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RICHARD W. WIEKING
 CLERK, U.S. DISTRICT COURT
 NORTHERN DISTRICT OF CALIFORNIA

10 Attorneys for Plaintiff
 11 Tessengerlo Kerley, Inc.

12 **UNITED STATES DISTRICT COURT FOR THE**
 13 **NORTHERN DISTRICT OF CALIFORNIA**

DMR

15
 16 **TESSENDERLO KERLEY, INC., a**
 17 **Delaware corporation,**

18 **Plaintiff,**

19 **v.**

20 **D & M CHEM, INC., a Washington**
 21 **corporation,**

22 **Defendant.**

CV 11 3955**Case No.****COMPLAINT FOR
PATENT INFRINGEMENT****DEMAND FOR JURY TRIAL****COMPLAINT FOR PATENT INFRINGEMENT**

1 Plaintiff Tessengerlo Kerley, Inc. ("TKI"), by and through its undersigned attorneys, files
2 this Complaint against defendant D & M Chem, Inc. ("D&M Chem"). In support of its claims,
3 TKI states as follows:

4 **PARTIES**

5 1. TKI is a Delaware corporation having its principal place of business at 2255
6 North 44th Street, Phoenix, Arizona 85008.

7 2. Upon information and belief, defendant D&M Chem is a Washington
8 corporation, having a place of business at 112 Charron Road, Moxee, Washington 98936.

9 3. Defendant D&M Chem markets and distributes crop protectants throughout the
10 United States including in the state of California, in direct competition with TKI.

11 **JURISDICTION AND VENUE**

12 4. This is an action for patent infringement arising under the Patent Act.

13 5. This Court has subject matter jurisdiction over this action under 28 U.S.C. §
14 1338(a), which confers jurisdiction over cases of patent infringement, and under 28 U.S.C. §
15 1331, which confers federal question jurisdiction in general.

16 6. Venue is proper in this district pursuant to 28 U.S.C. §§ 1391 and 1400.

17 **INTRADISTRICT ASSIGNMENT**

18 7. This is an Intellectual Property Action to be assigned on a district-wide basis
19 pursuant to Civil Local Rule 3-2(c).

20 **NATURE OF THE ACTION**

21 8. This action is brought under the Patent Act, 35 U.S.C. § 271 *et seq.*

22 9. TKI seeks injunctive and compensatory damages for D&M Chem's willful
23 infringement of TKI's United States Patent Nos. 6,110,867 ("the '867 patent") and 6,464,995
24 ("the '995 patent").

25 10. TKI and D&M Chem are direct competitors in the market for the manufacture
26 and sale of crop protectants.

27 11. D&M Chem's patent infringement has resulted in substantial harm to TKI in
28 the form of lost sales and lost profits. Injunctive relief from this Court is necessary to prevent

1 further harm to TKI.

2 **BACKGROUND**

3 **TKI's Patents**

4 12. The '867 patent, entitled "Method for Providing Enhanced Photosynthesis,"
5 issued on August 29, 2000 in the name of inventors David Michael Glenn, Dennis G.
6 Sekutowski, and Gary J. Puterka. TKI enjoys all exclusive rights and privileges with respect
7 to the '867 patent in the United States, including the right to enforce the '867 patent in its
8 own name. A copy of the '867 patent is attached hereto as Exhibit 1.

9 13. The '867 patent was the subject of reexamination proceedings, Application
10 Serial No. 90/006,658, before the United States Patent and Trademark Office ("USPTO"). A
11 reexamination certificate was issued by the USPTO on March 7, 2006.

12 14. The subject matter of the '867 patent relates to a method for enhancing
13 photosynthesis of a horticultural crop by increasing carbon dioxide assimilation of said
14 horticultural crop which comprises applying to the surface of said horticultural crop an
15 effective amount of one or more highly reflective particulate materials, said particulate
16 materials being finely divided, and wherein the particles as applied allow for the exchange of
17 gases on the surface of said crop and the finely divided particulate materials have a median
18 individual particle size below about 3 microns.

19 15. The '995 patent, entitled "Treated Horticultural Substrates," issued on October
20 15, 2002 in the name of inventors Dennis G. Sekutowski, and Gary J. Puterka, and David
21 Michael Glenn. TKI enjoys all exclusive rights and privileges with respect to the '995 patent
22 in the United States, including the right to enforce the '995 patent in its own name. A copy
23 of the '995 patent is attached hereto as Exhibit 2.

24 16. The subject matter of the '995 patent relates to a method for enhancing the
25 horticultural effect of horticultural substrates selected from the group consisting of fruits,
26 vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants which
27 comprises applying a slurry comprising water, a surfactant, and one or more particulate
28 materials, selected from the group consisting of calcium carbonate, hydrous kaolin, calcined

1 kaolin and mixtures thereof, to the surface of said substrate to form a membrane comprised
2 of one or more particulate layers and the surfactant, said layers comprising one or more
3 particulate materials, said particulate materials being finely divided, and wherein said
4 membrane allows for the exchange of gases on the surface of said substrate.

5 Crop Protectant Products

6 17. TKI manufactures and sells a kaolin-based crop protectant product under the
7 brand name Surround[®], for use as a broad spectrum agricultural crop protectant for
8 controlling damage from various physiological disorders such as sunburn and heat stress as
9 well as a growth enhancer. Exhibit 3 (Surround label). TKI has the right to exclude from the
10 marketplace any competing product for which its application infringes at least claim 1 of the
11 '867 patent and/or claim 23 of the '995 patent.

12 18. The Eclipse[®] crop protectant product has been distributed by defendant D&M
13 Chem. See Exhibit 4 (D&M Eclipse brochure).

14 19. The Eclipse product was also distributed by a third-party Novazone, Inc.
15 ("Novazone"), which has since changed its corporate name to Purfresh, Inc. ("Purfresh"). See
16 Exhibit 5 (Novazone Eclipse brochure). Purfresh is a Delaware corporation, having a place of
17 business in this judicial district at 47211 Bayside Parkway, Fremont, California 94538. On
18 information and belief, Purfresh obtained the Eclipse product from D&M Chem.

19 D&M Chem's Infringement of TKI's Patents

20 20. Upon information and belief, application of the Eclipse product infringes at
21 least claim 1 of the '867 patent and claim 23 of the '995 patent.

22 21. D&M Chem's promotional literature for the Eclipse product describes it as: (1)
23 a calcium carbonate and boron liquid suspension having submicron particles; (2) effective in
24 preventing and/or reducing sunburn and heat stress; and (3) increasing color, the integrity of
25 cell structure, and reducing physiological problems of crops. See Exhibit 4.

26 22. Novazone's promotional literature for the Eclipse product describes it as: (1) a
27 calcium carbonate and boron colloidal liquid having submicron particles; (2) effective in the
28 control of sunburn and lessening the effects of heat stress; and (3) enabling natural and better

1 coloring and photosynthesis as well as providing higher pack-outs, larger produce, and
2 earlier harvest dates. *See* Exhibit 5.

3 **COUNT I**

4 **Infringement of U.S. Patent No. 6,110,867**

5 23. Paragraphs 1 through 22 of the complaint are incorporated by reference as
6 though fully set forth herein.

7 24. All claims of the '867 patent are presumed valid and enforceable.

8 25. On information and belief, defendant D&M Chem has contributorily infringed
9 and/or actively induced the infringement under 35 U.S.C. § 271, and continues to
10 contributorily infringe and/or actively induce the infringement under 35 U.S.C. § 271, of at
11 least claim 1 of the '867 patent by (1) making, having made, selling, and/or offering for sale,
12 (2) authorizing others to make, have made, sell, and/or offer for sale, and/or (3) causing
13 others to use the Eclipse product.

14 26. Defendant D&M Chem's infringement has been committed with knowledge of
15 the '867 patent and has been intentional, willful and deliberate.

16 27. As a result of defendant D&M Chem's infringement of the '867 patent, TKI
17 has been damaged and will be further damaged, and is entitled to be compensated for such
18 damages, pursuant to 35 U.S.C. § 284, in an amount to be determined at trial.

19 28. As a result of defendant D&M Chem's infringement of the '867 patent, TKI
20 has suffered and will continue to suffer irreparable harm, for which TKI has no adequate
21 remedy at law, unless the Court enjoins such infringing activities pursuant to 35 U.S.C. §
22 283.

23 **COUNT II**

24 **Infringement of U.S. Patent No. 6,464,995**

25 29. Paragraphs 1 through 28 of the complaint are incorporated by reference as
26 though fully set forth herein.

27 30. All claims of the '995 patent are presumed valid and enforceable.

28 31. On information and belief, defendant D&M Chem has contributorily infringed

1 and/or actively induced the infringement under 35 U.S.C. § 271, and continues to
2 contributorily infringe and/or actively induce the infringement under 35 U.S.C. § 271, of at
3 least claim 23 of the '995 patent by (1) making, having made, selling, and/or offering for
4 sale, (2) authorizing others to make, have made, sell, and/or offer for sale, and/or (3) causing
5 others to use the Eclipse product.

6 32. Defendant D&M Chem's infringement has been committed with knowledge of
7 the '995 patent and has been intentional, willful and deliberate.

8 33. As a result of defendant D&M Chem's infringement of the '995 patent, TKI
9 has been damaged and will be further damaged, and is entitled to be compensated for such
10 damages, pursuant to 35 U.S.C. § 284, in an amount to be determined at trial.

11 34. As a result of defendant D&M Chem's infringement of the '995 patent, TKI
12 has suffered and will continue to suffer irreparable harm, for which TKI has no adequate
13 remedy at law, unless the Court enjoins such infringing activities pursuant to 35 U.S.C. §
14 283.

15 **REQUEST FOR RELIEF**

16 WHEREFORE, TKI respectfully requests that this Court enter a Judgment and Order
17 in its favor and against defendant D&M Chem as follows:

18 (a) A judgment that defendant D&M Chem has contributed to and/or actively
19 induced the infringement of the '867 patent by (i) making, having made, selling, and/or
20 offering for sale, (ii) authorizing others to make, have made, sell, and/or offer for sale, and/or
21 (iii) causing others to use the Eclipse product in the United States;

22 (b) A judgment that defendant D&M Chem has contributed to and/or actively
23 induced the infringement of the '995 patent by (i) making, having made, selling, and/or
24 offering for sale, (ii) authorizing others to make, have made, sell, and/or offer for sale, and/or
25 (iii) causing others to use the Eclipse product in the United States;

26 (c) A judgment and order permanently enjoining defendant D&M Chem from
27 further infringing the '867 patent by (i) making, having made, selling, and/or offering for
28 sale, (ii) authorizing others to make, have made, sell, and/or offer for sale, and/or (iii)

1 causing others to use the Eclipse product in the United States;

2 (d) A judgment and order permanently enjoining defendant D&M Chem from
3 further infringing the '995 patent by (i) making, having made, selling, and/or offering for
4 sale, (ii) authorizing others to make, have made, sell, and/or offer for sale, and/or (iii)
5 causing others to use the Eclipse product in the United States;

6 (e) A judgment and order requiring defendant D&M Chem to pay all available and
7 legally permissible damages to compensate TKI for defendant's infringing acts, but in no
8 event less than a reasonable royalty in accordance with 35 U.S.C. § 284;

9 (f) A finding that defendant's conduct has been willful, warranting an award of
10 treble damages under 35 U.S.C. § 284;

11 (g) A finding that this case is exceptional under 35 U.S.C. § 285, warranting an
12 award to TKI of its costs, including attorney fees, and other expenses incurred in connection
13 with this action;

14 (h) A judgment and order requiring that the defendant pay TKI pre-judgment
15 interest and post-judgment interest on all damages awarded;

16 (i) Such further relief as this Court deems just and appropriate.

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JURY DEMAND

TKI demands a trial by jury on all issues so triable.

STEPTOE & JOHNSON LLP

Dated: August 10, 2011

By: 

Daniel R. Blakey (State Bar No. 143748)

Seth A. Watkins (*pro hac vice* pending)

Timothy C. Bickham (*pro hac vice* pending)

Houda Morad (*pro hac vice* pending)

Attorneys for Plaintiff

TESSENDERLO KERLEY, INC.

EXHIBIT 1

US006110867A

United States Patent [19]**Glenn et al.**[11] **Patent Number:** **6,110,867**[45] **Date of Patent:** **Aug. 29, 2000**[54] **METHOD FOR PROVIDING ENHANCED PHOTOSYNTHESIS**[75] **Inventors:** **David Michael Glenn**, Shepherdstown, W. Va.; **Dennis G. Sekutowski**, Stockton, N.J.; **Gary J. Puterka**, Shepherdstown, W. Va.[73] **Assignee:** **Engelhard Corporation**, Iselin, N.J.[21] **Appl. No.:** **08/972,659**[22] **Filed:** **Nov. 18, 1997****Related U.S. Application Data**

[63] Continuation-in-part of application No. 08/812,301, Mar. 5, 1997, Pat. No. 5,908,708.

