(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
17 December 2020 (17.12.2020)

WIPO | PCT

(10) International Publication Number
WO 2020/250137 A1

(51) International Patent Classification:
A01N 25/12 (2006.01) *C05G 3/70* (2020.01)
A01N 25/30 (2006.01) *C05D 3/00* (2006.01)
A01N 59/02 (2006.01) *C05G 5/00* (2020.01)
C05G 3/00 (2020.01) *C05G 5/10* (2020.01)
C05G 3/50 (2020.01)

(21) International Application Number:
PCT/IB2020/055435

(22) International Filing Date:
10 June 2020 (10.06.2020)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
201921023332 12 June 2019 (12.06.2019) IN

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:
— *as to the identity of the inventor (Rule 4.17(i))*
— *of inventorship (Rule 4.17(iv))*

Published:
— *with international search report (Art. 21(3))*
— *in black and white; the international application as filed contained color or greyscale and is available for download from PATENTSCOPE*

WO 2020/250137 A1

(54) Title: PROCESS FOR PREPARATION OF WATER DISPERSIBLE FERTILIZER COMPOSITION AND COMPOSITIONS IN POWDER FORM MADE THEREFROM

(57) Abstract: The present disclosure relates to a process for the preparation of a water dispersible fertilizer composition in the form of a powder. The process of the present disclosure is simple, efficient, economical, less time consuming and easy to scale up. The present disclosure further relates to a water dispersible fertilizer composition in the form of a powder that contains micronized elemental sulphur. Micronized elemental sulphur oxidizes faster and has a better dispersibility as compared to bulk sulphur powder. The particle size of the water dispersible fertilizer composition in the form of a powder is less than 90 microns. Further, the particle size of the sulphur particles, when the water dispersible fertilizer composition is dispersed in water, is in the range of 15 microns to 25 microns. The water dispersible fertilizer composition in the form of a powder provides a stable dispersion.

PROCESS FOR PREPARATION OF WATER DISPERSIBLE FERTILIZER COMPOSITION AND COMPOSITIONS IN POWDER FORM MADE THEREFROM

FIELD

The present disclosure relates to a process for the preparation of a water dispersible fertilizer composition and compositions in powder form made there from.

BACKGROUND

The background information herein below relates to the present disclosure but is not necessarily prior art.

Sulphur (S) is considered as an essential plant nutrient as it has a major influence on crop production in agriculture. Sulphur is absorbed by plants in the form of sulphates. Sulphates are released by volcanic eruption and fossil fuels, which are further deposited in the soil through rainwater. However, due to the efforts for clean air, fossil fuels are made free from sulphates, resulting in an overall reduction in the sulphate input to the soil and hence in the plants. Excessive cultivation also causes overconsumption of the Sulphur available in the soil. Sulphur deficiency causes serious defects in crop production volume and crop quality. The resulting deficiency of Sulphur in the plants necessitates its supplementation through addition of fertilizers to the soil.

Direct application of sulphate to the soil is less productive as it is highly soluble in water and gets leached away before being absorbed by the plants. An alternative is to use elemental sulphur which is insoluble in water. The insoluble elemental sulphur gets oxidized to form sulphates due to bacterial activity in the soil and is subsequently absorbed by the plants. For the bacteria to act on the elemental sulphur, it is desirable that the sulphur particles should be in the size range of 15 to 40 microns. However, when the size of the particles is less than 25 microns, dispersion of the particles is not affected and is quick. Also, the uptake of the sulphur particles by the plants is not affected. Micronizing elemental sulphur ensures large surface to volume ratio. However, if the particles are below 15 microns, the possibility of the particles getting washed away before oxidization is high, causing loss of the nutrient as well as contamination. Therefore, a balance has to be maintained between bacterial activity and loss of nutrient.

Indian Patent Number 282429 discloses an agricultural composition comprising sulphur ingredient. The agricultural composition is in the form of water dispersible granules having a particle size in the range of 0.1 mm to 2.5 mm. The granules further comprise sulphur particles having a size in the range of 2 microns to 12 microns. However, the production method requires sulphur in a slurry form as a starting material. To obtain sulphur as a slurry, it is to be powdered to a greater extent from the generally available prilled form. Hence, in essence, a two stage size reduction process is involved here which is expensive. Also, when these granules are dispersed in water there is considerable foaming and micro bubbles are seen. Also, the granules take a long time for dispersing in water so that an imperfect suspension / dispersion is formed. The large size of the granules also causes choking in the nozzles of drip irrigation pipe. Also, because of the small size of the sulphur particles, there is considerable leaching of the particles which gets washed away causing loss of the nutrient.

There is, therefore, felt a need for a simple and economical process for preparation of a fertilizer composition and a fertilizer composition made therefrom that mitigates the drawbacks mentioned hereinabove.

OBJECTS

Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows:

It is an object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

An object of the present disclosure is to provide a process for the preparation of a water dispersible fertilizer composition containing micronized elemental sulphur.

Another object of the present disclosure is to provide a process for the preparation of a water dispersible fertilizer composition that is economical.

Still another object of the present disclosure is to provide a water dispersible fertilizer composition in the form of a powder.

Yet another object of the present disclosure is to provide a sulphur fertilizer composition that overcomes dissolution issues related to larger granular size when applied to soil directly.

Other objects and advantages of the present disclosure will be more apparent from the following description, which is not intended to limit the scope of the present disclosure.

SUMMARY

The present disclosure relates to a process for preparation of a water dispersible fertilizer composition in the form of a powder. The process comprises mixing sulphur prills with at least one filler material, at least one dispersing agent and at least one wetting agent, in a fluid medium, to obtain a first mixture. The first mixture is aged, under continuous stirring, for a predetermined period of time. A defoamer is added to the first mixture to obtain a second mixture. The second mixture is then ground to obtain a wet ground mixture. Subsequently, the wet ground mixture is dried at a predetermined temperature to obtain a final mixture. The final mixture is sieved through at least one sieve to obtain the water dispersible fertilizer composition in the form of a powder, wherein the particle size of the water dispersible fertilizer composition is up to 90 microns.

If the particle size of the composition is greater than 90 microns, there is a possibility of agglutination and non-dispersion. When the powder particles are greater than 90 microns, the dispersion of the particles in water is slower. Secondly, if the particles remain undispersed there is a possibility of choking the nozzles of the drip irrigation. Therefore the mixture is sieved to ensure the particle size is up to 90 microns and the larger particles that remain in the sieve are returned to the first mixture.

