



(22) Date de dépôt/Filing Date: 2009/04/16

(41) Mise à la disp. pub./Open to Public Insp.: 2010/10/16

(51) Cl.Int./Int.Cl. *C05D 9/00* (2006.01),
C05G 5/00 (2006.01), *A01C 15/00* (2006.01)

(71) Demandeur/Applicant:
SULPHUR SOLUTIONS INC., CA

(72) Inventeur/Inventor:
PEDERSEN, ERIC ANDREW, CA

(74) Agent: MILLER THOMSON LLP

(54) Titre : PASTILLES DISPERSIBLES D'ENGRAIS SOUFRE

(54) Title: DISPERSIBLE SULPHUR FERTILIZER PELLETS

(57) **Abrégé/Abstract:**

A water dispersible pellet and method of producing same comprising: micronized elemental sulphur with 80% of particles less than 30 micron in diameter; a binder component present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; a surfactant in the amount ranging from 0.05% to 10% by weight of the total dry weight of the pellet; a soluble salt present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; bentonite clay in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet, the pellet having a mean particle domain size and a mean crushing strength, all in a form such that within a few minutes of contact with water the pellet disperses into particles with more than 10% of said particles passing through a 50 mesh (US Standard Size) screen.



ABSTRACT

A water dispersible pellet and method of producing same comprising: micronized elemental sulphur with 80% of particles less than 30 micron in diameter; a binder component present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; a surfactant in the amount ranging from 0.05% to 10% by weight of the total dry weight of the pellet; a soluble salt present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; bentonite clay in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet, the pellet having a mean particle domain size and a mean crushing strength, all in a form such that within a few minutes of contact with water the pellet disperses into particles with more than 10% of said particles passing through a 50 mesh (US Standard Size) screen.

15

20

25

DISPERSIBLE SULPHUR FERTILIZER PELLETS

FIELD OF INVENTION

The invention relates to elemental sulphur fertilizer, and more particularly, to compositions
5 and methods for producing dispersible elemental sulphur fertilizer pellets.

BACKGROUND OF INVENTION

It is generally known in agricultural sciences that sulphur fertilization increases crop yield
and quality and further has a beneficial effect on nitrogen processing by plants. This nitrogen
processing is in turn related to protein synthesis, cell replication, photosynthesis, and disease
10 resistance.

However, more stringent air pollution regulations and the reduction of sulphur dioxide
emissions have resulted in a greater incidence of sulphur deficiencies in agricultural soils,
thereby increasing the demand for sulphur containing fertilizers.

Generally speaking, powdered sulphur has little utility as an agricultural fertilizer because of
15 difficulty in handling, and the sulphur dust acts as an eye irritant. Sulphur dust also presents
an explosive risk in handling facilities and segregates if blended with granular fertilizer
products.

Accordingly, most agricultural fertilizers used in field applications are formulated into
particles in order to alleviate these drawbacks. Particulate products can be easily handled
20 without the generation of excessive dust and segregate less when blended with other
particulate products.

Conventionally, sulphur has been applied in the form of elemental sulphur, ammonium
sulfate, ammonium thiosulfate, ammonium bisulfate, sulfides or calcium sulfate (gypsum).

Due to the high analysis of elemental sulphur fertilizer products, they are more cost effective
25 than sulfate products because of lower transportation, handling, and storage costs. High
analysis fertilizer products also allow more flexibility when blending with other nutrient
products. The main drawback of elemental sulphur products is that their oxidation to plant
available sulphate is slow often taking many years from the time of application.

The effectiveness of elemental sulphur fertilizers depends on the rate of their oxidation to the plant-available sulfate form. In soil, oxidation to sulphate is a microbial process involving bacterial species such as Thiobacillus. The rate of this reaction is strongly influenced by soil
5 and environmental factors as well as the physical and chemical properties of the fertilizer product. Sulphur oxidation is generally a surface reaction. Thus, the amount of sulphate produced per unit of time is a function of the total surface area of elemental sulphur present, not of its mass, hence the rate of oxidation is strongly influenced by sulphur particle size.