[51] **Int. Cl.⁷** **A01N 59/00; A01N 59/06; A01N 55/02; A01N 57/00**[52] **U.S. Cl.** **504/119; 504/120; 504/126; 504/127; 504/187; 504/188; 71/DIG. 1**[58] **Field of Search** **504/119, 120, 504/126, 127, 188, 187; 71/DIG. 1**[56] **References Cited****U.S. PATENT DOCUMENTS**

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Primary Examiner—S. Mark Clardy**Assistant Examiner**—Alton Pryor**Attorney, Agent, or Firm**—Raymond F. Keller[57] **ABSTRACT**

Disclosed is a method for enhancing the photosynthesis of horticultural crops which involves treating the surface of said horticultural crop with an effective amount of one or more highly reflective particulate materials.

53 Claims, No Drawings

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METHOD FOR PROVIDING ENHANCED PHOTOSYNTHESIS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of U.S. patent application Ser. No. 08/812,301, filed Mar. 5, 1997, now U.S. Pat. No. 5,908,708 which is incorporated herein by reference for its teachings related to the invention disclosed herein.

FIELD OF THE INVENTION

The present invention is directed to a method for enhancing the photosynthesis of horticultural crops.

BACKGROUND OF THE INVENTION

Improved yield or plant productivity is a desired horticultural effect on horticultural crops that is generally limited by the amount of light, temperature, relative humidity and other uncontrollable environmental factors when pests, water and nutrients are adequately controlled. Particulate matter from a wide range of sources is generally regarded as limiting plant productivity. See for example, Farmer, "The Effects of Dust on Vegetation—A Review," *Environmental Pollution* 79:63–75 (1993).

The prior art has discussed photosynthesis and the effects of environmental stresses on plants. See, for example; Nonomura and Benson, "Methods and compositions for enhancing carbon fixation in plants," U.S. Pat. No. 5,597,400, Stanhill, G., S. Moreshet, and M. Fuchs, "Effect of Increasing Foliage and Soil Reflectivity on the Yield and Water Use Efficiency of Grain Sorghum," *Agronomy Journal* 68:329–332 (1976); Moreshet, S., Y. Cohen, and M. Fuchs, "Effect of Increasing Foliage Reflectance on Yield, Growth, and Physiological Behavior of a Dryland Cotton Crop," *Crop Science* 19:863–868 (1979), which states that "within 2 days after spraying the kaolin reduced $^{14}\text{CO}_2$ uptake (photosynthesis) by more than 20%" and "the kaolin sprays would appear to reduce transpiration more than photosynthesis"; Bar-Joseph, M. and J. Frenkel, "Spraying citrus plants with kaolin suspensions reduces colonization by the spiraea aphid (*Aphis citricola* van der Goot)" *Crop Protection* 2(3):371–374 (1983), which states that "The reasons for this [yield increase of Stanhill, *Ibid.* and Moreshet, *Ibid.*] are uncertain [because photosynthesis is reduced] but aphid and virus control may have contributed to this yield increase"; Rao, N. K. S., "The Effects of Antitranspirants on Leaf Water Status, Stomatal Resistance and Yield in Tomato," *J. of Horticultural Science* 60:89–92 (1985); Lipton, W. J., and F. Matoba, "Whitewashing to Prevent Sunburn of 'Crenshaw' Melons," *HortScience* 6:434–345 (1971); Proctor, J. T. A. and L. L. Creasy "Effect of Supplementary Light on Anthocyanin Synthesis in 'McIntosh' Apples," *J. Amer. Soc. Hort. Sci* 96:523–526 (1971); Lord, W. J., and D. W. Greene, "Effects of Summer Pruning on the Quality of 'McIntosh' Apples," *HortScience* 17:372–373.

Therefore, there is still a need for cost effective inert, nontoxic methods for enhancing photosynthesis of horticultural crops. The prior art teaches away from the use of highly reflective inert particles of the instant invention in that increasing reflectivity reflects photosynthetically active light, thus, reducing photosynthesis. Unexpectedly, the instant invention results in an opposite effect—enhanced photosynthesis.

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SUMMARY OF THE INVENTION

This invention relates to a method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop.

DETAILED DESCRIPTION OF THE INVENTION

This invention relates to a method for enhancing the photosynthesis of horticultural crops. Photosynthesis is the process by which photosynthetic plants utilize solar energy to build carbohydrates and other organic molecules from carbon dioxide and water. The conversion of carbon dioxide to such organic molecules is generally referred to as carbon fixation or photosynthesis and, in most plants, occurs by the reductive pentose phosphate cycle, generally referred to as the C-3 cycle. The study of the path of carbon in photosynthesis four decades ago (A. A. Benson (1951), "Identification of ribulose in $^{14}\text{CO}_2$ photosynthesis products" *J. Am. Chem. Soc.* 73:2971; J. R. Quayle et al. (1954), "Enzymatic carboxylation of ribulose diphosphate" *J. Am. Chem. Soc.* 76:3610) revealed the nature of the carbon dioxide fixation process in plants. The effects of enhanced photosynthesis are typically observed by increased yields/productivity, e.g., increased fruit size or production (usually measured in weight/acre), improved color, increased soluble solids, e.g. sugar, acidity, etc., and reduced plant temperature.

The horticultural crops to which this invention relate are actively growing and/or fruiting agricultural and ornamental crops and the products thereof, including those selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants.

The particulate materials useful for the purposes of this invention are highly reflective. As used herein, "highly reflective" means a material having a "Block Brightness" of at least about 80 and preferably at least about 90 and more preferably at least about 95 as measured by TAPPI standard T 646. Measurements can be made on a Reflectance Meter Technidyne S-4 Brightness Tester manufactured by Technidyne Corporation which is calibrated at intervals not greater than 60 days using brightness standards (paper tabs and opal glass standards) supplied by the Institute of Paper Science, or Technidyne Corporation. Typically a particle block or plaque is prepared from 12 grams of a dry (<1% free moisture) power. The sample is loosely placed in a cylinder holder and a plunger is slowly lowered over the sample to a pressure of 29.5–30.5 psi and held for about 5 seconds. The pressure is released and the plaque is examined for defects. A total of three plaques are prepared and three brightness values are recorded on each plaque by rotating the plaque about 120 degrees between readings. The nine values are then averaged and reported.

The finely divided particulate materials useful for the purposes of this invention may be hydrophilic or hydrophobic materials and the hydrophobic materials may be hydrophobic in and of themselves, e.g., mineral talc, or may be hydrophilic materials that are rendered hydrophobic by application of an outer coating of a suitable hydrophobic wetting agent (e.g., the particulate material has a hydrophilic core and a hydrophobic outer surface).

Typical particulate hydrophilic materials useful for the purposes of this invention include: minerals, such as calcium

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carbonate, talc, kaolin (both hydrous and calcined kaolins, with calcined kaolins being preferred), bentonites, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, precipitated calcium carbonate, diatomaceous earth and barytes; functional fillers such as aluminum trihydrate, pyrogenic silica, and titanium dioxide.

The surfaces of such materials can be made hydrophobic by addition of hydrophobic wetting agents. Many industrial mineral applications, especially in organic systems such as plastic composites, films, organic coatings or rubbers, are dependent upon just such surface treatments to render the mineral surface hydrophobic; see, for example, Jesse Edenbaum, *Plastics Additives and Modifiers Handbook*, Van Nostrand Reinhold, New York, 1992, pages 497-500 which is incorporated herein by reference for teachings of such surface treatment materials and their application. So-called coupling agents such as fatty acids and silanes are commonly used to surface treat solid particles as fillers or additives targeted to these industries. Such hydrophobic agents are well known in the art and common examples include: organic titanates such as Tilcom® obtained from Tioxide Chemicals; organic zirconate or aluminate coupling agents obtained from Kenrich Petrochemical, Inc.; organofunctional silanes such as Silquest® products obtained from Witco or Prosil® products obtained from PCR; modified silicone fluids such as the DM-Fluids obtained from Shin Etsu; and fatty acids such as Hystrene® or Industrene® products obtained from Witco Corporation or Emersol® products obtained from Henkel Corporation (stearic acid and stearate salts are particularly effective fatty acids and salts thereof for rendering a particle surface hydrophobic).

Examples of preferred particulate materials suitable for the purposes of this invention that are commercially available from Engelhard Corporation, Iselin, N.J. are the calcined kaolins sold under the trademark Satintone® and the siloxane treated calcined kaolins sold under the trademark Translink®; and calcium carbonate commercially available from English China Clay under the trademarks Atomite® and Supermite® and stearic acid treated ground calcium carbonates commercially available from English China Clay under the trademarks Supercoat® and Kotamite®.

The term "finely divided" when utilized herein means that the particulate materials have a median individual particle size below about 10 microns and preferably below about 3 microns and more preferably the median particle size is about one micron or less. Particle size and particle size distribution as used herein are measured with a Micromeritics Sedigraph 5100 Particle Size Analyzer. Measurements were recorded in deionized water for hydrophilic particles. Dispersions were prepared by weighing 4 grams of dry sample into a plastic beaker adding dispersant and diluting to the 80 ml mark with deionized water. The slurries were then stirred and set in an ultrasonic bath for 290 seconds. Typically, for kaolin 0.5% tetrasodium pyrophosphate is used as a dispersant; with calcium carbonate 1.0% Calgon T is used. Typical densities for the various powders are programmed into the sedigraph, e.g., 2.58 g/ml for kaolin. The sample cells are filled with the sample slurries and the X-rays are recorded and converted to particle size distribution curves by the Stokes equation. The median particle size is determined at the 50% level.

Preferably, the particulate material has a particle size distribution wherein up to 90% by weight of the particles have a particle size of under about 10 microns, preferably below about 3 microns and more preferably about one micron or less.

The particulate materials particularly suitable for use in this invention are inert and nontoxic.

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As used herein "inert" particulate materials are particles that are not phytotoxic.

The particulate materials are preferably nontoxic meaning that in the limited quantities needed for effective enhanced horticultural effect such materials are not considered harmful to animals, the environment, the applicator and the ultimate consumer.

As previously discussed, this invention relates to horticultural crops wherein the surface of said crop is treated with one or more particulate materials. This treatment should not materially affect the exchange of gases on the surface of said crop. The gases which pass through the particle treatment are those which are typically exchanged through the surface skin of living plants. Such gases typically include water vapor, carbon dioxide, oxygen, nitrogen and volatile organics.

The surface of said horticultural crop is treated with an amount of one or more highly reflective particulate materials that is effective in enhancing photosynthesis of the horticultural crop. The treatment coverage of said crop is within the skill of the ordinary artisan. Less than full crop coverage is within the scope of this invention and can be highly effective, for example, neither the under surface of the crop (that which is not exposed directly to the source of light) need be treated by the method of this invention nor must the upper surface of the crop be completely covered; although full substrate coverage can provide additional benefits such as effective disease control, smoother fruit surface, reduced bark and fruit cracking, and reduced russetting. Reference is made to U.S. Ser. No. 08/972,648, filed concurrently herewith on Nov. 18, 1997, entitled "Treated Horticultural Substrates" which is incorporated herein by reference for its teachings regarding methods for achieving these additional benefits. The method of this invention may result in the residue of the treatment forming a membrane of one or more layers of highly reflective particulate materials on the crop surface.

The particulate materials useful for the purposes of this invention may be applied as a slurry of finely divided particles in a volatile liquid such as water, a low boiling organic solvent or low boiling organic solvent/water mixture. Adjuvants such as surfactants, dispersants or spreaders/stickers (adhesives) may be incorporated in preparing an aqueous slurry of the particulate materials of this invention. One or more layers of this slurry can be sprayed or otherwise applied to the crop surface. The volatile liquid is preferably allowed to evaporate between coatings. The residue of this treatment may be hydrophilic or hydrophobic. Applying particles as a dust, although not being commercially practical on a large scale due to drift and inhalation hazards, is an alternative for carrying out the method of this invention.

Spreader/stickers that can be mixed with hydrophilic particles (3% or more solids in water) to aid in spraying uniform treatments on horticultural substrates are: modified phthalic glycerol alkyd resins such as Latron B-1956 from Rohm & Haas Co.; Plant oil based materials (cocodithalymide) with emulsifiers such as Sea-wet from Salisbury lab, Inc.; Polymeric terpenes such as Pinene II from Drexel Chem. Co.; nonionic detergents (ethoxylated tall oil fatty acids) such as Toximul 859 and Ninex MT-600 series from Stephan.

The the particle treatment may be applied as one or more layers of finely divided particulate material. The amount of material applied is within the skill of one of ordinary skill in the art. The amount will be sufficient to improve photosynthesis of the crop to which these particles are applied.

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Typically, this treatment will be most effective when crop surface is white in appearance. For example, this can typically be accomplished by applying from about 25 up to about 5000 micrograms of particulate material/cm² of crop surface for particles having specific density of around 2-3 g/cm³, more typically from about 100 up to about 3000 and preferably from about 100 up to about 500. As the brightness of the highly reflective particles increases lesser amounts of these brighter particles are necessary to be effective for the purposes of this invention. In addition, environmental conditions such as wind and rain may reduce crop coverage of the highly reflective particulate materials and therefore it is within the scope of this invention to apply the highly reflective particles one or more times during the growing season of said horticultural crop so as to maintain the desired effect of invention.