The present disclosure further relates to a water dispersible fertilizer composition in the form of a powder prepared in accordance with the aforementioned process. The water dispersible fertilizer composition in the form of a powder comprises micronized elemental Sulphur in an amount in the range of 90% to 93% of the total weight of the composition; at least one filler material in an amount in the range of 3% to 8% of the total weight of the composition; at least one dispersing agent in an amount in the range of 1% to 8% of the total weight of the composition; at least one wetting agent in an amount in the range of 0.1% to 2% of the total weight of the composition; and at least one defoamer in an amount in the range of 0.005% to 1% of the total weight of the composition, wherein the particle size of the water dispersible fertilizer composition in the form of a powder is below 90 microns; and wherein the particle size of the sulphur particles, when the water dispersible fertilizer composition is dispersed in water, is in the range of 15 microns to 25 microns.

Further, the suspensibility of the existing Sulphur particles is observed to be in the range of 40% to 45 %, however for the water dispersible fertilizer composition in the form of a powder in accordance with this disclosure it is in the range of 50% to 85%.

Additionally, the moisture content of the water dispersible fertilizer composition in the form of a powder is in the range of 0.5% to 1% of the total weight of the composition.

BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWING

The present disclosure will now be described with the help of the accompanying drawing, in which:

Figure 1 illustrates the microscopic image of the Sulphur particles of the water dispersible fertilizer composition not containing defoamer;

Figure 2 illustrates the microscopic image of the Sulphur particles of the water dispersible fertilizer composition in the form of powder prepared in accordance with the present disclosure;

Figure 3a illustrates the photograph of groundnut crops after 60 days of field trials by using a commercially available fertilizer (NPK);

Figure 3b illustrates the photograph of groundnut crops after 60 days of field trials by using a mixture of commercially available fertilizer (NPK) and water dispersible fertilizer composition in the form of powder in accordance with the present disclosure; and

Figure 3c illustrates the photograph of groundnut crops after 60 days of field trials by using a mixture of commercially available fertilizer (NPK) and water dispersible fertilizer in the form of granules.

DETAILED DESCRIPTION

Embodiments of the present disclosure will now be described with reference to the accompanying drawing.

Embodiments are provided so as to thoroughly and fully convey the scope of the present disclosure to the person skilled in the art. Numerous details are set forth, relating to specific components, and methods, to provide a complete understanding of embodiments of the present disclosure. It will be apparent to the person skilled in the art that the details provided in the embodiments should not be construed to limit the scope of the present disclosure. In some embodiments, well-known processes, well-known apparatus structures, and well-known techniques are not described in detail.

The terminology used, in the present disclosure, is only for the purpose of explaining a particular embodiment and such terminology shall not be considered to limit the scope of the present disclosure. As used in the present disclosure, the forms "a," "an," and "the" may be intended to include the plural forms as well, unless the context clearly suggests otherwise.

The terms "comprises," "comprising," "including," and "having," are open ended transitional phrases and therefore specify the presence of stated features, integers, steps, operations, elements, modules, units and/or components, but do not forbid the presence or addition of one or more other features, integers, steps, operations, elements, components, and/or groups thereof. The particular order of steps disclosed in the method and process of the present disclosure is not to be construed as necessarily requiring their performance as described or illustrated. It is also to be understood that additional or alternative steps may be employed.
The terms first, second, third, etc., should not be construed to limit the scope of the present disclosure as the aforementioned terms may be only used to distinguish one element, component, region, layer or section from another component, region, layer or section. Terms such as first, second, third etc., when used herein do not imply a specific sequence or order unless clearly suggested by the present disclosure.

Plant growth is hindered due to the presence of coarse elemental Sulphur particles. It was found that elemental Sulphur particles of 100 microns size exhibit 10-fold higher oxidation rate than those of size of approximately 1 mm owing to the high surface area to volume ratio. Optimum particle size of Sulphur particles for fertilizer application is reported to be in the range of 15 µm to 40 µm. However, if the particle size of the Sulphur particles is above 25 microns, the dispersion property as well as uptake by the plants is adversely affected.

Conventional methods for the preparation of sulphur containing fertilizer product involve fusing bentonite clay with powdered sulphur. Dry bentonite clay particles are added to molten sulphur, followed by formation of pellets. The main disadvantages of the molten mixing production of sulphur and bentonite are that the sulphur particles produced are big in size and hence produce an unstable dispersion, i.e., display agglomeration and sedimentation over time. The products suitable for direct soil application must have a relatively smaller particle size of the sulphur particles for faster microbial mediated oxidation. Further, a stable dispersion, wherein the dispersed phase maintains a consistent particle size over time and remains in suspension, is another requirement for the oxidation to occur in the required time frame. An advantage of the smaller particle size of the sulphur particles is the fast oxidation for plant absorption. However, the smaller the particle size is, the larger the chances of leaching loss and removal from the vicinity of the plants. Hence an optimum particle size of sulphur is essential for its effectiveness as a fertilizer when applied to soil.

Other conventional methods use a bead mill to grind powdered sulphur, which is mixed with various dispersing agents as well as wetting agents to get the sulphur in a dispersible form. However, the method is highly energy consuming and also requires high set up cost.

Accordingly, there is felt a need for a process for the preparation of a fertilizer product that is economical and efficient as well as a fertilizer product made there from.

The present disclosure envisages a process for preparation of a water dispersible fertilizer composition in the form of a powder and a water dispersible fertilizer composition in the form of a powder made there from.

In an aspect of the present disclosure, there is provided a process for preparation of a water dispersible fertilizer composition in the form of a powder. The process comprises the following steps:

a. mixing, using a first mixing means, sulphur prills with at least one filler material, at least one dispersing agent and at least one wetting agent, in a fluid medium, to obtain a first mixture;
b. aging, under continuous stirring, the first mixture for a predetermined period of time;
c. adding a defoamer to the first mixture and mixing using a second mixing means to obtain a second mixture;
d. grinding, using grinding means, the second mixture to obtain a wet ground mixture;
e. drying, using drying means, the wet ground mixture at a predetermined temperature to obtain a final mixture; and
f. sieving the final mixture using a sieve to obtain a water dispersible fertilizer composition in the form of a powder,

wherein the particle size of the water dispersible fertilizer composition is up to 90 microns.

The particle size of the water dispersible fertilizer composition is maintained up to 90 microns to mitigate the possibility of agglutination and non-dispersion. When the powder particles are greater than 90 microns, the dispersion of the particles in water is slower. Secondly, if the particles remain undispersed, there is a possibility of choking the nozzles of the drip irrigation. Therefore the mixture is sieved to ensure the particle size is up to 90 microns and the larger particles that remain in the sieve are returned to the first mixture.

Mixing Means:

Any equipment suitable for mixing the material to obtain a uniform mixed mixture. The mixing means referred herein may be a device for mixing of dry or wet or liquid mixture.

