study were air, steam and superphosphate. The air was obtained from the compressed air lines present in the building. The steam was produced from tap water vaporized in a small gas-heated vaporizer. The superphosphate was commercial grade containing 20% P_2O_5 and was purchased from the Snyder Chemical Company.

It was necessary to have the superphosphate of the right particle size. Extremely coarse particles were very difficult to fluidize, while extremely fine particles were blown out of the fluidized beds. The commercial sample was screened and the particles larger than 25 mesh were rejected. Photographs of the as received material and the screened fractions are shown on Plate 1.

Equipment

Plate 2 and Figure 2 show the equipment used in this study of the agglomeration of superphosphate in a fluidized bed. The fluidizing column, shown in Plate 3, was 2 feet high and 4 inches in diameter. It was of plexiglass so that the material in the column could be observed easily.

Fluidizing air was obtained from the Physical Plant. The entrained water and oil in the air was removed in the trap (J). The trap was a cast iron pipe 1 foot high and 3 inches in diameter packed with fiberglass. Most of the condensed water was

PLATE I

Various fractions of the superphosphate

A. Commercial 0-20-0 Superphosphate as received.

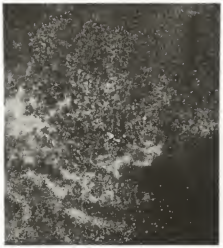
B. Superphosphate that passed through 25 mesh screen.

C. Superphosphate retained on the 25 mesh screen.

D. Superphosphate through which humidified air had passed.

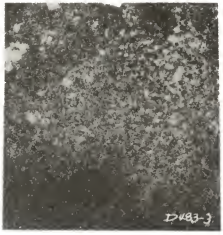
PLATE I

A



B

C

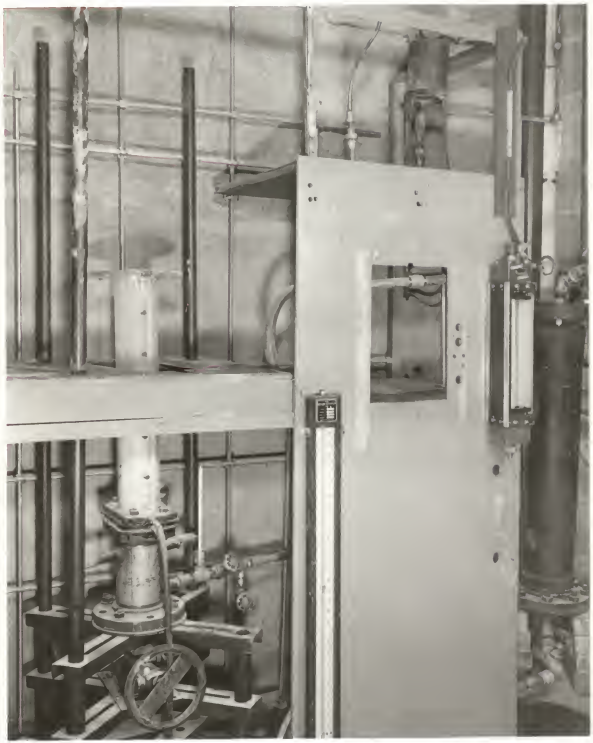


D

PLATE II

THE EQUIPMENT SET UP FOR AGGLOMERATING THE SUPERPHOSPHATE.