The low boiling organic liquids useful in the present invention are preferably water-miscible and contain from 1 to 6 carbon atoms. The term "low boiling" as used herein shall mean organic liquids which have a boiling point generally no more than 100° C. These liquids enable the particulate solids to remain in finely divided form without significant agglomeration. Such low boiling organic liquids are exemplified by: alcohols such as methanol, ethanol, propanol, i-propanol, i-butanol, and the like, ketones such as acetone, methyl ethyl ketone and the like, and cyclic ethers such as ethylene oxide, propylene oxide and tetrahydrofuran. Combinations of the above-mentioned liquids can also be employed. Methanol is the preferred low boiling organic liquid.

Low boiling organic liquids may be employed in applying the particles to crop substrates for the purposes of this invention. Typically, the liquids are used in an amount sufficient to form a dispersion of the particulate material. The amount of liquid is typically up to about 30 volume percent of the dispersion, preferably from about 3 up to about 5 volume percent, and most preferably from about 3.5 to about 4.5 volume percent. The particulate material is preferably added to a low boiling organic liquid to form a slurry and then this slurry is diluted with water to form an aqueous dispersion. The resulting slurry retains the particles in finely divided form wherein most of the particles are dispersed to a particle size of less than about 10 microns.

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the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) no treatment, 3) weekly application of Translink® 77 beginning in Mar. 11, 1997, 4) weekly application of calcined kaolin (Satintone® 5HP) beginning in Apr. 29, 1997, and 5) weekly application of treated calcium carbonate (SuperCoat®—commercially available from English China Clay) beginning in Apr. 29, 1997. Treatments (3) and (5) applied 25 pounds material suspended in 4 gal methanol and added to 100 gal water. Treatment (4) applied 25 pounds material suspended in 100 gal water with the addition of 27 oz Ninex® MT-603 and 2 pints Toximul. These treatments were applied at the rate of 125 gal/acre using an orchard sprayer. This mixture was applied at the rate of 125 gal/acre using an orchard sprayer. The treatments were arranged in a randomized complete block design with 4 replications and 3 trees/plot. Treatments were not irrigated and received 21.58 cm of precipitation from May 1 to Aug. 30, 1997. Fruit were harvested at maturity; fruit number, weight and color were measured. Color was measured using a Hunter calorimeter. Color values represent Hunter "a" value units, in which increasing value represents increasing red color. Photosynthesis and stomatal conductance were measured on Aug. 6 and 8, 1997. Photosynthesis and stomatal conductance data were collected using a Licor 6300 photosynthesis system. Increasing values of photosynthesis and stomatal conductance represent increasing assimilation of carbon dioxide from the atmosphere and transpiration of water from the leaf, respectively; both parameters reflect improved plant productivity when values increase. Treatments (1) and (3) were measured twice daily at 10 to 11 am and 2 to 3 pm. Three trees in each plot were measured with 2 sunlit leaves/tree. Canopy temperature was measured using an Everest Interscience (Model 110) infrared thermometer with +/-0.5° C. accuracy, in which the temperature of the plant surface approximately 1 m in diameter was determined on the sunlit side of the tree. Data for canopy temperature are presented as the difference between leaf and air temperature. A negative canopy temperature denotes a canopy cooler than air temperature due to transpiration and heat reflection. The data are reported in Table I.

TABLE I

Treatment	Yield/tree (kg)	Fruit weight (g)	Red Color	Photosynthesis rate (μmoles CO ₂ /m ² /sec)	Stomatal conductance (mol/m ² /sec)	Canopy Temperature (C.)
Conventional	43.7	136	19.7	6.7	0.35	-4.2
Control	30.1	123	23.2			
Translink® 77	51.6	135	23.9	9.2	0.57	-5.2
Calcined Kaolin	37.6	124	21.0			
Treated CaCO ₃	39.1	130	24.1			-5.5

The following examples are illustrative of embodiments of the invention and are not intended to limit the invention as encompassed by the claims forming part of the application.

EXAMPLE 1

"Red Delicious" apple trees received the following treatments: 1) Conventional pesticide applications applied according to the presence of economic levels of pests using

The use of hydrophobic kaolin (Translink® 77) increased yield compared to conventional management (51.6 vs 43.7 kg, respectively) without a meaningful reduction in fruit size (135 vs 136 g/fruit).

The use of hydrophobic kaolin (Translink® 77) improved fruit color compared to the conventional management (23.9 vs 19.7). Treated CaCO₃ (SuperCoat®) and calcined Kaolin (Satintone® 5HB) also improved color compared to the conventional management (24.1 and 21.0 vs 19.7). The

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untreated control improved color compared to the conventional management (23.2 vs 19.7) but this is likely due to defoliation of the tree due to poor pest control since no pesticides were applied (see Lord and Greene, *Ibid.*). Defoliation from pest damage increases light to the fruit surface which increases color development. Pest control levels were adequate in all other treatments and did not result in defoliation.

Average precipitation approximates 35.6 cm from April 1 to August 30; precipitation was 40% below normal.

The application of Translink® 77 increased photosynthesis, stomatal conductance and reduced plant temperature. Stomatal conductance is a measure of the width of stomates on the underside of the leaf. Water loss, in the form of transpiration, occurs through the stomates and is controlled by the size of the stomatal opening. The greater the size of the opening, the greater is the stomatal conductance, and so transpiration is greater. Similarly, the greater the size of the stomatal opening, the greater is the influx of carbon dioxide necessary for photosynthesis. Canopy temperature was reduced by the application of Translink® 77 due to the increased transpirational cooling of the leaf related to increased stomatal conductance resulting from the application of Translink® 77. The application of calcium carbonate (SuperCoat®) also reduced plant temperature, presumably due to increased transpirational cooling of the leaf related to increased stomatal conductance.

Yakima, Wash.

"Red Delicious" apple trees received the following treatments: 1) no treatment; this untreated control did not have pest pressures that exceeded the threshold for pesticide application, 2) application of Translink® 77 on April 5, May 8, 29; June 25; July 14; September 4, 3) application of Translink® 77 on the same dates as "(2)" and on May 22, June 9, and July 31. Treatments (2) and (3) applied 25 pounds material suspended in 4 gal methanol and added to 96 gal water. This mixture was applied at the rate of 100 gal/acre using an orchard sprayer. The treatments were arranged in a randomized complete block design with 3 replications of 3 trees/plot. Treatments were all irrigated on a weekly basis to meet plant water needs using sprinkler irrigation located beneath the trees. Photosynthesis and stomatal conductance were measured on Jul. 17 to 20, 1997. Photosynthesis data were collected using a Licor 6300 photosynthesis system. Treatments (1), (2) and (3) were measured twice daily at 10 to 11 am and 2 to 3 pm. Three trees in each plot were measured with 2 sunlight leaves/tree. Data are the mean values for all days and hours sampled. Canopy temperature was measured using an Everest Inter-science Infrared (Model 110) thermometer with ± 0.5 C. accuracy, in which the temperature of the plant surface approximately 1 meter in diameter was determined on the sunlit side of the tree. Data for canopy temperature are presented as the difference between leaf and air temperature. A negative canopy temperature denotes a canopy cooler than air temperature due to transpiration and heat reflection. Canopy temperature data were collected from Aug. 17 to 20, 1997. The data presented in Table IV are representative of the entire data set. At the time of harvest, 20 fruit were randomly collected from each of the 3 trees/plot (total of 180 fruit/treatment). Fruit were weighed and color determined. Color was determined with a Hunter colorimeter. Color values represent Hunter "a" values.

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TABLE II

Treatment	Fruit weight (g/fruit)	Photosynthesis ($\mu\text{mol CO}_2/\text{m}^2/\text{sec}$)	Stomatal conductance ($\text{mol}/\text{m}^2/\text{sec}$)	Canopy temperature ($^{\circ}\text{C}$)
Control	164	8.8	0.24	-4.5
Translink® 77 applied 7 times	177	11.3	0.43	-5.7
Translink® 77 applied 10 times	195	12.9	0.46	-6.0

Fruit size increased with increasing applications of Translink® 77.

Trees in the study had fruit size greater than the study in Kearneysville, W. Va. due to the use of irrigation.

The reduced canopy temperature of both Translink® 77 treatments illustrates that the application of these particles can reduce plant temperature.

The application of Translink® 77 increased photosynthesis, stomatal conductance and reduced plant temperature. Canopy temperature was reduced by the application of Translink® 77 due to the increased transpirational cooling of the leaf related to increased stomatal conductance resulting from the application of Translink® 77. Reducing the frequency of application from 7 applications did reduce photosynthesis, stomatal conductance, and canopy temperature compared to 10 applications, demonstrating that there is a beneficial response to increasing amounts of Translink® 77 coverage.

EXAMPLE 3

Santiago, Chile

"September Lady" peach, spaced 4 m x 6 m, received the following treatments: 1) Conventional pesticide application applied according to the presence of economic levels of pests, 2) no treatment, 3) weekly application of Translink® 77 beginning Oct. 29, 1996. Treatment (3) applied 25 pounds material suspended in 4 gal methanol and added to 96 gal water. This mixture was applied at the rate of 100 gal/acre using a high pressure hand sprayer. Treatments were irrigated weekly using surface irrigation. Fruit were harvested at maturity and the number and weight measured. The data are reported in Table III.

TABLE III

Treatment	Yield/tree (kg)	Fruit weight (g)	Fruit number/tree
Conventional	13.9	156	94
Control	14.6	139	109
Translink® 77	25.4	137	156

The use of hydrophobic kaolin (Translink® 77) increased yield compared to the conventional treatment and the control by increasing the number of fruit/tree. Fruit size was reduced, although not statistically, from 156 to 137 g due to the larger number of fruit on the peach tree (94 vs 156).

EXAMPLE 4

Biglerville, Pa.—Dan Pack Orchard

"Golden Delicious" apples received 3 treatments: 1) commercial pesticide application applied according to the presence of economic levels of pests using the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) full rate of Translink® 77, and 3) half rate of Translink® 77. Treatments (2) and (3) applied 25 and 12.5

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pounds material, respectively, suspended in 4 and 2 gal methanol, respectively, and added to 100 gal water. This mixture was applied at the rate of 200 gal/acre using an orchard sprayer. The treated area was approximately 1 acre plots with 2 replications of each treatment in a randomized block design. At harvest the plots were commercially harvested and processed by a commercial grading line. At the time of grading, 100 fruit from each plot were randomly chosen to determine fruit size, color, and surface defects. Color was determined using a Hunter colorimeter. Green color values represent Hunter "a" values in which higher values represent more yellow color, a beneficial trait in "Golden Delicious" apple. The data are reported in Table IV.

TABLE IV

Treatment	Fruit size (mm)	Green color
Translink® 77 full rate	69	-8.0
Translink® 77 half rate	67	-8.9
Conventional	67	-10.0

Application of Translink® 77 at the full and half rate reduced green color, and Translink® 77 at the full rate increased fruit size compared to the half rate and conventional treatment.

"Stayman" apples received 2 treatments: 1) commercial pesticide application applied according to the presence of economic levels of pests using the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) Translink® 77 treatment applied 25 pounds material suspended in 4 gal methanol and added to 96 gal water. This mixture was applied at the rate of 200 gal/acre using an orchard sprayer. Each treatment was applied to 1 acre blocks with no randomization. Apples were harvested commercially and processed on a commercial grading line. Data presented represent percent packout from the commercial grading line. The data are reported in Table V.

TABLE V

Treatment	Fruit size (mm)	<2.5 inches (%)	2.5-2.75 inches (%)	2.75-3.0 inches (%)	>3.0 inches (%)
Translink® 77	69	11	38	44	7
Conventional	62	66	28	6	0

The application of Translink® 77 increased the packout of larger fruit and reduced the losses due to small fruit (<2.5 inches) compared to the conventional treatment.

What is claimed is:

1. A method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the finely divided particulate materials have a median individual particle size below about 3 microns.

2. The method according to claim 1 wherein said particles have a Block Brightness of at least about 90.

3. The method of claim 1 wherein said particulate materials are hydrophobic.

4. The method of claim 1 wherein said particulate materials are hydrophilic.

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5. The method of claim 1 wherein the particulate material has a particle size distribution wherein most of the particles have a particle size of under about 10 microns.

6. The method of claim 1 wherein the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

7. The method of claim 6 wherein said hydrophilic core materials are selected from the group consisting of calcium carbonate, mica, kaolin, bentonite, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, diatomaceous earth, baryte, aluminum trihydrate, titanium dioxide and mixtures thereof.

8. The method of claim 4 wherein said hydrophilic materials are selected from the group consisting of calcium carbonate, talc, hydrous kaolin, calcined kaolin, bentonites, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, precipitated calcium carbonate, diatomaceous earth, barytes, aluminum trihydrate, pyrogenic silica, titanium dioxide and mixtures thereof.

9. The method of claim 6 wherein said hydrophobic outer surface materials are selected from the group consisting of organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

10. The method of claim 1 wherein the horticultural crop is selected from actively growing or fruiting agricultural and ornamental crops.