Grinding means:

Any equipment suitable for grinding the material to obtain fine particles of the material. The material may be a mixture of two or more materials. The grinding means referred herein may be a device for dry or wet grinding.

Drying means:

Any equipment suitable for drying the material, slurry, solution wet mixture and the like, to evaporate liquid from a wet mixture and provide dry material. The material may be a mixture of two or more materials.

In accordance with an embodiment of the present disclosure, the mixing step and the aging step is carried out in a tank/hopper of a slotted rotor – slotted stator colloidal mill.

In accordance with an embodiment of the present disclosure, the mixing step is carried out at a first predetermined speed, preferably 300 rpm.

In accordance with an exemplary embodiment of the present disclosure, the first mixture is aged for a period of five hours.

In accordance with an embodiment of the present disclosure, the grinding step is carried out using the slotted rotor-slotted stator colloidal mill running at a speed of about 700 rpm.

Just before commencing the grinding step, or immediately after the commencement of the grinding step when micro-bubbles are seen, a defoamer is slowly added until the bubbles disappear. The defoamer cannot be added to the first mixture initially. The defoamer only acts when the micro-bubble formation starts. This can happen during the mixing step when the mill is rotated at about 300 rpm but more so during the grinding step. Therefore, advantageously, the defoamer should be added immediately after commencement of the grinding step when the micro-bubbles are seen.

In accordance with an exemplary embodiment of the present disclosure, the grinding step is carried out for a duration in the range of 30 minutes to 120 minutes.

In accordance with an embodiment of the present disclosure, the grinding step is carried out at a second predetermined speed, preferably 700 rpm.

The ground mixture can be introduced to a spray dryer equipped with an atomizer and co-current hot air flow. Contact air temperature at the point of introduction of the ground

mixture through the atomizer can be kept in the range of 110°C to 130°C. Negative pressure can be maintained inside the spray dryer to ensure intimate contact of the hot air with the atomized sulphur particles to avoid melting. The temperature in the spray dryer can be maintained in the range of 70 to 90 °C in order to ensure complete removal of the fluid medium as well as to prevent steam deposition on the final product.

In accordance with an embodiment of the present disclosure, the drying is carried out using conventionally known spray drying equipment.

In accordance with an embodiment of the present disclosure, the drying is carried out at a temperature in the range of 70°C to 130°C.

In accordance with an exemplary embodiment of the present disclosure, the drying is carried out at a temperature of 120°C.

In accordance with an embodiment of the present disclosure, the sieving step is carried out using a vibrating sieve.

In accordance with an embodiment of the present disclosure, the sulphur prills have a particle size in the range of 1 mm to 10 mm.

In accordance with an exemplary embodiment of the present disclosure, the sulphur prills are reduced to a particle size of 5 mm.

The filler material is used for the purpose of long term stabilization, bulking up the composition, reducing viscosity, increasing the solubility and improving the suspensibility of the water dispersible fertilizer composition.

In accordance with an embodiment of the present disclosure, the filler material is selected from the group consisting of bentonite clay, kaolinite and zeolite.

In accordance with an exemplary embodiment of the present disclosure, the filler material is bentonite clay.

The dispersing agent facilitates the separation of the particles of the composition and prevents settling and/or clumping of the composition.

In accordance with an embodiment of the present disclosure, the dispersing agent can be a single dispersing agent or a mixture of two or more dispersing agents in a predetermined ratio.

In accordance with an embodiment of the present disclosure, the dispersing agent is selected from the group consisting of a copolymer of benzylmethacrylate, a copolymer of acrylic acid, a copolymer of 2-acrylamido-2-methyl propane sulfonic acid, a water soluble nanofibrilated cellulose, a cellulose derivatives, a co-polymer of styrene and a methacrylate and a alkene naphthalene sulphonate.

In accordance with a first exemplary embodiment of the present disclosure, the dispersing agent is a co-polymer of styrene and methacrylate.

In accordance with a second exemplary embodiment of the present disclosure, the dispersing agent is a mixture of:

a. a co-polymer of styrene and methacrylate; and
b. alkene naphthalene sulphonate,

wherein the ratio of (a) to (b) is in the range of 1:1 to 1:5.

Sulphur as such is not wettable in water. In order to wet-grind sulphur, it needs to be made wet; hence addition of a wetting agent is necessary. The wetting agent, by using hydrophilic and hydrophobic parts of its chemical structure, promotes spreading of the water on the surface of the sulphur prills by reducing the surface tension between the water and the sulphur prills.

In accordance with an embodiment of the present disclosure, the wetting agent is selected from the group consisting of alpha olefin sulphonate, sodium salt of alkene (C_{12}-C_{14}) hydroxyl sulphonic acid, alkenesulphonic acid and derivatives of alkene sulphonic acid.

In accordance with an exemplary embodiment of the present disclosure, the wetting agent is alpha olefin sulphonate.

The aging step is important since the filler used, for e.g., bentonite clay, has a swelling property. It can absorb water and can swell up to 40 % of its initial volume. In the initial mix when bentonite is used as the filler material, the aging step is included so that the bentonite is completely swollen. This will increase grindability and homogeneity of the mixture. Since the aging process includes continuous stirring also, proper mixing of ingredients with swollen bentonite also occurs. The swollen bentonite can be fine ground better.

Dry grinding of sulphur poses hazards of fire as well as creation of dust. Though the flash point of sulphur is 207°C, when dry ground, sulphur powder can catch fire at approximately 190°C. Development of static charges during dry grinding of sulphur also may lead to self-ignition. Hence a suitable fluid medium is used while grinding sulphur.

In accordance with an embodiment of the present disclosure, the ratio of the sulphur prills to the fluid medium is in the range of 1:0.1 to 1:2.

In accordance with an exemplary embodiment of the present disclosure, the fluid medium is water.

The presence of a defoamer prevents the formation of foam/bubbles during the process of wet grinding. Particularly, the micro-bubbles, if formed, create a fluffy material with very low density which results in a considerable increase in the volume of the material being ground, causing incomplete mixing and grinding. The formation of fluffy material with very low density will also lead to the requirement of large volume mixing vessels and storage vessels, thereby reducing the throughput of the process. Addition of the defoamer prevents the formation of bubbles during the grinding process. Hence, the volume and the density remain the same throughout the grinding process resulting in better mixing and grinding during the wet grinding process.

Addition of the defoamer is also advantageous during the end use of the water dispersible fertilizer composition in the form of a powder. The presence of the defoamer prevents foam formation at the time of dispersion of the water dispersible fertilizer composition in water at the time of application. This will help for easy application and uniform distribution of the composition in the field.