Two physical attributes of fertilizer products that have a substantial effect on oxidation rates
10 are the extent of dispersion from the fertilizer granule after it has been introduced on or in the soil and the size of the dispersed sulphur particles, with smaller particles oxidizing more rapidly than larger particles.

In order to provide greater dispersion of the sulphur some elemental sulphur products mix the sulphur with bentonite clay by means of a hot process utilizing molten sulphur during the
15 production stage. Upon application, the clay functions to imbibe water and break apart or disperse the particulate. In actuality the particulates do not readily disperse into finely divided particles and sulphur oxidation proceeds at a very slow rate. This occurs due to molten hydrophobic sulphur coating the clay particles during the production stage preventing contact with water.

20 Thus, it would be advantageous to have a granular elemental sulphur containing fertilizer which has a high nutrient content and quickly disperses into finely divided particles, say for example sub 30 micron particles, once contacted with water on or in the soil.

Various products and processes have heretofore been proposed. For example, U.S. patent 4,133,669 relates to a process for pelletizing a mixture of elemental sulphur and bentonite
25 clay to produce a water degradable prill which includes adding dry clay dust to molten sulphur at an elevated temperature to provide a molten sulphur-clay mixture, forming droplets of the mixture, providing a liquid fertilizer coolant at a temperature lower than the freezing temperature of the sulphur-bentonite mixture, feeding the droplets into the coolant, passing the droplets through the coolant for a time sufficient to anneal the droplets into
30 pellets, and removing the annealed pellets from the coolant.

Furthermore, U.S. patent 4,330,319 relates to a process for the production of a urea sulphur fertilizer by mixing urea and molten sulphur to obtain a molten mixture and solidifying the molten mixture to obtain a homogeneous, solid, particulate urea sulphur fertilizer wherein the sulphur has particle sizes of smaller than about 100 micron. The process comprises passing
5 molten urea and molten sulphur through a mixing device at a temperature above the melting points to produce a finely divided sulphur dispersed in urea with the molten sulphur added in amounts sufficient to produce said urea sulphur fertilizer, maintaining a pressure drop across said mixing device of at least about 200 kPa to form a homogenized melt of urea sulphur, and
10 solidifying said homogenized melt by prilling or agglomeration

Moreover, U.S. patent 4,394,150 teaches particles formed by passing a mixture of molten sulphur and a particulate swelling clay through a plurality of orifices and into an aqueous solution of a water soluble electrolyte salt, maintained at a temperature effective to cool the mixture below its solidification temperature.

15 Also, U.S. patent 4,569,859 relates to a process for prilling a mixture of sulphur and bentonite to produce a water-degradable prill by adding dry bentonite in powdered form to molten sulphur at a temperature to provide a molten sulphur-bentonite mixture, forming droplets of the mixture, providing a quenching solution of either sodium chloride, sodium sulphate, potassium chloride or potassium sulphate at a temperature low enough to solidify the sulphur-
20 bentonite mixture,

Yet another particulate sulphur based fertilizer is taught in U.S. patent 5,571,303 comprising a uniform dispersion of sulphur and at least one member selected from the group consisting of ammonium sulfate, ammonium phosphate and mixtures thereof,

Furthermore, U.S. patent 5,599,373 relates to a sulphur-based, chemical soil-corrective or
25 conditioner for agricultural use in the form of pellets, said product containing fly sulphur powder, at least 3% by weight of an inert compound selected from the group consisting of clay, bentonite, kaolin and mixtures thereof, and at least 0.5% by weight of a wetting agent

Other fertilizers , compositions and method of manufacture can be found in U.S. patent 5,599,373 , U.S. patent 5,571,303 and U.S. patent 4,330,319

The present invention alleviates the safety problems associated with handling powdered sulphur and the problem of very slow conversion to sulphate of sulphur benotnite granular sulphur.

5 SUMMARY OF THE INVENTION

Elemental sulphur pellets are provided that disperse into finely divided particles with more than 10% of said particles less than 50 mesh (US Standard Mesh). The particles include from 10% to 99.9% sulphur. Following application of a described pellet, water is allowed to contact the pellet, dispersing it into pieces and thereby delivering finely divided sulphur
10 particles to oxidizing bacteria for timely conversion to plant available sulphate.