11. The method of claim 1 wherein the horticultural crop is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants.

12. The method of claim 1 wherein the particulate materials have a median individual particle size below about 3 microns.

13. The method of claim 6 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

14. The method of claim 4 wherein the hydrophilic particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

15. A method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of an actively growing or fruiting horticultural crop selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants which comprises applying to the surface of said horticultural crop an effective amount of a slurry of one or more highly reflective particulate materials having a Block Brightness of at least about 90, said materials comprising one or more particulate materials, selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof, said particulate materials have a median individual particle size of about one micron or less, and wherein said particles as applied allow for the exchange of gases on the surface of said crop.

16. The method of claim 1 or 15 wherein the particulate materials are applied one or more times during the growing season of said horticultural crop.

17. A method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particles have a Block Brightness of at least about 90.

18. The method of claim 17 wherein said particulate materials are hydrophobic.

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19. The method of claim 17 wherein said particulate materials are hydrophilic.

20. The method of claim 17 wherein the particulate material has a particle size distribution wherein most of the particles have a particle size of less than about 10 microns.

21. The method of claim 17 wherein the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

22. The method of claim 21 wherein said hydrophilic core materials are selected from the group consisting of calcium carbonate, mica, kaolin, bentonite, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, diatomaceous earth, baryte, aluminum trihydrate, titanium dioxide and mixtures thereof.

23. The method of claim 19 wherein said hydrophilic materials are selected from the group consisting of calcium carbonate, talc, hydrous kaolin, calcined kaolin, bentonites, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, precipitated calcium carbonate, diatomaceous earth, barytes, aluminum trihydrate, pyrogenic silica, titanium dioxide and mixtures thereof.

24. The method of claim 21 wherein said hydrophobic outer surface materials are selected from the group consisting of organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

25. The method of claim 17 wherein the horticultural crop is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants.

26. The method of claim 17 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

27. The method of claim 21 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

28. The method of claim 19 wherein the hydrophilic particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

29. A method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particulate materials are hydrophobic wherein said particles have a Block Brightness of at least about 80.

30. The method according to claim 29 wherein said particles have a Block Brightness of at least about 90.

31. The method of claim 29 wherein the particulate material has a particle size distribution wherein most of the particles have a particle size of under about 10 microns.

32. The method of claim 29 wherein the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

33. The method of claim 32 wherein said hydrophilic core materials are selected from the group consisting of calcium carbonate, mica, kaolin, bentonite, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, diatomaceous earth, baryte, aluminum trihydrate, titanium dioxide and mixtures thereof.

34. The method of claim 32 wherein said hydrophobic outer surface materials are selected from the group consisting of organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

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35. The method of claim 29 wherein the horticultural crop is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants.

36. The method of claim 29 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

37. The method of claim 32 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

38. A method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particulate materials have a particle size distribution wherein most of the particles have a particle size of under about 10 microns.

39. The method of claim 38 wherein said particulate materials are hydrophobic.

40. The method of claim 38 wherein said particulate materials are hydrophilic.

41. The method of claim 38 wherein the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

42. The method of claim 41 wherein said hydrophilic core materials are selected from the group consisting of calcium carbonate, mica, kaolin, bentonite, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, diatomaceous earth, baryte, aluminum trihydrate, titanium dioxide and mixtures thereof.

43. The method of claim 40 wherein said hydrophilic materials are selected from the group consisting of calcium carbonate, talc, hydrous kaolin, calcined kaolin, bentonites, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, precipitated calcium carbonate, diatomaceous earth, barytes, aluminum trihydrate, pyrogenic silica, titanium dioxide and mixtures thereof.

44. The method of claim 41 wherein said hydrophobic outer surface materials are selected from the group consisting of organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

45. The method of claim 38 wherein the horticultural crop is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants.

46. The method of claim 41 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

47. The method of claim 40 wherein the hydrophilic particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

48. A method for enhancing the photosynthesis of horticultural crops which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particulate materials comprise a hydrophilic core and a hydrophobic outer surface.

49. The method of claim 48 wherein said hydrophilic core materials are selected from the group consisting of calcium carbonate, mica, kaolin, bentonite, clays, pyrophyllite, silica, feldspar, sand, quartz, chalk, limestone, diatomaceous earth, baryte, aluminum trihydrate, titanium dioxide and mixtures thereof.

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50. The method of claim 48 wherein said hydrophobic outer surface materials are selected from the group consisting of organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

51. The method of claim 48 wherein the horticultural crop is selected from actively growing or fruiting agricultural and ornamental crops.

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52. The method of claim 48 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

53. The method of claim 48 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

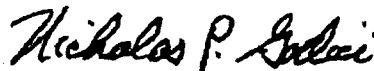
PATENT NO. : 6,110,867
DATED : August 29, 2000
INVENTOR(S) : David Michael Glenn, Dennis G. Sekutowski, Gary J. Puterka

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

On the title page, item: [73], the Assignees should read "Engelhard Corporation, Iselin, New Jersey USA" and "The United States of America, as represented by the Secretary of Agriculture, Washington, D.C., USA".

Signed and Sealed this
Eighth Day of May, 2001

Attest:



NICHOLAS P. GODICI

Attesting Officer

Acting Director of the United States Patent and Trademark Office



US006110867C1

(12) **EX PARTE REEXAMINATION CERTIFICATE (5292nd)****United States Patent****Glenn et al.**(10) Number: **US 6,110,867 C1**(45) Certificate Issued: ***Mar. 7, 2006**(54) **METHOD FOR PROVIDING ENHANCED PHOTOSYNTHESIS**(75) Inventors: **David Michael Glenn**, Shepherdstown, WV (US); **Dennis G. Sekutowski**, Stockton, NJ (US); **Gary J. Puterka**, Shepherdstown, WV (US)(73) Assignees: **Engelhard Corporation**, Iselin, NJ (US); **The United States of America as represented by the Secretary of Agriculture**, Washington, DC (US)**Reexamination Request:**

No. 90/006,658, Jun. 6, 2003

Reexamination Certificate for:

Patent No.: **6,110,867**
 Issued: **Aug. 29, 2000**
 Appl. No.: **08/972,659**
 Filed: **Nov. 18, 1997**

(*) Notice: This patent is subject to a terminal disclaimer.

Certificate of Correction issued May 8, 2001.

Related U.S. Application Data

(63) Continuation-in-part of application No. 08/812,301, filed on Mar. 5, 1997, now Pat. No. 5,908,708.

(51) **Int. Cl.**

A01N 59/00 (2006.01)
A01N 59/06 (2006.01)
A01N 55/02 (2006.01)
A01N 57/00 (2006.01)

(52) **U.S. Cl.** **504/119; 504/120; 504/126; 504/127; 504/187; 504/188; 504/367**(58) **Field of Classification Search** 504/119, 504/120, 126, 127, 187, 188, 367
See application file for complete search history.(56) **References Cited****U.S. PATENT DOCUMENTS**

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Primary Examiner—John Pak(57) **ABSTRACT**

Disclosed is a method for enhancing the photosynthesis of horticultural crops which involves treating the surface of said horticultural crop with an effective amount of one or more highly reflective particulate materials.

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**EX PARTE
REEXAMINATION CERTIFICATE
ISSUED UNDER 35 U.S.C. 307**

THE PATENT IS HEREBY AMENDED AS
INDICATED BELOW.

Matter enclosed in heavy brackets [] appeared in the patent, but has been deleted and is no longer a part of the patent; matter printed in italics indicates additions made to the patent.

AS A RESULT OF REEXAMINATION, IT HAS BEEN DETERMINED THAT:

Claim 12 is cancelled.

Claims 1, 15, 17, 29, 38 and 48 are determined to be patentable as amended.

Claims 2-11, 13-14, 16, 18-28, 30-37, 39-47 and 49-53, dependent on an amended claim, are determined to be patentable.

1. A method for enhancing [the] photosynthesis of a horticultural crop[s] *by increasing carbon dioxide assimilation of said horticultural crop* which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the finely divided particulate materials have a median individual particle size below about 3 microns.

15. A method for enhancing [the] photosynthesis of a horticultural crop[s] *by increasing carbon dioxide assimilation of said horticultural crop* which comprises applying to the surface of an actively growing or fruiting horticultural crop selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, seeds and landscape and ornamental plants [which comprises applying to the surface of said horticultural crop] an effective amount of a slurry of one or more highly reflective particulate materials having a Block Brightness of at least about 90, said materials comprising one or more particulate materials, selected from the group consisting of calcium carbonate, calcined kaolin and

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mixtures thereof, said particulate materials have a median individual particle size of about one micron or less, and wherein said particles as applied allow for the exchange of gases on the surface of said crop.

17. A method for enhancing [the] photosynthesis of a horticultural crop[s] *by increasing carbon dioxide assimilation of said horticultural crop* which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particles have a Block Brightness of at least about 90.

29. A method for enhancing [the] photosynthesis of a horticultural crop[s] *by increasing carbon dioxide assimilation of said horticultural crop* which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particulate materials are hydrophobic wherein said particles have a Block Brightness of at least about 80.

38. A method for enhancing [the] photosynthesis of a horticultural crop[s] *by increasing carbon dioxide assimilation of said horticultural crop* which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particulate materials have a particle size distribution wherein most of the particles have a particle size of under about 10 microns.

48. A method for enhancing [the] photosynthesis of a horticultural crop[s] *by increasing carbon dioxide assimilation of said horticultural crop* which comprises applying to the surface of said horticultural crop an effective amount of one or more highly reflective particulate materials, said particulate materials being finely divided, and wherein the particles as applied allow for the exchange of gases on the surface of said crop and the particulate materials comprise a hydrophilic core and a hydrophobic outer surface.

* * * * *

EXHIBIT 2

US006464995B1

(12) **United States Patent**
Sekutowski et al.(10) **Patent No.:** **US 6,464,995 B1**
(45) **Date of Patent:** ***Oct. 15, 2002**(54) **TREATED HORTICULTURAL SUBSTRATES**(75) **Inventors:** **Dennis G. Sekutowski**, Stockton, NJ
(US); **Gary J. Puterka**; **David Michael Glenn**, both of Shepherdstown, WV
(US)(73) **Assignees:** **Engelhard Corporation**, Iselin, NJ
(US); **The United States of America as represented by the Secretary of Agriculture**, Washington, DC (US)(*) **Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

This patent is subject to a terminal disclaimer.

(21) **Appl. No.:** **09/553,538**(22) **Filed:** **Apr. 20, 2000****Related U.S. Application Data**

(63) Continuation of application No. 08/972,648, filed on Nov. 18, 1997, now Pat. No. 6,154,327, which is a continuation-in-part of application No. 08/812,301, filed on Mar. 5, 1997, now Pat. No. 5,908,708.

(51) **Int. Cl.⁷** **A01N 25/04; A01N 25/32**(52) **U.S. Cl.** **424/405; 47/2; 47/58.1; 47/DIG. 11; 424/421; 424/DIG. 10; 427/384; 427/393.1; 428/541; 504/362; 516/77; 516/88; 516/104**(58) **Field of Search** **47/2, 58.1, DIG. 11; 427/384, 393.4; 428/541; 516/79, 88, 104; 424/405, 421, DIG. 10; 504/362**(56) **References Cited****U.S. PATENT DOCUMENTS**

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Primary Examiner—Richard D. Lovering(74) **Attorney, Agent, or Firm**—Raymond F. Keller

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ABSTRACT

Disclosed are horticultural substrates coated with a particulate membrane and a method for controlling pests and providing enhanced horticultural effect by applying a particulate membrane to the surface of the horticultural substrate.