In accordance with an embodiment of the present disclosure, the defoamer is selected from the group consisting of silicone based defoamer, silicone polymeric defoamer, dioxosilane, amorphous silica, hydrolyzed silica, poly(dimethylsiloxane), poly (ethylene oxide)block copolymer, poly[dimethylsiloxane-co-ethyl(3-hydroxypropyl)siloxane] – graft – poly (ethylene glycol) methyl ether, silicone glycol block copolymer and emulsified silicone oil.

In accordance with an exemplary embodiment of the present disclosure, the defoamer is a silicone polymeric defoamer.

In accordance with an embodiment of the present disclosure, the particle size of the water dispersible fertilizer composition in the form of a powder is kept below 90 microns.

In accordance with an exemplary embodiment of the present disclosure, the particle size of the water dispersible fertilizer composition in the form of a powder is 90 microns and below, with a majority of the particles having a particle size of 80 microns or less.

In accordance with another aspect of the present disclosure, there is provided a water dispersible fertilizer composition in the form of a powder prepared in accordance with the aforementioned process, the composition comprising:

1. micronized elemental sulphur in an amount in the range of 90% to 93% of the total weight of the composition;

2. at least one filler material in an amount in the range of 3% to 8% of the total weight of the composition;
3. at least one dispersing agent in an amount in the range of 1% to 8% of the total weight of the composition;
4. at least one wetting agent in an amount in the range of 0.1% to 2% of the total weight of the composition; and
5. at least one defoamer in an amount in the range of 0.005% to 1% of the total weight of the composition,

wherein the particle size of the water dispersible fertilizer composition in the form of a powder is up to 90 microns; and

wherein the particle size of the sulphur particles, when the water dispersible fertilizer composition is dispersed in water, is in the range of 15 microns to 25 microns.

In accordance with an embodiment of the present disclosure, the suspensibility of the water dispersible fertilizer composition in the form of a powder is in the range of 50% to 85%.

In accordance with an embodiment of the present disclosure, the moisture content of the water dispersible fertilizer composition in the form of a powder is in the range of up to 0.5% to 1% of the total weight of the composition.

In accordance with an embodiment of the present disclosure, the filler material is selected from the group consisting of bentonite clay, kaolinite and zeolite.

In accordance with an exemplary embodiment of the present disclosure, the filler material is bentonite clay.

When the water dispersible fertilizer composition is dispersed in water, the dispersing agents helps the sulphur particles to remain dispersed in water, even though the size of the sulphur particles is above 14 microns.

In accordance with an embodiment of the present disclosure, the dispersing agent may be a single dispersing agent or a combination of two or more dispersing agents in a predetermined ratio.

In accordance with an embodiment of the present disclosure, the dispersing agent is selected from the group consisting of copolymer of benzylmethacrylate, copolymer of acrylic acid, copolymer of 2-acrylamido-2-methyl propane sulfonic acid, water soluble nanofibrilated cellulose, cellulose derivatives, co-polymer of styrene and methacrylate and alkene naphthalene sulphonate.

In accordance with a first exemplary embodiment of the present disclosure, the dispersing agent is a co-polymer of styrene and methacrylate.

In accordance with a second exemplary embodiment of the present disclosure, the dispersing agent is a mixture of:

a. a co-polymer of styrene and methacrylate; and
b. an alkene naphthalene sulphonate,

wherein the ratio of (a) to (b) is in the range of 1:1 to 1:5.

In accordance with an embodiment of the present disclosure, the wetting agent is selected from the group consisting of alpha olefin sulphonate, sodium salt of alkene (C_{12}-C_{14}) hydroxyl sulphonic acid, alkenesulphonic acid and derivatives of alkene sulphonic acid.

In accordance with an embodiment of the present disclosure, the wetting agent is alpha olefin sulphonate.

In accordance with an embodiment of the present disclosure, the defoamer is selected from the group consisting of silicone based defoamer, silicone polymeric defoamer, dioxosilane, amorphous silica, hydrolyzed silica, poly(dimethylsiloxane), poly (ethylene oxide)block copolymer, poly[dimethylsiloxane-co-ethyl(3-hydroxypropyl)siloxane] – graft – poly (ethylene glycol) methyl ether, silicone glycol block copolymer and emulsified silicone oil.

In accordance with an exemplary embodiment of the present disclosure, the defoamer is a silicone polymeric defoamer.

In accordance with an exemplary embodiment of the present disclosure, the particle size of the water dispersible fertilizer composition in the form of a powder is up to 90 microns, with a majority of the particles having a particle size of 80 microns or less.

In accordance with an embodiment of the present disclosure, the suspensibility of the water dispersible fertilizer composition in the form of a powder is in the range of 50% to 85%.

In accordance with an exemplary embodiment of the present disclosure, the suspensibility of the water dispersible fertilizer composition in the form of a powder is greater than 50% and up to 85%.

In accordance with an embodiment of the present disclosure, the moisture content of the water dispersible fertilizer composition in the form of a powder is in the range of 0.5% to 1% of the total weight of the composition.

In accordance with an exemplary embodiment of the present disclosure, the moisture content of the water dispersible fertilizer composition in the form of a powder is 1% of the total weight of the composition. 1% is the maximum value and it can be any number below 1%

The process of the present disclosure is simple, efficient, economical and less time consuming. The higher moisture content helps overcome processing limitations associated with drying of powders such as dusting and material loss. The water dispersible fertilizer

composition in the form of a powder of the present disclosure contains micronized sulphur which oxidizes faster and has better dispersibility as compared to bulk sulphur powder or sulphur granules.

The water dispersible fertilizer composition in the form of a powder is dispersed in water at the time of application and applied to a plant, a locus of a plant or a part of a plant. The water dispersible fertilizer composition in the form of a powder can also be applied directly to the soil, where it uses the water content of the soil to form a dispersion.

The foregoing description of the embodiments has been provided for purposes of illustration and is not intended to limit the scope of the present disclosure. Individual components of a particular embodiment are generally not limited to that particular embodiment, but, are interchangeable. Such variations are not to be regarded as a departure from the present disclosure, and all such modifications are considered to be within the scope of the present disclosure.

The present disclosure is further described in light of the following experiments which are set forth for illustration purposes only and not to be construed for limiting the scope of the disclosure. The following experiments can be scaled up to industrial/commercial scale and the results obtained can be extrapolated to industrial scale.