PLATE II



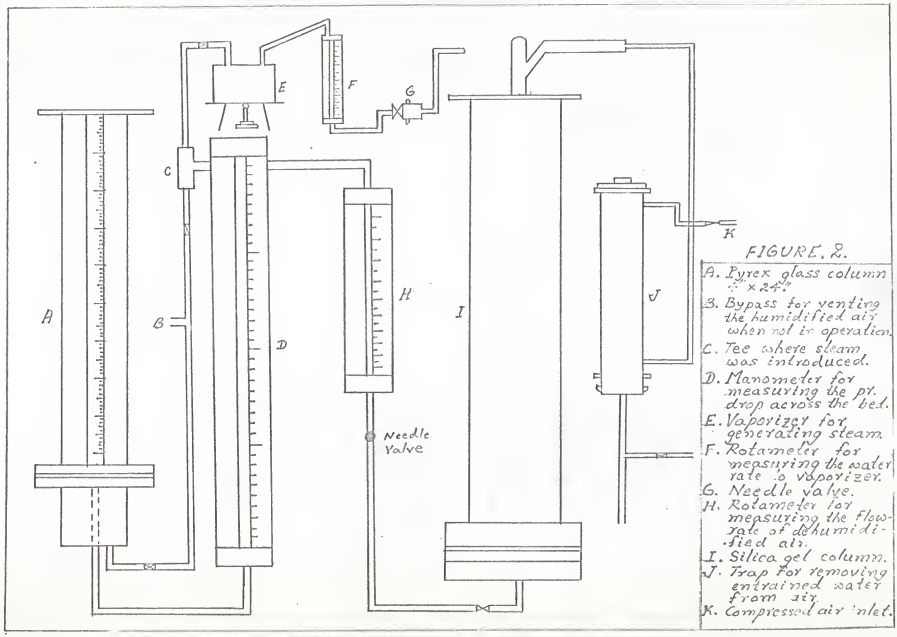


FIGURE. 2.

A. Pyrex glass column ⅜" x 24".
B. Bypass for venting the humidified air when not in operation.
C. Tee where steam was introduced.
D. Manometer for measuring the pr. drop across the bed.
E. Vaporizer for generating steam.
F. Rotameter for measuring the water rate to vaporizer.
G. Needle valve.
H. Rotameter for measuring the flow-rate of dehumidified air.
I. Silica gel column.
J. Trap for removing entrained water from air.
K. Compressed air inlet.

PLATE III

COLUMN IN WHICH SUPERPHOSPHATE IS FLUIDIZED.

PLATE III

absorbed by the fiberglass.

The air leaving the trap (J) entered the dehumidifying column (I). The column was a cast iron flanged pipe 3 feet high and 6 inches in diameter. The upper and lower ends were flanged to 1-inch pipe. The column was filled with silica-gel, which rested on a wire gauze. The air entered through a Y at the upper end of the trap. The vertical run of the Y was used for removing and recharging silica-gel.

Twenty-five lbs. of silica-gel were placed in the column. This could absorb about 5 lbs. of water before becoming saturated. This was sufficient capacity to dehumidify some 12,000 cu.ft. of air, and this was sufficient for all of the runs performed (4, 18, 20).

The air leaving from the bottom of the dehymidifying column was passed through a tri-flat variable area rotameter (H). A globe valve and a needle valve were used to control the rate of the dehumidified air. The humidity of the air leaving the silica-gel column was assumed to be nearly zero. It was then humidified to the desired percentage humidity, by introducing steam produced in the water vaporizer. The water rate to the vaporizer was measured by the rotameter (F). The moment the water entered the vaporizer it was vaporized since the vaporizer was hot. The superheated steam formed was mixed with the dehumidified air obtained from the dehumidifying column. The temperature of the humidified air was never more than 7 to 8°F higher than that of dehumidified air.

The humidified air entered the column (A) from the bottom

through a 1-inch diameter rubber hose. The upper end of the column was open to the atmosphere. The pressure drop across the fluidized bed of superphosphate in the column was measured by the manometer (D) connected to the lower end of the column.

Experimental Procedure

Commercial superphosphate was screened through a 25 mesh screen. The fine fraction was used for the experimental work. Three lbs. of the material was the standard charge for each run. This gave a bed height of 6 inches. The burner under the water vaporizer was lighted, and the water rate was adjusted to the desired rate. The flow rate was kept as constant as possible by constant manual control.