39 Claims, 6 Drawing Sheets

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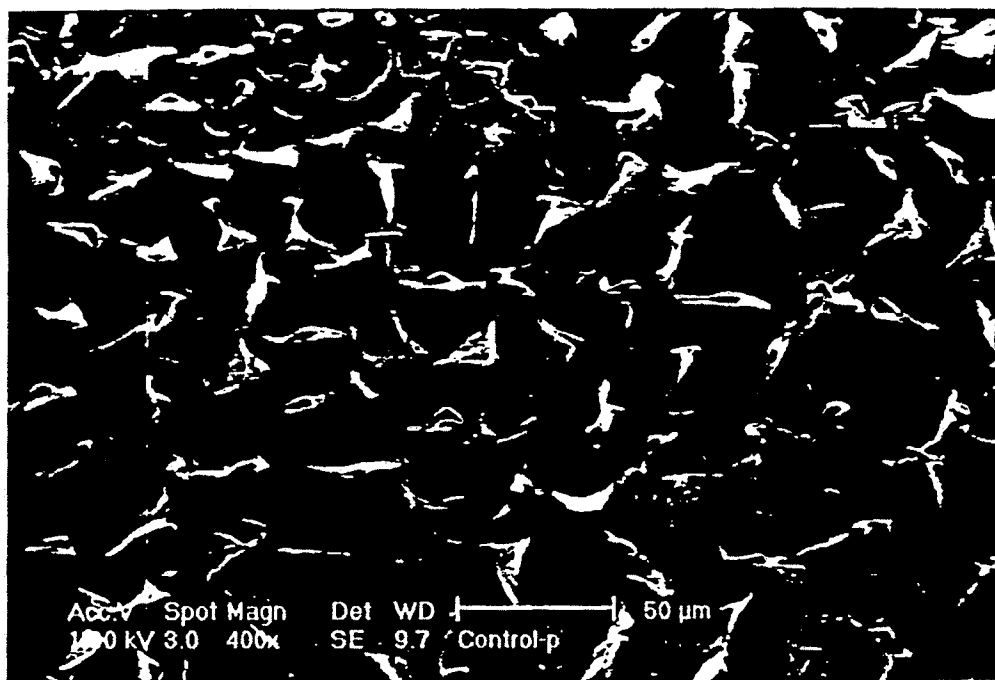
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Figure 1



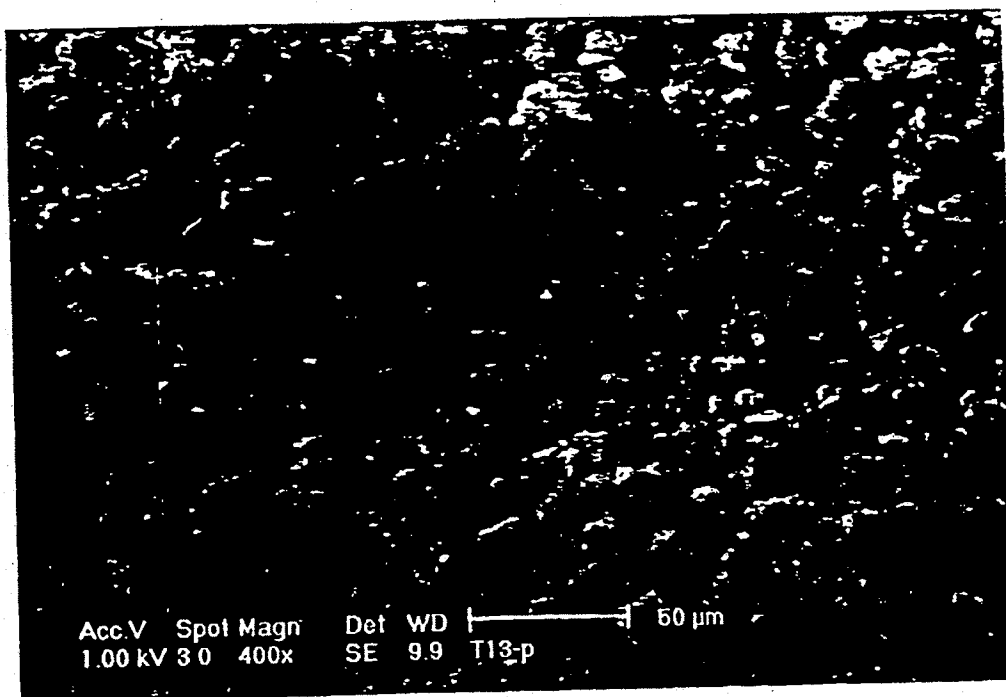
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Figure 2



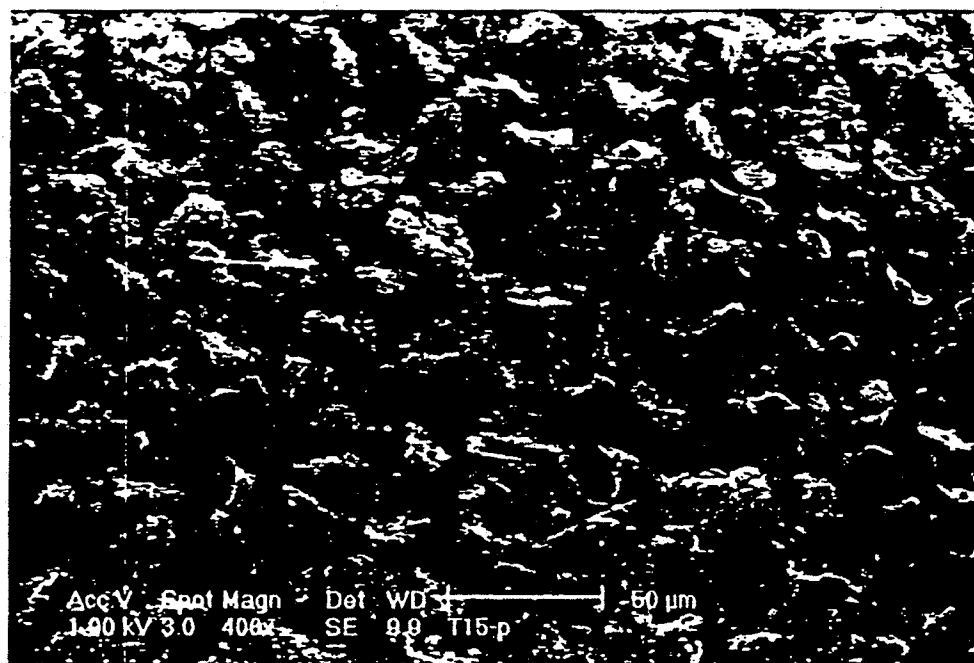
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Figure 3



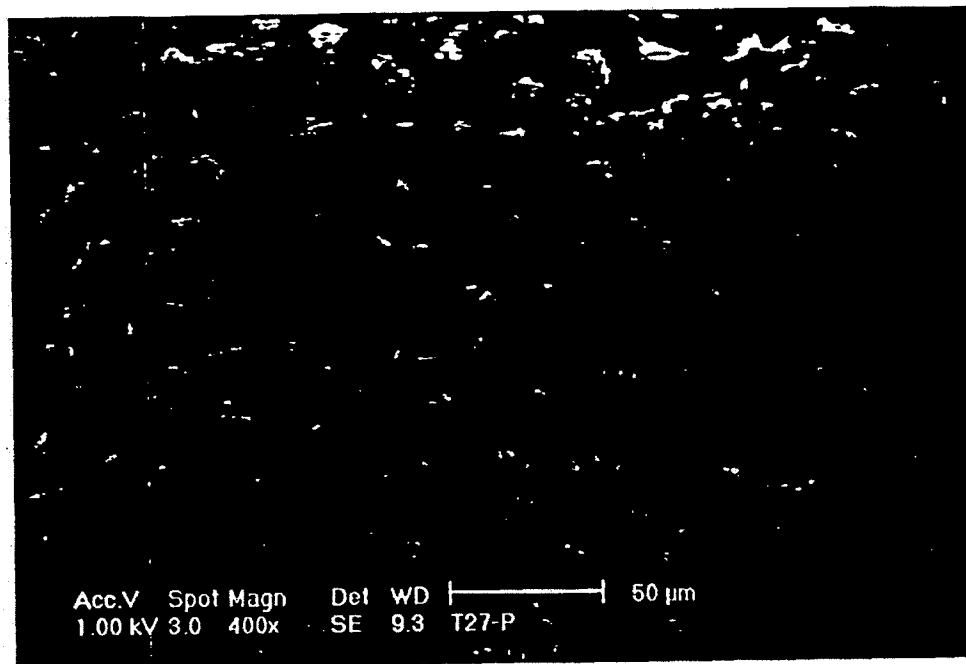
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Figure 4



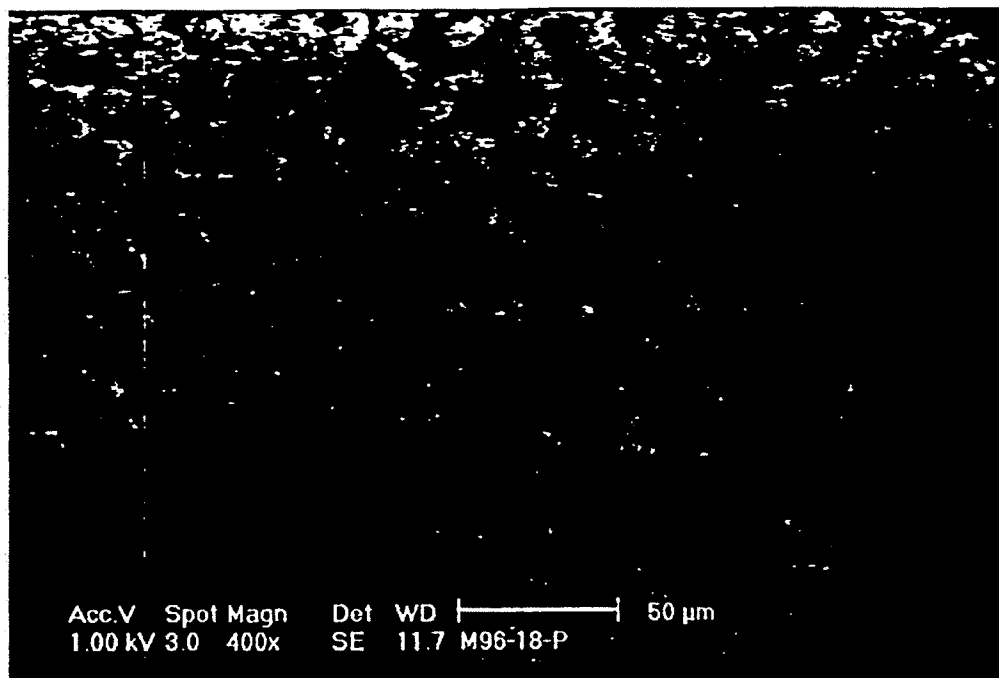
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Figure 5



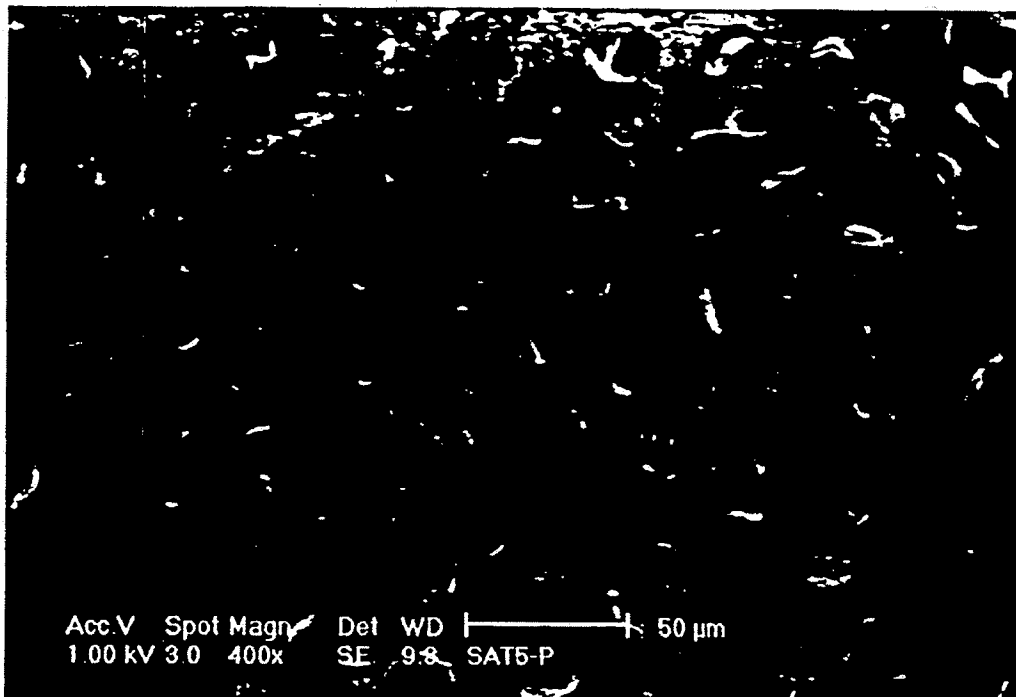
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Figure 6



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TREATED HORTICULTURAL SUBSTRATES

This application is a continuation of U.S. patent application Ser. No. 08/972,648, filed Nov. 18, 1997 now U.S. Pat. No. 6,156,327, which is a continuation-in-part of U.S. patent application Ser. No. 08/812,301, filed Mar. 5, 1997 now U.S. Pat. No. 5,908,708.

FIELD OF THE INVENTION

The present invention is directed to horticultural substrates treated with a particulate membrane and methods for controlling pests associated with such substrates and for providing enhanced horticultural effects.

BACKGROUND OF THE INVENTION

The prior art has discussed the use of certain inert particulate solids as insecticides, see for example; Driggers, B. F., "Experiments with Talc and Other Dusts Used Against Recently Hatched Larvae of the Oriental and Codling Moths," *J. Econ. Ent.*, 22 327-334 (1929); Hunt, C. R., "Toxicity of Insecticide Dust Diluents and Carriers to Larvae of the Mexican Bean Beetle," *J. Econ. Ent.*, 40 215-219 (1947); P. Alexander, J. A. Kitchener and H. V. A. Briscoe, "Inert Dust Insecticides," Parts I, II, and III, *Ann. Appl. Biol.*, 31 143-159, (1944); and U.S. Pat. No. 3,159,536 (1964) and U.S. Pat. No. 5,122,518 (1992), each of which is incorporated herein by reference with regard to its teachings relating to particulate materials.