EXPERIMENTAL DETAILS

1. PROCESS FOR PREPARATION OF WATER DISPERSIBLE FERTILIZER COMPOSITION IN THE FORM OF POWDER

a. EFFECT OF USE OF DEFOAMER

Comparative Example 1:

30 kg sulphur, 1.48 kg bentonite, 52 L of water, 0.85 kg dispersing agent and 0.3 kg wetting agent were mixed in the hopper of a slotted rotor – slotted stator colloidal mill to obtain a first mixture. The first mixture was mixed at 300 rpm and aged for a period of five hours. The first mixture was then ground in the mill rotating at a speed of 700 rpm in an attempt to obtain a wet ground mixture. Microbubble formation started and volume started to increase considerably. The calculated density of the mixture before bubbling was 1.4 g/cc. However, due to increase in volume, density of the ground mixture reduced to 0.5 g/cc. The mixture also became less flow able. Huge increase in its volume due to formation of bubbles was

observed. Further processing had to stop as there was considerably low flowability as well as overflow due to bubble formation.

Example 1:

30 kg sulphur, 1.48 kg bentonite, 52 L of water, 0.85 kg dispersing agent and 0.3 kg wetting agent were mixed in the hopper of a slotted rotor – slotted stator colloidal mill to obtain a first mixture. The first mixture was mixed at 300 rpm and aged for a period of five hours. Thereafter the first mixture was taken for grinding in the mill rotating at 700 rpm. Microbubble formation started and the volume started to increase considerably. Immediately, 200g of silicone polymeric defoamer (HPAF 6285 from Resil Chemicals Pvt. Ltd) was added slowly to the first mixture to obtain a second mixture. The deformer was added slowly. Grinding was continued for 30 minutes. The bubbles did not reappear and the volume decreased. Flowability became normal and the density was retained at 1.4 g/cc and a wet ground mixture was obtained.

The wet ground mixture collected after grinding was then fed to a spray dryer at 120 °C drying chamber temperature at the contact point and 75°C temperature at chamber exit to obtain the final mixture in the form of a powder. The final mixture was sieved through a 90 micron sieve to obtain the water dispersible fertilizer composition in the form of a powder and the particles having a particle size above 90 microns were returned to the step of preparation of the first mixture.

From comparative example 1 and example 1, it can be concluded that addition of a defoamer is an important step of the process disclosed herein since it prevents bubble or foam formation while grinding and makes the process efficient and economical.

Comparative Example 2:

To the hopper of a colloidal mill, 1 L tap water was added and the mill was started in recirculation mode. Material from the hopper was directly fed to a slotted rotor – slotted stator milling assembly of 500 ml milling volume and the outlet was fed back to the hopper. To the water in the hopper, 9 g of a dispersant (Tersperse 2700), 455 g of sulphur prills of size 5 mm, 5 g of wetting agent (Terwet 1004) and 31 g of bentonite clay were added and blended at 300 rpm to obtain a first mixture. The first mixture was aged for a period of five hours thereafter and wet milling was started by increasing the rotation of the mill to 700 rpm. Within a few seconds, the 7 L hopper was filled with bubbles, although milling was

continued. Flow rate of the mixture being ground reduced as micro-bubbling increased. After 1 hour, the ground material was collected from the mill through the outlet into a container and then fed to a spray chamber where the flash - contact temperature was kept at 120°C. Dried material was collected and analyzed for moisture, elemental sulphur content and suspensibility. The dried product was made into a suspension in 1:500 solid to water proportion. The suspension prepared exhibited bubbles on the top, making it difficult to handle. The images of the sulphur particles were recorded on a microscope and it was seen that the particle size of the sulphur particles was above 25 microns (Figure 1).

Example 2:

To 2 L of tap water in the hopper of a slotted rotor – slotted stator colloidal mill, 15 g of a dispersant (Tersperse 2700) and 3g of a wetting agent (Terwet 1004) were added. To the liquid in the hopper, 910 g sulphur prills of size 5 mm and 63 g of bentonite clay were added slowly and mixed at 300 rpm to obtain a first mixture. The first mixture was aged for a period of five hours thereafter and wet milling was started by increasing the rotation of the mill to 700 rpm. Immediately when the bubbling started and the volume of the mixture being ground started to increase in the hopper, 0.1 g of concentrated defoamer (amorphous silica based) was added slowly. It was observed that the bubbling stopped within 15 seconds. Grinding was continued. The material collected after grinding was then fed to a spray dryer at 120°C drying chamber temperature at the contact point and 75°C temperature at chamber exit. Dried material was collected and analyzed for moisture, elemental sulphur content and suspensibility. The dried product was made into a suspension in 1:500 solid to water proportion. The images of the sulphur particles were recorded on a microscope and it was seen that the particle size of the sulphur particles was below 25 microns (Figure 2).

As illustrated in Figures 1 and 2, the absence of a defoamer (as in comparative example 2) has a detrimental effect on the particle size of the sulphur particles, when the water dispersible fertilizer composition was dispersed in water. The particle size of the sulphur particles, when the composition prepared in accordance with comparative example 2 was dispersed in water, is 26.52 microns. On the contrary, the particle size of the sulphur particles, when the composition was prepared in accordance with example 2 and is dispersed in water, is on an average between 17.76 microns and 20.84 microns.

Comparative Example 3:

In a laboratory scale slotted rotor – slotted stator mill, 100 ml water was added followed by 182g sulphur prills of size 5 mm. 15 g of a mix of dispersing agents (Tersperse 2700 and Tersperse 2020) was then added to the mill followed by 3g wetting agent (Terwet 1004) to form a mixture. No filler was added to this mixture. This mixture was treated in accordance with the treatment given to the first mixture in Examples 1 & 2 in terms of blending, aging and grinding. 0.1g of defoamer was added slowly to see that all the foam formed disappeared. The mixture was ground for 30 minutes to obtain a smooth free flowing composition, which was then transferred to a tray – dryer kept at 80°C overnight to obtain a water dispersible fertilizer composition in the form of a powder. The composition after drying was collected for analysis.

2. PHYSICOCHEMICAL CHARACTERIZATION OF WATER DISPERSIBLE FERTILIZER COMPOSITION IN THE FORM OF A POWDER

The compositions prepared in accordance with Example 2, Comparative Example 2 and Comparative Example 3 were drawn and analyzed for the sulphur content, moisture content and suspensibility.

Sulphur content measurement:

The method described in Fertilizer Control Order (1985) to determine elemental sulphur was followed. The pre-weighed sample aliquot was treated with 4N HCl and then filtered. The residue was washed with distilled water first and then washing was repeated with sulphur–saturated acetone 4 – 5 times. The washed residue was then dried and weighed and then washed repeatedly with carbon disulphide to dissolve and remove all the elemental sulphur. The washed residue was dried and then weighed. The difference in weights before and after removal of sulphur is reported as a percentage sulphur content.