Air coming from the main was let out first through the trap to remove entrained water. When no more liquid appeared in the air, the purge valve was closed. The air was then passed through the silica-gel bed, and the flow rate adjusted to the desired value. The steam and air were mixed and vented to the atmosphere until the system came to equilibrium. The temperature of the humidified air was checked for every run.

The runs lasted for predetermined periods and at the end of the run, a representative sample was taken from the column by means of a sampling probe. The sample was kept in a glass bottle for average particle size analysis.

In the runs at higher air velocities, it was felt that fine particles were being carried away from the column. To collect the fines, a cloth bag was tied on the top of the column. After

each run the cloth was tapped slowly so that the fine particles fell back into the column. This material was mixed with the material in the column by slow fluidization before sampling.

The effect of direct addition of water was investigated by adding liquid water from a burette above the center of the fluidized mass. The rest of the procedure was the same as above.

Both microscopic measurement and standard sieving were found to be unsatisfactory for determining the average particle size or the size distribution. The Fisher Sub-sieve Sizer was finally selected for use, because it gave a single average particle size rather than a size distribution. It was rapid, and it did not use vibration or shaking which could change the particle size of the sample through abrasion. This device gave fast, accurate and reproducible results in a form which made it possible to determine particle growth readily (5, 7). The Fisher Sub-sieve Sizer measures average particle diameters in the range of 0.2 to 50 microns. The instrument employs what is generally known as the air-permeability method for measuring the average particle size of a powder. It is based upon the fact that a current of air flows more readily through a bed of coarse powder than through an otherwise equal bed of fine powder; i.e., equal in shape of bed, apparent volume and percentage of voids; but by reason of difference in general coarseness of material, differing in average pore diameter and in total interstitial

surface. The value of the average particle diameter, obtained with this instrument, is numerically equal to six times the total volume of the sample under test divided by the total surface of all the particles contained in this sample, regardless of particle size distribution. The actual mechanism of the Sub-sieve Sizer is described, very briefly, as follows:

An air-pump supplies air at a constant pressure to the regulator. The air under this pressure head, is conducted to the packed powder sample contained in the sample tube. The flow of air through this packed bed of powder is measured by means of a calibrated flowmeter, in which the level of the fluid indicates directly on a chart the average diameter of the powder particle.

Starting with the fundamental equations relating the flow of fluid through a bed of particles to the physical characteristics of that bed, Gooden and Smith (8) have derived the formula

$$d_m = \frac{60{,}000}{14} \sqrt{\frac{ncF\ DI^2\ M^2}{(VD - M)^3\ (P - F)}}$$ in which

d_m = average diameter in microns (one micron = 10^{-3} cms)

n = viscosity of air in Poises

c = conductance of the flowmeter resistance in cc per second per unit pressure (grams per sq. cm.)

F = pressure difference in grams per sq. cm. across the flowmeter resistance

D = density of the sample in grams per cc.

L = length or height of the compacted sample; i.e., depth of bed in cm.

M = mass of the sample in grams

V = apparent volume of compacted sample in cc.

P = overall air pressure head in grams per sq. cm.

By the expedients of using a sample weight equal in grams to the true density of the material, of packing the sample to a known degree, and of selecting and fixing several of the other variables, the equation may be simplified to the extent that the average particle diameter is indicated by the value of F only; i.e., as far as the instrument is concerned, by the height of a column of liquid in a glass tube. In the sub-sieve sizer, it is not necessary to make an actual measurement of the height of the liquid column. Instead, by proper use of the calculator chart, the average particle diameter may be read directly from the chart.

EXPERIMENTAL RESULTS

The experimental data are presented in Tables 1, 2, 3, and 4. The values of the average particle diameter in Tables 1 and 2 are about the same as that of the superphosphate charged. No significant change in particle size has occurred.

The values of the average particle diameters in Tables 3 and 4 indicate a definite trend toward larger particles. It is believed that this was caused by elutriation of fine particles from the bed at the higher air rates used.