Plant diseases are caused by various pathogens, e.g., fungi, bacteria and virus, and these diseases have generally been controlled commercially by the use of chemical pesticides. For example, commercial fungicides generally belong to the following types of chemical compounds: inorganic (copper or sulfur based), organic (anilines, anilides, dithiocarbamates, halogen compounds and heterocyclic nitrogen compounds), antibiotics and biologicals. Chemically toxic fungicides and bactericides are often formulated with inert particulates. Inert particulates, however, have been shown to be ineffective toward these plant pests when applied by themselves (see W. O. Cline and R. D. Milholland, "Root Dip Treatments for Controlling Blueberry Stem Blight Caused by *Botryosphaeria dothidea* in Container-Grown Nursery Plants," *Plant Disease* 76 136-138 (1992)). Furthermore, not only have inert particulates been shown to be ineffective in plant disease control, but it has been reported by S. K. Bhattacharyya and M. K. Basu, "Kaolin Powder as a Fungal Carrier," *Appl. Envir. Microb.* 44 751-753 (1982) that kaolin powder may be used to carry and preserve an *Aspergillus* sp. for at least 90 days. In another report, S. M. Lipson and G. Stotzky, "Effect of Kaolinite on the Specific Infectivity of Reovirus," *FEMS Micr. Let.* 37 83-88 (1986), it was reported that the infectivity of enteric viruses (e.g., poliovirus, rotavirus and reovirus) is prolonged when these viruses are adsorbed on naturally occurring particulates (sediments, clay materials) in terrestrial and aquatic environments.

O. Ziv and R. A. Frederiksen, "The Effect of Film-forming Anti-transpirants on Leaf Rust and Powdery Mildew Incidence on Wheat," *Plant Path.* 36 242-245 (1987); M. Kamp, "Control of *Erysiphe cichoracearum* on *Zinnia elegans*, with a Polymer-based Antitranspirant," *Hort. Sci.* 20 879-881 (1985); and J. Zekaria-Oren and Z. Eyal, "Effect of Film-forming Compounds on the Development of Leaf Rust on Wheat Seedlings," *Plant Dis.* 75 231-234 (1991)) discuss the use of anti-transpirant polymer films to control disease. Of course, the use of anti-transpirants is undesirable

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because they reduce the exchange of necessary gases on the surface of living plants.

For prior art regarding horticultural effects see, for example, Byers, R. E., K. S. Yoder, and G. E. Mattus, "Reduction in Russetting of 'Golden Delicious' Apples with 2,4,5-TP and Other Compounds," *HortScience* 18:63-65; Byers, R. E., D. H. Carbaugh, and C. N. Presley, "Stayman' Fruit Cracking as Affected by Surfactants, Plant Growth Regulators, and Other Chemicals," *J. Amer. Soc. Hort. Sci.* 115:405-411 (1990); Durner, E. F., and T. J. Gianfagna, "Peach Pistil Growth Inhibition and Subsequent Bloom Delay by Midwinter Bud Whitewashing," *HortScience* 25:1222-1224 (1990); and M. N. Westwood, *Temperate-zone Pomology*, page 313 W. H. Freeman and Co. (1978).

Therefore, there is still a need for cost effective inert, nontoxic improved agents for pest control and for enhanced horticultural effects and methods for their use.

SUMMARY OF THE INVENTION

This invention relates to horticultural substrates where the surface of said substrates is coated with a particulate membrane and to methods for pest control and enhanced horticultural effects by forming said membrane on the surface of the horticultural substrate.

In one embodiment, this invention relates to coated substrates comprising a horticultural substrate wherein the surface of said substrate is coated with a membrane comprising one or more particulate layers, said layers comprising one or more particulate materials, said particulate materials being finely divided, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

In another embodiment, this invention relates to a method for pest control on horticultural substrates which comprises forming on the surface of said substrate a membrane comprising one or more particulate layers, said layers comprising one or more particulate materials, said particulate materials being finely divided, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

In still another embodiment, this invention relates to a method for providing enhanced horticultural effects which comprises forming on the surface of a horticultural substrate a membrane comprising one or more particulate layers, said layers comprising one or more particulate materials, said particulate materials being finely divided, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a scanning electron micrograph of an untreated petunia petal.

FIG. 2 is a scanning electron micrograph of a petunia petal coated with a membrane of octylsilane treated calcined kaolin particles.

FIG. 3 is a scanning electron micrograph of a petunia petal coated with a membrane of vinyl silane calcined kaolin particles.

FIG. 4 is a scanning electron micrograph of a petunia petal coated with a membrane of methylethoxysiloxane treated calcined kaolin particles.

FIG. 5 is a scanning electron micrograph of a petunia petal coated with a membrane of a calcined kaolin treated with a siloxane material.

FIG. 6 is a scanning electron micrograph of a petunia petal coated with a membrane of a calcined kaolin.

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DETAILED DESCRIPTION OF THE
INVENTION

The horticultural substrates to which this invention relates are agricultural and ornamental crops, including those selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants.

The membranes of this invention comprise one or more particulate layers, said layers comprising one or more particulate materials, said particulate materials being finely divided.

The finely divided particulate materials which make up the particulate membrane of this invention may be hydrophilic or hydrophobic materials and the hydrophobic materials may be hydrophobic in and of themselves, e.g., mineral talc, graphite, and Teflon® or may be hydrophilic materials that are rendered hydrophobic by application of an outer coating of a suitable hydrophobic wetting agent (e.g., the particulate material has a hydrophilic core and a hydrophobic outer surface).

Typical particulate hydrophilic materials useful for the purposes of this invention include: minerals, such as calcium carbonate, talc, kaolin (both hydrous and calcined kaolins, with calcined kaolins being preferred), bentonites, clays, attapulgite, pyrophyllite, wollastonite, silica, feldspar, sand, quartz, chalk, limestone, precipitated calcium carbonate, diatomaceous earth and barytes; functional fillers such as microspheres (ceramic, glass and organic), aluminum trihydrate, pyrogenic silica, ceramic fibers and glass fibers; and pigments such as colorants or titanium dioxide.

The surfaces of such materials can be made hydrophobic by addition of hydrophobic wetting agents. Many industrial mineral applications, especially in organic systems such as plastic composites, films, organic coatings or rubbers, are dependent upon just such surface treatments to render the mineral surface hydrophobic; see, for example, Jesse Edenbaum, *Plastics Additives and Modifiers Handbook*, Van Nostrand Reinhold, New York, 1992, pages 497-500 which is incorporated herein by reference for teachings of such surface treatment materials and their application. So-called coupling agents such as fatty acids and silanes are commonly used to surface treat solid particles as fillers or additives targeted to these industries. Such hydrophobic agents are well known in the art and common examples include: chrome complexes such as Volvan® and Quilon® obtained from DuPont; organic titanates such as Tilcom® obtained from Tioxide Chemicals; organic zirconate or aluminate coupling agents obtained from Kenrich Petrochemical, Inc.; organofunctional silanes such as Silquest® products obtained from Witco or Prosil® products obtained from PCR; modified silicone fluids such as the DM-Fluids obtained from Shin Etsu; and fatty acids such as Hystrene® or Industrene® products obtained from Witco Corporation or Emersol® products obtained from Henkel Corporation (stearic acid and stearate salts are particularly effective fatty acids and salts thereof for rendering a particle surface hydrophobic).

Examples of preferred particulate materials suitable for the purposes of this invention that are commercially available from Engelhard Corporation, Iselin, N.J. are the siloxane treated calcined kaolins sold under the trademark Translink®, and stearic acid treated ground calcium carbonates commercially available from English China Clay under the trademarks Supercoat® and Kotamite®.

The term "finely divided" when utilized herein means that the particulate materials have a median individual particle size below about 10 microns and preferably below about 3 microns and more preferably the median particle size is

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about one micron or less. Particle size and particle size distribution as used herein are measured with a Micromeritics Sedigraph 5100 Particle Size Analyzer. Measurements were recorded in deionized water or hydrophilic particles.

Dispersions were prepared by weighing 4 grams of dry sample into a plastic beaker adding dispersant and diluting to the 80 ml mark with deionized water. The slurries were then stirred and set in an ultrasonic bath for 290 seconds. Typically, for kaolin 0.5% tetrasodium pyrophosphate is used as a dispersant; with calcium carbonate 1.0% Calgon T is used. Typical densities for the various powders are programmed into the sedigraph, e.g., 2.58 g/ml for kaolin. The sample cells are filled with the sample slurries and the X-rays are recorded and converted to particle size distribution curves by the Stokes equation. The median particle size is determined at the 50% level.

Preferably, the particulate material has a particle size distribution wherein up to 90% by weight of the particles have a particle size of under about 10 microns, preferably below about 3 microns and more preferably about one micron or less.

The particulate materials particularly suitable for use in this invention are inert, nontoxic and hydrophobic.

As used herein "inert" particulate materials are particles that are not physiological poisons, that is, the particulate materials of this invention do not, as their primary function, kill pests. While not being bound by theory, it is believed that the pest control of this invention is achieved primarily by prophylactic means rather than primarily through the destruction of the unwanted pests.

The particulate materials are preferably nontoxic meaning that in the limited quantities needed for effective pest control or enhanced horticultural effect such materials are not considered harmful to horticultural substrate, animals, the environment, the applicator and the ultimate consumer.

The preferred particulate materials of the instant invention are hydrophobic. Hydrophobicity refers to the physical property of a surface to dislike or repel water. Most mineral particle surfaces are hydrophilic, i.e., water liking. The terms hydrophobic and hydrophilic are not always accurately used in the literature and both are often confused with similar terms such as, lipophilic or lipophobic, oleophilic or oleophobic, lyophilic or lyophobic, and polar or nonpolar. Hydrophobicity can be described in more quantitative terms by using contact angle measurements. The contact angle is defined by the equilibrium forces that occur when a liquid sessile drop is placed on a smooth surface. The tangent to the surface of the convex liquid drop at the point of contact among the three phases, solid (S), liquid (L) and vapor (V) is the contact angle θ .

The relationship between the surface tension of the solid-vapor (γ_{SV}), liquid-vapor (γ_{LV}) and solid-liquid (γ_{SL}) can be defined by the following Young's equation:

$$F = \gamma p \cos \theta$$

where F =wetting force; γ =liquid surface tension; and p =wetting perimeter.

If the water droplet spreads out on the surface the contact angle is less than 90 degrees and the surface is hydrophilic. If the surface is hydrophobic then the contact angle is greater than 90 degrees. Thus, 180 degrees is the maximum hydrophobicity that a surface can have.

Many surfaces change their surface energy upon contact with water (see J. Domingue, Amer. Lab, October 1990). Dynamic contact angle measurements provide both an advancing and receding contact angle. The advancing contact angle is a measurement of the surface hydrophobicity

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upon initial contact with a liquid, while the receding contact angle measures the hydrophobicity after the surface has been wetted with a liquid. Thus, for the purposes of this invention, "hydrophobic" or "hydrophobicity," when used in reference to the particulate materials useful for the purposes of this invention, such particles may have either an advancing and/or receding contact angle of greater than 90°. Preferred materials have receding contact angles of greater than 90°.

The dynamic contact angles referred to herein are based on a gravimetric principle of the Wilhelmy plate technique and are determined by measurement on the Dynamic Contact Angle Instrument which can measure both advancing and receding contact angles of powdered samples. A dynamic contact angle analysis system (model DCA 315) from ATI Cahn Instruments Inc. was used for all contact angle measurements referred to and reported herein. The surface tension (γ) of deionized water was determined with a standard platinum calibration plate. Powder samples were deposited on dual sided adhesive tape. The perimeter (p) of the tape was determined with a caliper. The impregnated tape was placed in the DCA 315 and lowered and raised in the deionized water at a rate of 159 microns/second for two immersion cycles. The contact angles were determined from the advancing and receding wetting hysteresis curves of the first immersion cycle. Most samples were prepared and run in duplicate and the results averaged. The data analysis was made with a WinDCA software for Windows diagnostic package from the manufacturer, ATI Cahn Instruments Inc.

Representative contact angle values for a variety of inert particulate materials are given in Table I. Although many of the powders listed are hydrophilic and have advancing and receding contact angles less than 90°, some hydrophobic particles as measured by the advancing contact angle, for example talc, become hydrophilic upon wetting.