An aspect of this invention relates to a water dispersible pellet comprising: micronized elemental sulphur where 80% of the particles are less than 30 microns in size, which disperse into particles upon contact with water.

Another aspect of this invention relates to a water dispersible pellet comprising: micronized
15 sulphur, a binder, a surfactant and a soluble compound providing cations and anions in a moist environment.

Still a further aspect of this invention relates to a water dispersible pellet comprising: micronized elemental sulphur with 80% of particles less than 30 microns in diameter; a binder component present in the amount ranging from 0.05% too 95% by weight of the total
20 dry weight of the pellet; a surfactant in the amount ranging from 0.05% to 10% by weight of the total dry weight of the pellet; a soluble salt present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; bentonite clay in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet, the pellet having a mean particle domain size and a mean crushing strength, all in a form such that within a few
25 minutes of contact with water the pellet disperses into particles with more than 20% of said particles passing through a 50 mesh (US Standard Size) screen.

Yet another aspect of this invention relates to a method of producing a water dispersible pellet comprising: mixing micronized elemental sulphur particles having 80% of the particles less than 30 microns in size with a binder; adding water to provide a moisture content and
30 aggregating the moist mixture into pellets.

DETAILED DESCRIPTION OF THE INVENTION

General

The present invention relates to a method of formulating an elemental sulphur fertilizer pellet with enhanced dispersal properties. The pellet retains its size and shape during handling and application to a desired area. Following application the pellet rapidly disperses on contact with moisture from the treated area itself, from irrigation or from natural precipitation or absorption of moisture from the soil. The dispersion of the pellet allows rapid oxidation of the smaller sulphur particles into plant available sulphate for crop fertility needs. Larger particles oxidize more slowly and provide sulphate throughout the growing season.

The term dispersion in the context of the present invention is intended to mean that the pellet disperses by breaking into numerous smaller finely divided pieces upon contact with water. The ability of the pellet to disperse in water is generally measured in a wet sieve test. The test involves placing 40 grams of the pellets onto a 50 mesh sieve submersed in water at room temperature. The pellets are allowed to imbibe water for 60 seconds then the sieve is gently swirled three revolutions. The pellets are then left soaking for a further 5 minutes at which time the swirling procedure is repeated. Immediately after the second swirling procedure the sieve is removed from the water and all material remaining on the surface of the sieve is washed onto a pre-weighed filter disc in a Buchner funnel. Excess water is removed from the Buchner funnel using light suction. The filter disc and all remaining material is then transferred to a pre-weighed weigh boat and placed in an oven at 70 degrees C until fully dried. The weight of the remaining material is determined and the amount of dispersed material is calculated.

Dispersion

In a preferred embodiment, pellets disperse within 6 minutes into particles with more than 5% less than 50 mesh in size. Preferably, pellets disperse within a few minutes into particles with more than 20% less than 50 mesh in size. Even more preferably, pellets disperse within a few minutes into particles with more than 50% less than 50 mesh in size. Most preferably, pellets disperse within a few minutes into particles with more than 90% less than 50 mesh in size.

Elemental Sulphur

By elemental sulphur, we mean sulphur which consists of essentially sulphur, but which can have impurities or trace amounts in the vicinity of 20% or less.

5 Composition of Pellets

A pellet in the present invention has a micronized sulphur ingredient, a binder component, a surfactant, and a soluble compound providing cations and anions. The micronized sulphur can be produced using a dry milling process, a wet milling process, or by use of high speed mixers. In a preferred embodiment the size of the micronized sulphur is 80% less than 30
10 micron in diameter. Preferably, the size of the micronized sulphur is 80% less than 20 micron in diameter or size. Even more preferably, the size of the micronized sulphur is 80% less than 10 micron in diameter. Most preferably, the size of the micronized sulphur is 80% less than 5 micron in diameter.