The data in Table 5 confirm this conclusion. For these runs the fine material elutriated from the bed was caught in a bag filter and returned to the bed at the end of the run. It was mixed with the bed by slow fluidization before sampling. These data show that for the first 20 minutes of fluidization, no apparent change in particle size occurs, though a slow

Table 1. Agglomeration of superphosphate. Air flow rate, 0.4 cu.ft./min.; pressure drop across the bed, 0.1 inch of water; temperature of fluidizing air, 81°F; average particle diameter of charge, 33.2 μ.

Run No.	Relative Humidity, %	Time of run, minutes	Average Particle Dia., microns
1	25	20	32.2
2	25	40	32.4
3	25	60	32.6
4	50	20	32.0
5	50	40	33.0
6	50	60	33.6
7	75	20	33.0
8	75	40	31.2
9	75	60	30.0
10	100	20	31.0
11	100	40	31.2
12	100	60	32.6

Table 2. Agglomeration of superphosphate. Air rate, 0.6 cu.ft./min.; pressure drop due to superphosphate 0.1 inch of water; temperature of entering steam 81°F.; material taken in the fluidized bed 3 lbs.

Run No.	Relative Humidity, %	Time of run, minutes	Average Particle Dia., microns
13	25	20	30.0
14	25	40	31.0
15	25	60	32.8
16	50	20	31.0
17	50	40	33.4
18	50	60	34.2
19	75	20	30.2
20	75	40	33.2
21	75	60	32.2
22	100	20	31.6
23	100	40	31.2
24	100	60	33.4

Table 3. Agglomeration of superphosphate. Air flow rate, 0.8 cu.ft./min.; pressure drop across the bed 0.1 inch of water; temperature of fluidizing air, 81°F; average particle diameter of charge, 33.2 microns.

Run No.	Relative Humidity, %	Time of run, minutes	Average Particle Dia., microns
25	25	20	33.0
26	25	40	35.0
27	25	60	35.6
28	50	20	34.0
29	50	40	35.8
30	50	60	36.0
31	75	20	36.0
32	75	40	36.0
33	75	60	35.0
34	100	20	35.6
35	100	40	35.8
36	100	60	38.0

Table 4. Agglomeration of superphosphate. Air flow rate, 1.2 cu.ft./min.; pressure drop across the bed 0.1 inch of water; temperature of fluidizing air, 81°F; average particle diameter of charge, 33.2 microns.

Run No.	Relative Humidity, %	Time of run, minutes	Average Particle Dia., microns
37	25	20	38.0
38	25	40	36.6
39	25	60	39.2
40	50	20	36.0
41	50	40	37.4
42	50	60	39.2
43	75	20	36.0
44	75	40	36.0
45	75	60	38.0
46	100	20	34.4
47	100	40	37.4
48	100	60	36.8

growth appears at the longer fluidizing times, particularly with air of high humidity. The growth is not enough at best to make the process look promising.

Since agglomeration did not occur in beds fluidized with humidified air only, a series of runs was made in which liquid water was added dropwise from a burette to the top of the fluidized bed. The results of three runs in which water equal to 1, 2 and 5% of the weight of the bed was added are shown in Table 6. The air velocity for this series of runs was low, 0.6 cu.ft./min., and the relative humidity of the air was maintained at 50%. Visual observation of the column showed the presence of almost spherical balls of agglomerated superphosphate. The mechanical strength of these balls was so low, however, that removal from the column resulted in so much breakage that the average particle size was not increased much.

A second set of three runs was made at a high air velocity and with low humidity air. To prevent elutriation of fines, a terry cloth filter was stretched over the top of the column, and the tip of the burette was inserted through the center of the cloth. See Plate 4.