Moisture content measurement:

The method was adopted from the analytical procedures described in Fertilizer Control Order (1985). A pre-weighed sample was kept in a convection or forced draft oven for a specified time and temperature (e.g. 2 hours at 105^{o}C) and its dried mass was determined. Difference in loss on drying is expressed as percentage moisture content.

Suspensibility:

A procedure detailed in IS: 6940 – 1982 was followed to determine suspensibility of water dispersible fertilizer composition. A weighed sample aliquot was made into a suspension in standard hard water in a graduated measuring cylinder and kept steady for 30 minutes. After 30 minutes, 90 % of the total volume of suspension was removed without disturbing the portion below 90 % volume. The left out 10 % volume material was then dried and weighed. The ratio of the weight of the material that remained in the suspension to the weight of the material that settled down was expressed as suspensibility.

The results obtained were as follows:

Table 1

PHYSICOCHEMICAL CHARACTERISTICS	EXAMPLE 2	COMPARATIVE EXAMPLE 2	COMPARATIVE EXAMPLE 3
Sulphur Content (%)	91	90.5	90.1
Moisture Content (%)	1	0.5	0.9
Suspensibility (%)	>80	40	42

This characterization shows that the filler is essential to maintain the suspensibility greater than 80%.

The suspensibility parameter has an indirect correlation to dispersibility. The greater the suspensibility, the greater is the dispersibility of the final product i.e. sulphur particles in water. A homogeneous dispersion is required for proper administration to the plants.

Product Comparison

100g of the powder in accordance with this disclosure was mixed with 250 ml of water and was stirred. Similarly, 100g of commercially available sulphur granules were added to 250 ml of water and stirred. It was observed that the dispersion of the powder in accordance with the present disclosure was very fast and took about 10 to 15 seconds. In comparison, the dispersion of the granules was slow and even after stirring for 1 minute, complete dispersion did not take place. There was some foaming in the case of the granules and micro bubbles were formed. In the case of the powder, in accordance with this disclosure, no micro bubbles were seen when the powder was mixed with water.

3. FIELD TRIALS ON SUMMER GROUNDNUT GG-2 PLANT:

The water dispersible fertilizer composition in the form of a powder, in accordance with the present disclosure, was applied on the summer groundnut plant of GG-2 variety in the plot size of 12 m^2. The spacing of the plant was chosen to be 60 by 10 cm. The experimental design chosen was randomized block design (RBD). Measurements were averaged over 3 replications (R-I, R-II, R-III).

The comparative study was carried out by using following 3 treatments to the above mentioned groundnut plants:

Treatment 1 (T1): Commercially available fertilizer (NPK)

Treatment 2 (T2): Commercially available fertilizer (NPK) and a water dispersible fertilizer composition in the form of a powder in accordance with the present disclosure.

Treatment 3 (T3): Commercially available fertilizer (NPK) and water dispersible sulphur granules.

Along with the commercially available fertilizer (NPK), a water dispersible fertilizer composition in the form of a powder in accordance with the present disclosure and water dispersible granules were applied at the dose of 20 kg/ha and following parameters were recorded:

1) Plant height at 60 days after sowing
2) Number of days at 50% flowering
3) Number of pods per plant
4) Yield/plot
5) Percentage oil content in the kernels collected.

The Tables 2-6 provided below show the results of the comparative study conducted.

Table 2: Measurement of plant Height (cm) 60 days after the field trials:

Treatments	R-I	R-II	R-III	Average Mean
T1	35	38.4	32.6	35.330
T2	41.2	41.2	39.6	40.667

T3	37.4	38.4	38.4	38.067

Table 3: Measurement of number of days to 50% flowering after the field trials:

Treatments	R-I	R-II	R-III	Average
T1	45	46	46	45.67
T2	43	44	45	44.00
T3	42	44	43	43.00

Table 4: Measurement of number of pods per plant after the field trials:

Treatments	R-I	R-II	R-III	Average
T1	67.2	102	89.4	86.2
T2	108.6	110	101	106.5
T3	96	92.6	94.8	94.5

Table 5: Measurement of the yield /Plot (kg/12 m^2) after the field trials:

Treatments	R-I	R-II	R-III	Average
T1	3.90	6.37	5.90	5.39
T2	6.71	7.20	6.00	6.64
T3	4.20	5.11	7.00	5.44

Tables 2-5 show that the treatment with the mixture of commercially available fertilizer (NPK) and the water dispersible fertilizer composition in the form of a powder in accordance with the present disclosure indicates good response towards vegetative growth and yield attributing characters as compared to other treatments. (commercially available fertilizer (NPK) alone and combination of commercially available fertilizer (NPK) with water dispersible sulphur granules)

Table 6: Measurement of percentage Oil Content in the kernels collected after the field trials:

Treatments	R-I	R-II	R-III	Average
T1	42.94	41.76	51.9	45.53
T2	46.8	49.18	49.14	48.37
T3	46.08	45.88	50.18	47.38

Table 6 shows that the treatment with the mixture of commercially available fertilizer (NPK) and water dispersible fertilizer composition in the form of a powder in accordance with the present disclosure yields crops with better oil content as compared to other treatments(commercially available fertilizer (NPK) alone and combination of commercially available fertilizer (NPK) with water dispersible sulphur granules).

Further, figures 3a, 3b and 3c show that treatment with a mixture of commercially available fertilizer (NPK) and water dispersible fertilizer composition in accordance with the present disclosure shows better result as compared to other treatments(commercially available fertilizer (NPK) alone and combination of commercially available fertilizer (NPK) with water dispersible sulphur granules).

4. FIELD TRIALS ON MUSTARD PLANT

The water dispersible fertilizer composition in the form of a powder, in accordance with the present disclosure, was applied on the mustard plant. The comparative study was carried out by using following 3 treatments to the above mentioned mustard plants:

Treatment 1 (T1): Recommended Dose of Fertilizer (RDF) comprising Urea & DAP + powder in accordance with the present disclosure

Treatment 2 (T2): Recommended Dose of Fertilizer (RDF) + Commercially Available Sulphur Granules

Treatment 3 (T3): Recommended Dose of Fertilizer (RDF)

T1, T2 and T3 were applied to the mustard plant and the yield was recorded. The products (T1, T2 and T3) were applied to the leaves just before flowering through a spray using a handheld pressurized pump. The products were made into suspensions in the tank of the pump along with water in a 1:10 ratio. In the case of T1, no bubbling occurred during the preparation of the suspension. Also, no choking or settling of particles occurred until the

completion of application of T1 as well. However, bubbling, choking and settling occurred in the case of T2. The yield of the mustard plant was recorded and is tabulated in table 7.