TABLE I

Contact Angle Values of Powders		
Particle	Advancing Contact Angle (°)	Receding Contact Angle (°)
Calcium Carbonate ¹	28.4	32.5
Calcium Carbonate ²	37.8	38.1
Calcium Carbonate ³ (ST)	180	171.1
Barytes ⁴	32.2	30.3
Mica ⁵	42.3	39.9
Mica ⁶	31.5	25.0
Silica ⁷	38.5	38.2
Diatomite ⁸	39.4	35.3
ATH ⁹	38.7	0
Wollastonite ¹⁰	23.1	27.5
Wollastonite ¹¹	9.4	14.1
Talc ¹²	180	12.8
Talc ¹³	159.2	11.5
Feldspar ¹⁴	35.9	39.2
Nepheline Syenite ¹⁵	19.4	25.4
Kaolin hydrous ¹⁶	29	30.1
Kaolin calcined ¹⁷	26	20.5

ST = Surface Treated

¹Atomite ® (ECC Int.) ²GS 6532 (Georgia Marble) ³Kotamite ® (ECC Int.) ⁴Bartex ® 65 (Hitox) ⁵WG 325 (KMG Minerals) ⁶C-3000 (KMG Minerals) ⁷Novacite ® L-207A (Malvern Min Co.) ⁸Diafil ® 340 (CR Mineral Corp.) ⁹Alcan ® SF (Alcan Chemicals) ¹⁰NYAD ® 1250 (NYCO) ¹¹Wollastokup ® (NYAD) ¹²Vantalc ® 6H (RT Vanderbilt)

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TABLE I-continued

Contact Angle Values of Powders		
Particle	Advancing Contact Angle (°)	Receding Contact Angle (°)
¹³ Vertal ® 710 (Luzenac Amer Inc.) ¹⁴ Minspar ® 4 (K-T Feldspar Corp)		
¹⁵ Minex ® 10 (Unimin) ¹⁶ ASP ® 900 (Engelhard Corp) ¹⁷ Satintone ® W (Engelhard Corp)		

Hydrophilic surfaces can be made hydrophobic by addition of hydrophobic wetting agents as shown in Table II for hydrous and calcined kaolin. However, not all hydrophobic surface treatments render hydrophobicity to a particle as shown in Table II.

TABLE II

Surface Treated Kaolin Particles				
Surface Treatment (1%)	Hydrous Kaolin Advancing Angle (°)	Hydrous Kaolin Receding Angle (°)	Calcined Kaolin Advancing Angle (°)	Calcined Kaolin Receding Angle (°)
no treatment	31	30	26	21
Stearic acid ¹	155.5	0	166	102
Octyltriethoxysilane ²	158	0	180	180
Vinyltriethoxysilane ³	120	22	164	140
polydimethylsiloxane ⁴	27	26	24	26
linear methylethoxysiloxane polymer ⁵	89	24	180	154
polydimethyl siloxane cyclic ⁶	112	45	155	154

¹Industrene 7018 (Witco) ²A-137 (Witco) ³A-151 (Witco) ⁴L-45 (Witco)

⁵A-272 (Witco) ⁶CG-4491 (HULS America Inc.)

The preferred hydrophilic core particles are those, which when treated with a hydrophobic wetting agent and are applied to the surface of a horticultural substrate, form a membrane on the substrate. Examples of such particles are calcium carbonate and kaolin. Calcined kaolin is preferred to hydrous kaolin.

As previously discussed, this invention relates to horticultural substrates wherein the surface of said substrate is coated with a membrane comprising one or more particulate layers. This membrane allows for the exchange of gases on the surface of said substrate. The gases which pass through the membrane are those which are typically exchanged through the surface skin of living plants. Such gases typically include water vapor, carbon dioxide, oxygen, nitrogen and volatile organics.

The portion of a substrate to be covered with said membrane is within the skill of the ordinary artisan. Optimally, the substrate is fully covered with said membrane, and although diminished disease control and/or horticultural effects may result, less than full substrate coverage is within the scope of this invention; preferably, however, the substrate is substantially covered. Reference is made to U.S. Ser. No. 08/972,659, filed concurrently herewith on Nov. 18, 1997, entitled "Method for Providing Enhanced Photosynthesis" now U.S. Pat. No. 6,110,867 and to U.S. Ser. No. 08/972,653 filed concurrently herewith on Nov. 18, 1997, entitled "Method for Protecting Surfaces from Arthropod Infestation" now U.S. Pat. No. 6,027,740 which are incorporated herein by reference for their teachings regarding methods for insect control and improved photosynthesis. Preferably, the membranes of this invention are sufficiently continuous so as to provide effective control of disease. The membrane may have imperfections such as gaps or voids, but such imperfections should not be so large as to materi-

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ally affect the disease control of such membrane. Such gaps or voids typically will not exceed about 5μ , and are preferably less than 1μ . In another preferred embodiment, the membrane is water repellent. The membrane may be formed by applying one or more layers of finely divided particulate material until a membrane is formed of sufficient thickness and continuity to be an effective disease control barrier, i.e., the particles on the surface of the substrate are so closely associated that pathogens are unable to penetrate the particulate coating and infect the underlying horticultural substrate. For example, this can typically be accomplished by applying in a uniform manner from about 25 up to about 3000 micrograms of particulate material/cm² of substrate for particles having specific density of around 2–3 g/cm³. In addition, environmental conditions such as wind and rain may reduce coverage of the membrane and, therefore, it is within the scope of this invention to apply the particles one or more times during the growing season of said horticultural crop so as to maintain the desired effect of invention.

This particulate membrane may be prepared by applying a slurry of finely divided particles in a volatile liquid such as water, a low boiling organic solvent or low boiling organic solvent/water mixture. One or more layers of this slurry can be sprayed or otherwise applied to the substrate. The volatile liquid is preferably allowed to evaporate between coatings. Surfactants or dispersants may be useful in preparing an aqueous slurry of the particulate materials of this invention. The membrane of this invention may be hydrophilic or hydrophobic, but is preferably hydrophobic. Normal dusting of particles, aside from not being commercially practical on a large scale due to drift and inhalation hazards, is not effective at forming a membrane on a horticultural substrate suitable for disease control. The membrane of this invention may be formed, however, by carefully applying the finely divided particles to the substrate, e.g., with a paint brush. While not being bound by theory, it is believed that the one or more layers of finely divided particulate material form a membrane due to particle to particle cohesion of evenly distributed, closely associated particles.

The low boiling organic liquids useful in the present invention are preferably water-miscible and contain from 1 to 6 carbon atoms. The term "low boiling" as used herein shall mean organic liquids which have a boiling point generally no more than 100° C. These liquids enable the particulate solids to remain in finely divided form without significant agglomeration. Such low boiling organic liquids are exemplified by: alcohols such as methanol, ethanol, propanol, i-propanol, i-butanol, and the like, ketones such as acetone, methyl ethyl ketone and the like, and cyclic ethers such as ethylene oxide, propylene oxide and tetrahydrofuran. Combinations of the above-mentioned liquids can also be employed. Methanol is the preferred low boiling organic liquid.

Low boiling organic liquids may be employed in applying the particles to form the membranes of this invention. Typically, the liquids are used in an amount sufficient to form a dispersion of the particulate material. The amount of liquid is typically up to about 30 volume percent of the dispersion, preferably from about 3 up to about 5 volume percent, and most preferably from about 3.5 to about 4.5 volume percent. The particulate material is preferably added to a low boiling organic liquid to form a slurry and then this slurry is diluted with water to form an aqueous dispersion. The resulting slurry retains the particles in finely divided form wherein most of the particles are dispersed to a particle size of less than about 10 microns.

This invention also provides methods for pest control and enhanced horticultural effects by forming said membrane on

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the surface of the horticultural substrate. The foregoing discussions regarding i) said membrane being comprised of one or more layers of particulate material, ii) said particulate material being finely divided, iii) said membrane allowing for the transpiration of water vapor from said substrate through said membrane, and iv) application techniques for applying said layers to the horticultural substrate, as well as the specific embodiments discussed herein, also apply to these methods.

The pests controlled by this invention refer to arthropods including insects, mites, spiders and related animals and diseases of various pathogens such as fungi, bacteria and virus. Diseases can be transmitted in a number of ways such as wind currents, water splash and/or arthropod transmission. Examples of diseases commonly caused by wind currents and water splash include: Fire blight (bacteria—*Erwinia amylovora*), apple scab (fungus—*Venturia inaequalis*), Potato Blight (fungus—*Phytophthora infestans*), Soft rots (fungus—*Botrytis cinerea*), Leaf blight and leaf spot (bacteria—*Xanthomonas* sp.), and bacterial leaf spot and leaf blight (bacteria—*Pseudomonas* sp.). Examples of diseases commonly caused by arthropod transmission are the fungus disease, Dutch Elm disease, of American Elm by the European elm beetle; the bacterial disease, Fire blight, of apples and pears by flies, beetles and other insects; the virus disease, Curly Top, of sugar beets by the beet leaf hopper. Disease control also applies to those secondary infections of wound sites on a plant that results from arthropod feeding such as brown rot infection of stone fruits that results when the disease organism enters the plant through plum curculio feeding sites.

This invention can also provide the benefit of enhanced horticultural effects including improved color, smoother fruit surface, increased soluble solids, e.g., sugars, acidity, etc., reduced bark and fruit cracking, reduced plant temperature and reduced russetting.

The following examples are illustrative of embodiments of the invention and are not intended to limit the invention as encompassed by the claims forming part of the application.

EXAMPLE 1

This example demonstrates that coating a plant substrate with a membrane of finely divided particles greatly reduces the degree of infection as compared to a substrate not coated with a particulate membrane. Efficacy of various particulate membranes toward disease control was demonstrated by screening evaluations of *Botrytis cinerea* on strawberry petals (*Fragaria x ananassa* Pucheene). All preparations in Tables III and IV were made by applying suspensions of the particles listed in the table prepared by first dispersing 5 grams of the identified particle in 10 ml methanol which is then brought to 100 ml with deionized water. Petals were then sprayed with this suspension using a Paasche air brush to run off. The petals were allowed to air dry and then 10 μ l of *Botrytis inoculum* (3.6×10^7 spores/ml) was added over the petals. The petals were then incubated in a 100% humidity chamber for 24 hours.

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TABLE III

Fungus Efficacy of surface treated and untreated particles			
Particle	% Infection after 24 hrs	Advancing Contact Angle (°)	Receding Contact Angle (°)
Control - no particles	88.9	—	—
Methanol	76.5	—	—
Kaolin hydrous [ST] ¹	73.0	155.5	0
Kaolin calcined ²	68.0	19.4	20.5
Kaolin hydrous ³	63.8	29	30.1
Kaolin calcined [ST] ⁴	62.0	166	102
Calcium Carbonate ⁵	57.0	28.4	32.5
Talc ⁶	49.3	180	12.8
Calcined Kaolin [ST] ⁷	44.7	146	128
Calcium Carbonate [ST] ⁸	36.8	180	171
Translink® 77	23.6	153	120

¹ASP® 900 (Engelhard Corporation) treated with stearate ²Satintone® W (Engelhard Corporation) ³ASP® 900 (Engelhard Corporation) ⁴Satintone® W (Engelhard Corporation) treated with stearate ⁵Atomite® (ECC Int.) ⁶Vantaic® 6H (RT Vanderbilt) ⁷Translink® 37 (Engelhard Corporation) ⁸Kotamite® (ECC Int.) Data are the mean of 3 independent replications, each containing 10 strawberry petals.

Infection was measured by the presence of a necrotic lesion characteristic of a Botrytis infection. The data was analyzed by the Duncan's multiple range test (P=0.05) on the arcsine transformed percentages and are presented as untransformed mean for convenience).

EXAMPLE 2

Performing the same evaluations and comparing surface treated particles to untreated particles of calcined kaolin gave the results in Table IV.

TABLE IV

Fungus Efficacy of surface treated calcined kaolin				
Particles	% Infection after 24 hrs	Advancing Contact Angle (°)	Receding Contact Angle (°)	Character of particle coating
Control - no particles	88	—	—	FIG. 1
octylsilane treated ¹	25	180	180	FIG. 2
Satintone® W vinylsilane treated ²	29	164	140	FIG. 3
Satintone® W methylethoxysiloxane treated ³	25	180	154	FIG. 4
Satintone® W Translink® 77	0	153	120	FIG. 5
Satintone® W	—	—	—	FIG. 6

¹1% A-137 (Witco) ²1% A-151 (Witco) ³1% A-272 (Witco)

Scanning electron micrographs shown in FIGS. 1-6 were collected with a Philips XL 30 FEG scanning electron microscope (SEM) at 1 Kv accelerating voltage and 1x10⁻⁵ mbar vacuum. Samples of petunia petals were coated with particle membranes as described in Example 1 and placed in the instrument without any additional sample preparation. The vacuum caused a collapse of the surface irregularities of the petal substrate, but did not affect the particle membranes as illustrated in FIGS. 2-6. All images are presented at 400x magnification.

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FIG. 1 illustrates the uneven surface of an uncoated petunia petal. Under an ordinary optical go microscope one observes a surface containing many peaks and valleys. These peaks are collapsed under the conditions necessary to collect the SEM image. Ordinary optical images, however, often do not show the membrane surface because the membranes are very thin and transparent to visible light. SEM techniques, however, can capture an image of the surface of such membranes.

FIGS. 2-4 illustrate the membrane surface prepared from calcined kaolin particles (1.2 micron median particle size) treated with the various hydrophobic wetting agents listed in Table IV.

FIG. 5 illustrates the surface of the membrane prepared from Translink® 77 which has fewer and smaller voids than those voids appearing in FIGS. 2-4.

FIG. 6 illustrates the surface of the membrane prepared from the same calcined kaolin particles (0.8 micron median particle size) used in the manufacture of Translink® 77. The image clearly shows regularly spaced large voids on the order of 20 microns diameter.