The formation of agglomerates around the drops of water was easily observed in these experiments. In order to increase their strength, the charge was left in the column for several hours (overnight) while air was passed through the bed. The drying action which resulted made it possible to remove the charge from the bed by using the suction generated by an industrial vacuum cleaner. The results of these tests are shown

Table 5. Agglomeration of superphosphate with elutriated fines returned to bed. Air rate 1.2 cu.ft./min.; pressure drop due to superphosphate 0.1 inch water; temperature of the entering stream 81°F. Av Particle Dia 33.2 μ

Run No.	Relative Humidity, %	Time of run, minutes	Average Particle Dia., microns
49	25	20	31.0
50	25	40	33.4
51	25	60	36.0
52	50	20	33.6
53	50	40	32.0
54	50	60	35.0
55	75	20	32.0
56	75	40	33.0
57	75	60	36.0
58	100	20	32.6
59	100	40	34.6
60	100	60	37.0

Table 6. Agglomeration of superphosphate with liquid water. Air rate 0.6 cu.ft./min.; % humidity maintained constant at 50%; pressure drop due to superphosphate 0.1 inch water; temperature of the entering stream 88°F. Time of run 20 minutes. Av Particle Dia 33.2 μ

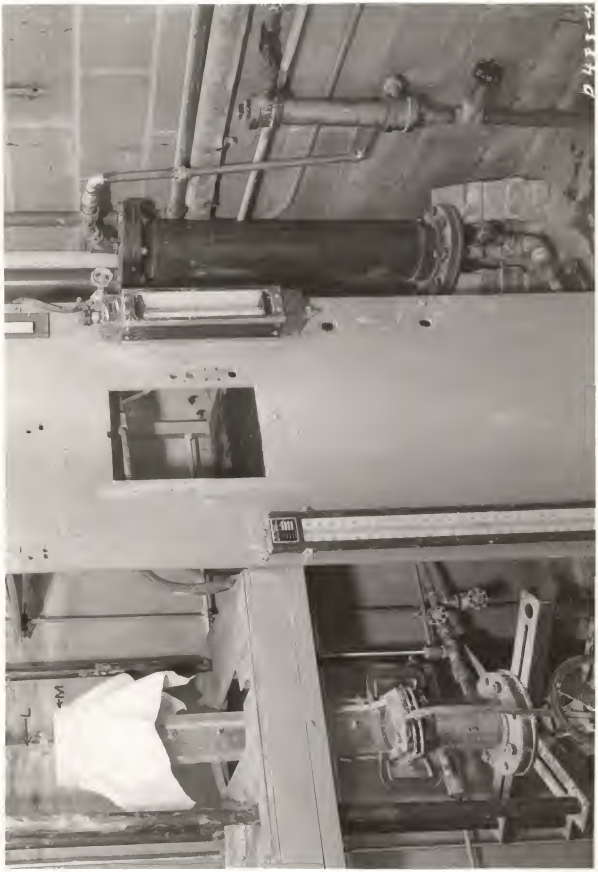
Run No.	% H_2O	Average Particle Dia., microns
61	1	34.6
62	2	35.8
63	5	36.6

PLATE IV

L. Burette through which water is added to the material.

M. Terry cloth filter attached so that fines may be recovered.

PLATE IV



in Table 7. The appearance of the agglomerates in comparison to the charge is shown on Plate 5.

The material retained on a 30 mesh sieve from these runs was dried in an oven at 100°F. The screen analysis of the dried agglomerates from run 66 is shown in Table 8. This material was hard and had essentially the same strength as commercially agglomerated superphosphate.

CONCLUSION

The results shown in Table 6 indicate that agglomeration could occur in a fluidized bed. The agglomerates were weak, however, and broke when they were removed from the column. Some drying of agglomerates in the bed probably will be necessary.

Scale up of a fluidized bed would presumably involve keeping the bed height constant, but increasing the bed's cross-sectional area to accommodate the increased volume of air while maintaining the superficial velocity constant.

Agglomerates tend to settle in a fluidized bed. A conical column would help to keep the larger particles in motion. After a period of growth and drying, they could be removed continuously. Figure 3 shows the design of a proposed column for an experimental continuous process.