In a preferred embodiment the pellets contain a binder that produces or promotes cohesion of
15 the micronized elemental sulphur. The binder component is present in amounts ranging from 0.05% to 95% by weight of the total dry weight of the pellet. Preferably, the binder component is present in amounts ranging from 0.05% to 50% by weight of the total dry weight of the pellet. More preferably, the binder component is present in amounts ranging from 0.05% to 25% by weight of the total dry weight of the pellet. Most preferably, the
20 binder component is present in amounts ranging from 0.05% to 5% by weight of the total dry weight of the pellet. Examples of suitable binders are carbohydrates such as monosaccharides, disaccharides, oligosaccharides, and polysaccharides; glycolipids; glycoproteins; lipids; proteins; lipoproteins, and combinations and derivations thereof. Specific carbohydrate binders illustratively include arabinose, glucose, fructose, galactose,
25 lactose, maltose, mannose, sucrose, trehalose, xylose, and mixtures thereof such as corn syrup; celluloses such as carboxymethylcellulose, ethylcellulose, hydroxyethylcellulose, hydroxymethylethylcellulose, hydroxyethylpropylcellulose, methylhydroxyethylcellulose, methylcellulose; starches such as amylose, seagel, starch acetates, starch hydroxyethyl ethers, ionic starches, long-chain alkyl starches, dextrans, amine starches, phosphates starches, and
30 dialdehyde starches; plant starches such as corn starch and potato starch; other carbohydrates such as pectin, amylopectin, xylan, glycogen, agar, alginic acid, phycocolloids, chitin, gum

arabic, guar gum, gum karaya, gum tragacanth and locust bean gum; complex organic substances such as lignin and nitrolignin; derivatives of lignin such as lignosulfonate salts illustratively including calcium lignosulfonate, ammonium lignosulfonate and sodium lignosulfonate and complex carbohydrate-based compositions containing organic and inorganic ingredients such as molasses. Suitable protein binders illustratively include soy extract, zein, protamine, collagen, and casein. Binders operative herein also include synthetic organic polymers capable of promoting or producing cohesion of micronized sulphur and these illustratively include ethylene oxide polymers, polyacrylamides, polyacrylates, polyvinyl pyrrolidone, polyethylene glycol, polyvinyl alcohol, polyvinylmethyl ether, polyvinyl acrylates, polylactic acid, and latex. In a preferred embodiment, the binder is ammonium lignosulfonate, calcium lignosulfonate, sodium lignosulfonate or a combination thereof.

In another embodiment the pellets contain a surfactant that promotes wetting and dispersion of the micronized elemental sulphur. The surfactant component is present in amounts ranging from 0.05% to 10% by weight of the total dry weight of the pellet. Preferably, the surfactant is present in amounts ranging from 0.05% to 5% by weight of the total dry weight of the pellet. More preferably, the surfactant is present in amounts ranging from 0.05% to 1% by weight of the total dry weight of the pellet. Most preferably, the surfactant is present in amounts ranging from 0.05% to 0.5% by weight of the total dry weight of the pellet. Examples of suitable surfactants are alkylbenzene sulfonates, sodium alpha olefin sulfonate, sodium dioctyl sulfosuccinate, sodium alkylnaphthalenesulfonates, sodium alkylnaphthalenesulfonate condensates, nonyl phenol ethoxylates, carboxylates, and phosphate esters.

In another embodiment the pellets contain a soluble salt that provides anions and cations and promotes dispersion of the micronized elemental sulphur. The soluble salt is present in amounts ranging from 0.05% to 95% by weight of the total dry weight of the pellet. Preferably, the soluble salt is present in amounts ranging from 0.05% to 50% by weight of the total dry weight of the pellet. More preferably, the soluble salt is present in amounts ranging from 0.05% to 25% by weight of the total dry weight of the pellet. Even more preferably, the soluble salt is present in amounts ranging from 0.05% to 10% by weight of the total dry weight of the pellet. Most preferably, the soluble salt is present in amounts ranging from 0.05% to 5% by weight of the total dry weight of the pellet. Specific soluble salts

illustratively include but are not limited to, ammonium chloride, ammonium sulfate, calcium chloride, calcium sulfate, iron sulfate, magnesium chloride, magnesium sulphate, potassium chloride, potassium sulfate, and sodium chloride.