Table 7: Yield of mustard plant

Treatments	Test weight (gm per 1000 seeds)	Yield (Kg/ Hectare)
T_1	4.187	1563
T_2	4.147	1493
T_3	3.834	1210

As illustrated in Table 7, the use of the powder in accordance with the present disclosure along with the recommended dose of fertilizer provides a higher yield as compared to the other treatments.

TECHNICAL ADVANCEMENTS

The present disclosure described herein above has several technical advantages including, but not limited to, the realization of

- a simple, economical, less time consuming process for the preparation of a water dispersible fertilizer composition in the form of a powder;
- a water dispersible fertilizer composition in the form of a powder, having faster and smooth dispersion in water for optimal absorption by plants, with higher yields and faster rate of growth of the plants.
- a water dispersible fertilizer composition characterized by having a particle size below 90 microns; and
- a water dispersible fertilizer composition in the form of a powder, wherein the particle size of the sulphur particles, when the composition is dispersed in water, is in the range of 15 microns to 25 microns.

The embodiments as described herein above, and various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the following description.

Throughout this specification the word "comprise", or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression "at least" or "at least one" suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

The foregoing description of specific embodiments so fully reveal the general nature of the embodiments herein, that others can, by applying current knowledge, readily modify and/or adapt for various applications of such specific embodiments without departing from the generic concept, and therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein. Further, it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

Having described and illustrated the principles of the present disclosure with reference to the described embodiments, it will be recognized that the described embodiments can be modified in arrangement and detail without departing from the scope of such principles.

While considerable emphasis has been placed herein on the particular features of this disclosure, it will be appreciated that various modifications can be made, and that many changes can be made in the preferred embodiment without departing from the principles of the disclosure. These and other modifications in the nature of the disclosure or the preferred embodiments will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

CLAIMS:

1. A process for the preparation of a water dispersible fertilizer composition in the form of a powder, said process comprising the following steps:
   a. mixing, using a first mixing means, sulphur prills with at least one filler material, at least one dispersing agent and at least one wetting agent, in a fluid medium, to obtain a first mixture;
   b. aging, under continuous stirring, the first mixture for a predetermined period of time;
   c. adding a defoamer to the first mixture and mixing using a second mixing means to obtain a second mixture;
   d. grinding, using grinding means, the second mixture to obtain a wet ground mixture;
   e. drying, using drying means, the wet ground mixture at a predetermined temperature to obtain a final mixture; and
   f. sieving the final mixture using at least one sieve to obtain the water dispersible fertilizer composition in the form of a powder,

   wherein the particle size of the water dispersible fertilizer composition is up to 90 microns.

2. The process as claimed in claim 1, wherein the particle size of the sulphur prills is in the range of 1 mm to 10 mm.

3. The process as claimed in claim 1, wherein the particle size of the sulphur prills is 5 mm.

4. The process as claimed in claim 1, wherein the filler material is selected from the group consisting of bentonite clay, kaolinite and zeolite.

5. The process as claimed in claim 1, wherein the filler material is bentonite clay.

6. The process as claimed in claim 1, wherein the dispersing agent is a single dispersing agent or comprises a mixture of two or more dispersing agents in a predetermined ratio.

7. The process as claimed in claim 1, wherein the dispersing agent is selected from the group consisting of copolymer of benzylmethacrylate, copolymer of acrylic acid,

copolymer of 2-acrylamido-2-methyl propane sulfonic acid, water soluble nanofibrilated cellulose, cellulose derivatives, co-polymer of styrene and methacrylate and alkene naphthalene sulphonate.

8. The process as claimed in claim 1, wherein the dispersing agent is a co-polymer of styrene and methacrylate.

9. The process as claimed in claim 1, wherein the dispersing agent is a mixture of:
   a. a co-polymer of styrene and methacrylate; and
   b. alkene naphthalene sulphonate.

10. The process as claimed in claim 9, wherein the ratio of (a) to (b)is in the range of 1:1 to 1:5.

11. The process as claimed in claim 1, wherein the wetting agent is selected from the group consisting of alpha olefin sulphonate, sodium salt of alkene (C_{12}-C_{14}) hydroxyl sulphonic acid, alkenesulphonic acid and derivatives of alkene sulphonic acid.

12. The process as claimed in claim 1, wherein the wetting agent is alpha olefin sulphonate.

13. The process as claimed in claim 1, wherein the fluid medium is water.

14. The process as claimed in claim 1, wherein the defoamer is added slowly before, at the start of or during the grinding process of step (d) until the micro-bubbles formed during mixing or grinding disappear.

15. The process as claimed in claim 1, wherein the defoamer is selected from the group consisting of silicone based defoamer, dioxosilane, amorphous silica, hydrolyzed silica, poly(dimethylsiloxane), poly (ethylene oxide)block copolymer, poly[dimethylsiloxane-co-ethyl(3-hydroxypropyl)siloxane]– graft – poly (ethylene glycol) methyl ether, silicone glycol block copolymer and emulsified silicone oil.

16. The process as claimed in claim 1, wherein the defoamer is a silicone polymeric defoamer.

17. The process as claimed in claim 1, wherein the aging step is carried out for a period of time in the range of 180 minutes to 480 minutes.

18. The process as claimed in claim 1, wherein the milling step is carried out using a slotted rotor-slotted stator assembly.

19. The process as claimed in claim 1, wherein the milling step is carried out for a period of 30 minutes to 120 minutes.

20. The process as claimed in claim 1, wherein the drying step is carried out at a temperature in the range of 70°C to 120°C.

21. The process as claimed in claim 1, wherein the particle size of the water dispersible fertilizer composition in the form of a powder is less than 90 microns.

22. The process as claimed in claim 1, wherein the particle size of the water dispersible fertilizer composition in the form of a powder is less than 90 microns, with a majority of the particles having a particle size of 80 microns or less.

23. A water dispersible fertilizer composition in the form of a powder, the composition comprising:

a. micronized elemental sulphur in an amount in the range of 90% to 93% of the total weight of the composition;
b. at least one filler material in an amount in the range of 3% to 8% of the total weight of the composition;
c. at least one dispersing agent in an amount in the range of 1% to 8% of the total weight of the composition;
d. at least one wetting agent in an amount in the range of 0.1% to 2% of the total weight of the composition; and
e. at least one defoamer in an amount in the range of 0.005% to 1% of the total weight of the composition,

wherein the particle size of the water dispersible fertilizer composition in the form of a powder is less than 90 microns; and

wherein the particle size of the sulphur particles, when the water dispersible fertilizer composition is dispersed in water, is in the range of 15 microns to 25 microns.