The cost of agglomerating superphosphate in a fluidized bed would be an important factor in determining the economics of a process plant such as this. One of the cost elements would be the power consumed in blowing air through the fluidized bed. The pressure drop to be overcome would be at least equal

Table 7. Agglomeration of superphosphate with liquid water. Air rate 3.3 cu.ft./min.; humidity of air zero; time of run 60.0 minutes. Charge was 100% through 30 mesh sieve.

Run No.	% water added	% Retained on a 30 mesh sieve
64	2%	5.17
65	5%	5.17
66	10%	10.30

Table 8. Sieve analysis of agglomerated material from Run 66 (after drying).

Mesh	Wt. in gms.
30	21
20	21
14	29
12	12
10	11
below 10	58
	152

PLATE V

A. Screened commercial superphosphate taken for fluidizing.

B. Superphosphate obtained by adding water from the burette and fluidizing in air.

PLATE V

A

B

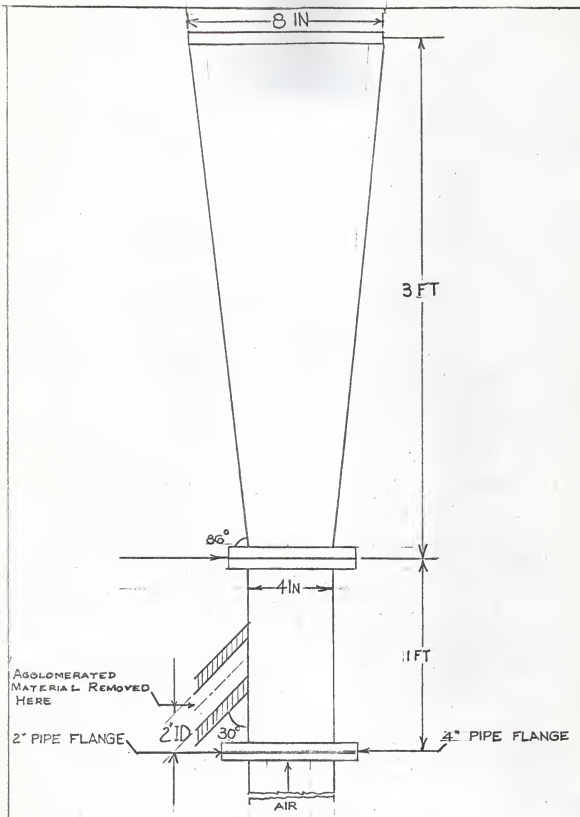


FIGURE 3

FLUIDIZED COLUMN FOR FUTURE WORK

to the weight of the bed per unit area, amounting in the typical case to about 1.0 lb./sq.in., and increasing only slightly with the air velocity.

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AGGLOMERATION OF SUPERPHOSPHATE IN A FLUIDIZED BED

by

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AN ABSTRACT OF A THESIS

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Agglomerated superphosphate is generally accepted as superior to pulverized superphosphate. It yields better crops, cakes less in storage, blends easily with other granular materials, is more free flowing, handles easier and requires less space as compared with the pulverized product.

The purpose of this study was to investigate the agglomeration of superphosphate in a fluidized bed when air of different relative humidities was passed for different periods of time at different rates through a fixed quantity of superphosphate in the fluidized bed.

For the purpose of this investigation a pilot plant was designed and constructed. The equipment consisted of (i) silica-gel bed for drying air to low humidity; (ii) vaporizer for generating steam; (iii) a 4-inch cylindrical column of lucite for the fluidized bed. The experiments were carried out at atmospheric pressure and at room temperature.

Superphosphate is not very hygroscopic. Therefore, the amount of moisture absorbed from the air was not enough to cause agglomeration to occur, since no liquid films were formed on the superphosphate.

When liquid water was added dropwise, agglomerates were formed. The size of the agglomerates were about proportional to the size of the water drops. The agglomerates formed were weak in strength unless dried.