In another embodiment the pellets contain bentonite clay that swells when wetted and promotes dispersion of the micronized elemental sulphur. The bentonite clay is present in amounts ranging from 0.05% to 95% by weight of the total dry weight of the pellet. Preferably, the bentonite clay is present in amounts ranging from 0.05% to 50% by weight of the total dry weight of the pellet. More preferably, the bentonite clay is present in amounts ranging from 0.05% to 25% by weight of the total dry weight of the pellet. Even more preferably, the bentonite clay is present in amounts ranging from 0.05% to 10% by weight of the total dry weight of the pellet. Most preferably, the bentonite clay is present in amounts ranging from 0.05% to 5% by weight of the total dry weight of the pellet.

Method of Making Pellets

In a preferred embodiment, micronized elemental sulphur is mixed with the binder, and or surfactant, and or soluble salt, and or bentonite clay. Water is then added to the mixture to provide a moisture content ranging from 5% to 25% on a dry weight basis. In a preferred embodiment the moisture content ranges from 10% to 20% by weight of the total dry weight. In the most preferred embodiment, the moisture content ranges from 15% to 20% by weight of the total dry weight. The mixture is then mechanically aggregated into pellets using a pellet press (pellet mill). Illustrative examples of suitable pellet presses are the Kahl Pellet Mill and the California Pellet Press. Alternatively, the binder can be omitted from the mixing step and agglomeration can be achieved using a pan or drum granulator in the presence of the binder. In this embodiment, the binder is sprayed into the pan or drum granulator with the micronized sulphur mixture. The particles are dried and the resulting particles are size-screened and particles of desired size are stored. Optionally, the particles are transferred to a coating drum for addition of a conditioner material.

Various means of drying the material are available. Preferred methods are fluid bed drying, tray drying, or rotary drum drying, however, other forms of drying equipment may be utilized. The material is placed in a drier and the drier inlet air temperature ranges from about 50 degrees C to 100 degrees C. More preferably, the temperature ranges from 60 degrees C to 70 degrees C. Further methods of drying particles will be apparent to one of skill in the art

and illustratively include drying under vacuum conditions or with the use of infra-red heat or micro-waves.

Pellet Size

The pellets of the present invention have a mean particle domain size that ranges from 0.4 millimeter to 15 millimeters. More preferably, the mean particle domain size ranges from 0.6 millimeter to 10 millimeters. Still more preferably, the mean particle domain size ranges from 0.8 millimeter to 5 millimeters. The pellets formed by the process of the present invention have a Uniformity Index rating in the range of 30 to 95 where the Uniformity Index rating is calculated as the 10th percentile particle size expressed as a percentage of the 95th percentile particle size. More preferably, the Uniformity Index rating ranges from 60 to 90.

Pellet Shape

Pellets of the present invention take any shape illustratively including spheres, cylinders, ellipses, rods, cones, discs, needles and irregular. In a preferred embodiment the pellets are approximately spherical.

15 Pellet Crushing Strength

Pellets of the present invention have a crushing strength that ranges from 1.4 kg per pellet to 8 kg per pellet. Preferably the crushing strength ranges from 2.2 kg per pellet to 8 kg per pellet.

20

25

CLAIMS

What is claimed is:

1. A water dispersible pellet comprising: micronized elemental sulphur particles where
5 80% of particles are less than 30 microns in size which disperse into particles upon contact with water.
2. A water dispersible pellet comprising: micronized sulphur; a binder or surfactant, and a soluble compound providing cations and anions in a moist environment.
3. A water dispersible pellet comprising: micronized elemental sulphur with 80% of
10 particles less than 30 micron in diameter; a binder component present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; a surfactant in the amount ranging from 0.05% to 10% by weight of the total dry weight of the pellet; a soluble salt present in the amount ranging from 0.05% to 95% by weight of the total dry weight of the pellet; bentonite clay in the amount ranging from
15 0.05% to 95% by weight of the total dry weight of the pellet, the pellet having a mean particle domain size and a mean crushing strength, all in a form such that within a few minutes of contact with water the pellet disperses into particles with more than 10% of said particles passing through a 50 mesh (US Standard Size) screen.
4. The pellet of claim 3 wherein the elemental sulphur ingredient is present in an amount
20 ranging from 1% to 99.9% of the weight of the total dry weight of the pellet.
5. The pellet of claim 3 wherein the elemental sulphur ingredient is micronized with a process of dry milling, wet milling, or high speed mixing.
6. The pellet of claim 3 wherein the binder ingredient is present in an amount ranging from 0.05% to 5% by weight of the total dry weight of the pellet.
- 25 7. The pellet of claim 3 wherein the surfactant ingredient is present in an amount ranging from 0.05% to 0.5% by weight of the total dry weight of the pellet.
8. The pellet of claim 3 wherein the soluble salt ingredient is present in an amount ranging from 0.05% to 5% by weight of the total dry weight of the pellet.

9. The pellet of claim 3 wherein the bentonite clay ingredient is present in an amount ranging from 0.05% to 5% by weight of the total dry weight of the pellet.
10. The pellet of claim 3 wherein the binder ingredient is selected from the group consisting of: carbohydrate, protein, lipid, synthetic polymer, glycolipid, glycoprotein,
5 lipoprotein, lignin, a lignin derivative, a carbohydrate-based composition, and a combination thereof.
11. The pellet of claim 3 wherein the binder ingredient is ammonium lignosulfonate, calcium lignosulfonate, sodium lignosulfonate or a combination thereof.
12. The pellet of claim 3 wherein the surfactant ingredient is selected from the group
10 consisting of: alkylbenzene sulfonates, sodium alpha olefin sulfonate, sodium dioctyl sulfosuccinate, sodium alkylnaphthalenesulfonates, sodium alkylnaphthalenesulfonate condensates, nonyl phenol ethoxylates, carboxylates, and phosphate esters.
13. The pellet of claim 3 wherein the soluble salt ingredient is selected from the group
15 consisting of: ammonium chloride, ammonium sulfate, calcium chloride, calcium sulfate, iron sulfate, magnesium chloride, magnesium sulphate, potassium chloride, potassium sulfate, and sodium chloride.
14. The pellet of claim 3 wherein the mean particle domain size ranges from 0.4 mm to 15 mm.
15. The pellet of claim 3 wherein the mean particle domain size ranges from 0.8 mm to 5
20 mm.
16. The pellet of claim 3 wherein the Uniformity Index ranges from 30 to 95.
17. The pellet of claim 3 wherein the Uniformity Index ranges from 60 to 90.
18. The pellet of claim 3 wherein the mean crushing strength ranges from 1.4 kg per pellet to 8 kg per pellet.
- 25 19. The pellet of claim 3 wherein the mean crushing strength ranges from 2.2 kg per pellet to 8 kg per pellet.

20. The pellet of claim 3 wherein the pellets are produced by using a pellet press, pan granulator or drum granulator.
21. The pellet of claim 3 wherein the pellets are formed using a pre-pelleting ingredient mixture with a moisture content ranging from 5% to 20% by weight of the total dry weight.
22. The pellet of claim 3 wherein the pellets are formed using a pre-pelleting ingredient mixture with a moisture content ranging from 10% to 20% by weight of the total dry weight.
23. The pellet of claim 3 wherein the green pellets are dried at a temperature ranging from 50 degrees C to 100 degrees C.
24. The pellet of claim 3 wherein the green pellets are dried at a temperature ranging from 60 degrees C to 70 degrees C.
25. A method of producing a water dispersible pellet comprising:
- (a) mixing micronized elemental sulphur particles with a binder, the elemental sulphur having 80% of the particles less than 30 microns in size;
 - (b) adding water to provide a moisture content;
 - (c) aggregating the moist mixture into pellets.
26. A method as claimed in claim 25 further drying the aggregated pellets.
27. A method as claimed in claim 26 where the pellets are sized from 0.6 to 10 millimeters.