24. The composition as claimed in claim 23, wherein the suspensibility of the composition is in the range of 50% to 85%.

25. The composition as claimed in claim 23, wherein the moisture content of the composition is in the range of 0.5% to 1% of the total weight of the composition.

26. The composition as claimed in claim 23, wherein the filler material is selected from the group consisting of bentonite clay, kaolinite and zeolite.

27. The composition as claimed in claim 23, wherein the filler material is bentonite clay.

28. The composition as claimed in claim 23, wherein the dispersing agent is a single dispersing agent or comprises a mixture of two or more dispersing agents in a predetermined ratio.

29. The composition as claimed in claim 23, wherein the dispersing agent is selected from the group consisting of copolymer of benzylmethacrylate, copolymer of acrylic acid, copolymer of 2-acrylamido-2-methyl propane sulfonic acid, water soluble nanofibrilated cellulose, cellulose derivatives, co-polymer of styrene and methacrylate and alkene naphthalene sulphonate.

30. The composition as claimed in claim 23, wherein the dispersing agent is a co-polymer of styrene and methacrylate.

31. The composition as claimed in claim 23, wherein the dispersing agent is a mixture of:
    a. co-polymer of styrene and methacrylate; and
    b. alkene naphthalene sulphonate.

32. The composition as claimed in claim 31, wherein the ratio of (a) to (b) is in the range of 1:1 to 1:5.

33. The composition as claimed in claim 23, wherein the defoamer is selected from the group consisting of silicone based defoamer, dioxosilane, amorphous silica, hydrolyzed silica, poly(dimethylsiloxane), poly (ethylene oxide)block copolymer, poly[dimethylsiloxane-co-ethyl(3-hydroxypropyl)siloxane]– graft – poly (ethylene glycol) methyl ether, silicone glycol block copolymer and emulsified silicone oil.

34. The composition as claimed in claim 23, wherein the defoamer is a silicone polymeric defoamer.

35. The composition as claimed in claim 23, wherein the particle size of the composition is less than 90 microns, with a majority of the particles having a particle size of 80 microns or less.

36. The composition as claimed in claim 23, wherein the composition comprises:
    a. micronized elemental sulphur in an amount in the range of 90% to 93% of the total weight of the composition;
    b. at least one filler material in an amount in the range of 3% to 8% of the total weight of the composition;
    c. a dispersing agent in an amount in the range of 1% to 8% of the total weight of the composition; said dispersing agent comprising:
        i. a first dispersing agent; and
        ii. a second dispersing agent,
        wherein the ratio of the first dispersing agent to the second dispersing agent is in the range of 1:1 to 1:5;
    d. at least one wetting agent in an amount in the range of 0.01% to 2% of the total weight of the composition; and
    e. at least one defoamer in an amount in the range of 0.005% to 1% of the total weight of the composition,

wherein the particle size of the water dispersible fertilizer composition in the form of a powder is less than 90 microns, with a majority of the particles having a particle size of 80 microns or less;

wherein the particle size of the sulphur particles, when the water dispersible fertilizer composition is dispersed in water, is in the range of 15 microns to 25 microns; and

wherein the suspensibility of the water dispersible fertilizer composition in the form of a powder is greater than 50% and up to 85%.

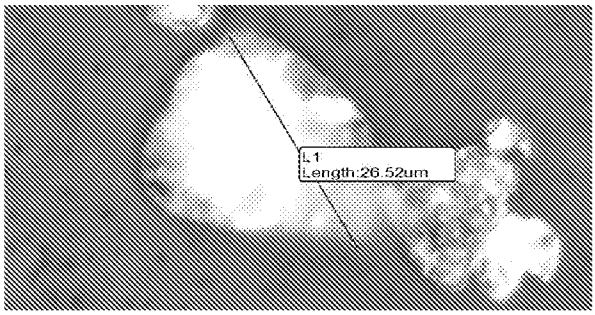

FIGURE 1

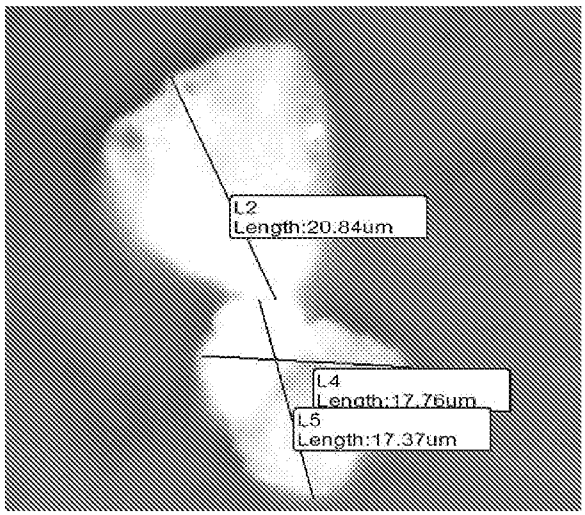

FIGURE 2

Figure 3a

Figure 3b

Figure 3c

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2020/055435

A. CLASSIFICATION OF SUBJECT MATTER
A01N25/12,A01N25/30,A01N59/02,C05G3/00,C05G3/50,C05G3/70,C05D3/00,C05G5/00,C05G5/10 Version=2020.01
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
A01N, C05D, C05G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
TotalPatent One, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO2019102359A1 (RELIANCE INDUSTRIES LIMITED [IN/IN]), 31 May 2019. Abstract, claims 1-10	23-36
A	The whole document	1-22
Y	IN655/MUM/2000A (SULPHUR MILLS LTD [IN]), 04 March 2005. Abstract, page 4, claims 1-9	23-36
A	The whole document	1-22
Y	IN40/MUM/2007A (DEEPAK PRANJIVANDAS SHAH [IN]), 02 February 2007 Abstract, claims 1-15	23-36
A	The whole document	1-22
A	GB2292140A (HAYS CHEM DISTRIBUTION Ltd [UK]), 14 February 1996 The whole document	1-36
A	US5599373A (FPS-FINANCES PRODUCTS SERVICES SRL [IT]), 04 February 1997 The whole document	1-36
A	WO1990003350A1 (FPS-ITALIANA FINANCES PRODUCTS	

☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"D" document cited by the applicant in the international application
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"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16-09-2020	16-09-2020
Name and mailing address of the ISA/ Indian Patent Office Plot No.32, Sector 14,Dwarka,New Delhi-110075 Facsimile No.	Authorized officer Manoj Kumar Telephone No. +91-1125300200

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	SERVICES SRL [IT]), 05 April 1990 The whole document	1-36
A	WO2010118532A1 (SULPHUR SOLUTIONS INC [CA]), 21 October 2010 The whole document	1-36

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/IB2020/055435

Citation	Pub.Date	Family	Pub.Date
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