EXAMPLE 3

"Seckel" pear trees received the following treatments: 1) conventional pesticide applications applied according to the presence of economic levels of pests using the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) no treatment, 3) weekly application of Translink® 77 beginning in Apr. 29, 1997, 4) weekly application of calcined kaolin (Satintone® 5HP), beginning in Apr. 29, 1997, 5) weekly application of treated calcium carbonate (SuperCoat®—commercially available from English China Clay) beginning in Apr. 29, 1997, 6) weekly application of Translink 37® beginning in Apr. 29, 1997. Treatments (3), (5) and (6) applied 25 pounds material suspended in 4 gal methanol and added to 100 gal water. Treatment (4) applied 25 pounds material suspended in 100 gal water with the addition of 27 oz Ninex® MT-603 and 2 pints Toximul. These treatments were applied at the rate of 125 gal/acre using an orchard sprayer. This mixture was applied at the rate of 125 gal/acre using an orchard sprayer. Treatments ended Sep. 15, 1997. The treatments were arranged in a randomized complete block design with 2 replications and 4 trees/plot. A freeze of 25° F. occurred on Oct. 23, 1997 and freeze damage of foliage was evaluated on Oct. 28, 1997. Freeze damage was evaluated by collecting 40 leaves/plot (10 from each tree). Leaves with necrosis on the leaf margin to the midvein that extended to the abaxial side of the leaf exhibited freeze damage. Undamaged leaves lacked this necrosis. Each leaf was categorized as damaged or undamaged and percentage undamaged from each plot calculated. Data were analyzed using Analysis of variance using a randomized complete block design.

TABLE V

Treatment	Undamaged leaves (%)
Conventional	2.5
Control	2.5
Translink 77	81.5
Satintone 5HP	11.5
Supercoat	67.0
Translink 37	69.0

These data demonstrate that freeze damage was extensive when no particles were applied (conventional and control, 2.5% each). Freeze damage was extensive when a hydrophilic particle was applied to the tree (Satintone 5HP,

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11.5%). Freeze damage was moderated when hydrophobic particles were applied to the trees (Translink 77, Supercoat, and Translink 37, 81.5%, 67%, and 69%, respectively). These data demonstrate that the presence of a hydrophobic particle membrane will moderate freeze damage.

EXAMPLE 4

"Red Delicious" apple trees received the following treatments: 1) Conventional pesticide applications applied according to the presence of economic levels of pests using the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) no treatment, 3) weekly application of Translink® 77 beginning in Mar. 11, 1997, 4) weekly application of calcined kaolin (Satintone® 5HP) beginning in Apr. 29, 1997, and 5) weekly application of treated calcium carbonate (SuperCoat®—commercially available from English China Clay) beginning in Apr. 29, 1997. Treatments (3), and (5) applied 25 pounds material suspended in 4 gal methanol and added to 100 gal water. Treatment (4) applied 25 pounds material suspended in 100 gal water with the addition of 27 oz Ninex® MT-603 and 2 pints Toximul. These treatments were applied at the rate of 125 gal/acre using an orchard sprayer. This mixture was applied at the rate of 125 gal/acre using an orchard sprayer. The treatments were arranged in a randomized complete block design with 4 replications and 3 trees/plot. Treatments were not irrigated and received 21.58 cm of precipitation from May 1 to Aug. 30, 1997. Fruit were harvested at maturity; fruit number were measured at harvest. Data were analyzed using Analysis of variance using a randomized complete block design.

TABLE VI

Treatment	Fruit number/tree
Conventional	322
Control	246
Translink 77	382
applied 3/11/97	
Satintone 5HB	302
applied 4/29/97	
Supercoat applied	301
4/29/97	

The weekly application of Translink® 77 before bud break and the occurrence of a severe frost on Apr. 9, 1997 with a minimum temperature of 20° F., moderated the frost damage as demonstrated by a larger number of fruit (382) reaching maturity compared to Satintone® HB (302) or Supercoat® (301). The weekly application of Translink® 77 before bud break also moderated the frost damage to fruit compared to the conventional treatment and the untreated control (322 and 246 respectively), neither of, which received any pesticide applications prior to the frost. The application after the frost of Supercoat®, a hydrophobic particle, or Satintone® 5HB, a hydrophilic particle, did not increase the number of fruit/tree.

EXAMPLE 5

"Golden Delicious" apples received 3 treatments: 1) commercial pesticide application applied according to the presence of economic levels of pests using the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) full rate of Translink® 77, and 3) half rate of Translink® 77. Treatments (2) and (3) applied 25 and 12.5

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pounds material, respectively, suspended in 4 and 2 gal methanol, respectively, and added to 100 gal water. This mixture was applied at the rate of 200 gal/acre using an orchard sprayer. The treated area was approximately 1 acre plots with 2 replications of each treatment in a randomized block design. At harvest the plots were commercially harvested and processed by a commercial grading line. At the time of grading, 100 fruit from each plot were randomly chosen to determine surface defects. The data are reported in Table VII.

TABLE VII

Treatment	Russetting downgrade (%)
Translink ® 77 full rate	3.3
Translink ® 77 half rate	3.9
Conventional	13.8

Application of Translink® 77 at the full and half rate reduced russetting on the apple surface compared to the conventional treatment.

EXAMPLE 6

"Stayman" apples received 2 treatments: 1) commercial pesticide application applied according to the presence of economic levels of pests using the Virginia, West Virginia and Maryland Cooperative Extension 1997 Spray Bulletin for Commercial tree Fruit Growers publication 456-419, 2) Translink® 77 treatment applied 25 pounds material suspended in 4 gal methanol and added to 96 gal water. This mixture was applied at the rate of 200 gal/acre using an orchard sprayer. Each treatment was applied to 1 acre blocks with no randomization. Apples were harvested commercially and processed on a commercial grading line. Data presented represent percent packout from the commercial grading line. At time of grading 100 fruit each treatment were randomly chosen to evaluate for surface defects. Cracking percentage was the percentage of fruit with visible cracks in the fruit. The data are reported in Table VIII.

TABLE VIII

Treatment	Fruit cracking (%)
Translink ® 77	2
Conventional	22

The application of Translink® 77 decreased the cracking of apple fruit compared to the conventional treatment.

What is claimed is:

1. A coated substrate comprising a horticultural substrate selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants wherein the surface of said substrate is coated with a membrane formed from a slurry comprising water and one or more particulate materials, the membrane comprised of one or more particulate layers, said layers comprising one or more particulate materials selected from the group consisting of calcium carbonate, hydrous kaolin, calcined kaolin and mixtures thereof, said particulate materials being finely divided, and wherein said membrane allows for the exchange of gases on the surface of said substrate and the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

2. The coated substrate of claim 1 wherein said particulate materials are hydrophobic.

3. The coated substrate of claim 1 wherein said particulate material has a Receding Contact Angle of greater than 90°.

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4. The coated substrate of claim 1 wherein the particulate material has a particle size distribution wherein up to 90% of the particles have a particle size of under about 10 microns.

5. The coated substrate of claim 1, wherein the particulate materials comprise one of calcium carbonate and hydrous kaolin, hydrous kaolin and calcined kaolin, calcium carbonate and calcined kaolin.

6. The coated substrate of claim 1 wherein said hydrophobic outer surface materials are selected from the group consisting of chrome complexes, organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

7. The coated substrate of claim 1 wherein the substrate is selected from the group consisting of agricultural and ornamental crops.

8. The coated substrate of claim 1 wherein the substrate is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants.

9. The coated substrate of claim 1 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

10. The coated substrate of claim 1 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

11. A coated substrate comprising a horticultural substrate selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants wherein the surface of said substrate is coated with a membrane comprised of one or more particulate layers, said layers comprising one or more hydrophobic particulate materials, said hydrophobic particulate materials comprising i) a hydrophilic core selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof and i) a hydrophobic outer surface, said particulate materials have a median individual particle size of about one micron or less, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

12. A method for pest control on horticultural substrates selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants which comprises applying a slurry comprising water and one or more particulate materials, selected from the group consisting of calcium carbonate, hydrous kaolin, calcined kaolin and mixtures thereof, to the surface of said substrate to form a membrane comprised of one or more particulate layers, said layers comprising the particulate materials, said particulate materials being finely divided, and wherein said membrane allows for the exchange of gases on the surface of said substrate and the membrane is hydrophobic.

13. The method of claim 12 wherein said particulate material has a Receding Contact Angle of greater than 90°.

14. The method of claim 12 wherein the particulate material has a particle size distribution wherein up to 90% of the particles have a particle size of under about 10 microns.

15. The method of claim 12, wherein the particulate materials comprise one of calcium carbonate and hydrous kaolin, hydrous kaolin and calcined kaolin, calcium carbonate and calcined kaolin.

16. The method of claim 12 wherein the substrate is selected from the group consisting of agricultural and ornamental crops.

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17. The method of claim 12 wherein the substrate is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants.

18. The method of claim 12 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

19. The method of claim 12 wherein the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

20. The method of claim 19 wherein said hydrophobic outer surface materials are selected from the group consisting of chrome complexes, organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

21. The method of claim 19 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

22. A method for pest control on horticultural substrates selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants, which comprises forming on the surface of said substrate a membrane comprised of one or more particulate layers, said layers comprising one or more hydrophobic particulate materials, said hydrophobic particulate materials comprising i) a hydrophilic core selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof, and ii) a hydrophobic outer surface, said particulate materials have a median individual particle size of about one micron or less, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

23. A method for enhancing the horticultural effect of horticultural substrates selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants which comprises applying a slurry comprising water, a surfactant, and one or more particulate materials, selected from the group consisting of calcium carbonate, hydrous kaolin, calcined kaolin and mixtures thereof, to the surface of said substrate to form a membrane comprised of one or more particulate layers and the surfactant, said layers comprising one or more particulate materials, said particulate materials being finely divided, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

24. The method of claim 23 wherein said particulate materials are hydrophobic.

25. The method of claim 23 wherein said particulate material has a Receding Contact Angle of greater than 90°.

26. The method of claim 23 wherein the particulate material has a particle size distribution wherein up to 90% of the particles have a particle size of under about 10 microns.

27. The method of claim 23, wherein the particulate materials comprise one of calcium carbonate and hydrous kaolin, hydrous kaolin and calcined kaolin, calcium carbonate and calcined kaolin.

28. The method of claim 23 wherein the substrate is selected from the group consisting of agricultural and ornamental crops.

29. The method of claim 23 wherein the substrate is selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants.

30. The method of claim 23 wherein the finely divided particulate materials have a median individual particle size below about 3 microns.

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31. The method of claim 23 wherein the particulate material comprises a hydrophilic core and a hydrophobic outer surface.

32. The method of claim 31 wherein said hydrophobic outer surface materials are selected from the group consisting of chrome complexes, organic titanates, organic zirconate or aluminate coupling agents, organofunctional silanes, modified silicone fluids and fatty acids and salts thereof.

33. The method of claim 31 wherein the hydrophilic core particulate materials are selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof.

34. A method for enhancing the horticultural effect of horticultural substrates selected from the group consisting of fruits, vegetables, trees, flowers, grasses, seeds, roots, and landscape and ornamental plants, which comprises forming on the surface of said substrate a membrane comprised of one or more particulate layers, said layers comprising one or more hydrophobic particulate materials, said hydrophobic particulate materials comprising i) a hydrophilic core selected from the group consisting of calcium carbonate, calcined kaolin and mixtures thereof, and ii) a hydrophobic outer surface, said particulate materials have a median individual particle size of about one micron or less, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

35. A coated substrate comprising a horticultural substrate selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants wherein the surface of said substrate is coated with a membrane comprised of one or more particulate layers, said layers comprising one or more particulate materials, said particulate materials selected from the group consisting of calcium carbonate, calcined kaolin, hydrophobic treated hydrous kaolin, hydrophobic treated talc, mica, attapulgite, pyrophyllite, wollastonite, feldspar, sand, quartz, diatomaceous earth, baryte, ceramic, glass and organic microspheres, aluminum trihydrate, ceramic fibers, glass fibers, colorants, titanium dioxide, and mixtures thereof and being finely divided and hydrophobic, and wherein said membrane contains gaps that do not exceed about 5 μ m and

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the membrane allows for the exchange of gases on the surface of said substrate and the membrane is hydrophobic.

36. A coated substrate comprising a horticultural substrate selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants wherein the surface of said substrate is coated with a membrane comprised of one or more particulate layers, said layers comprising one or more particulate materials, said particulate materials comprising hydrous kaolin and being finely divided wherein the particulate materials have a mean individual particle size below about 10 microns, and wherein said membrane allows for the exchange of gases on the surface of said substrate and the particulate materials further comprise calcium carbonate, calcined kaolin, or calcium carbonate and calcined kaolin.

37. The coated substrate of claim 36, wherein the particulate materials have a mean individual particle size below about 1 micron.

38. A method for disease control on horticultural substrates selected from the group consisting of fruits, vegetables, trees, flowers, grasses, roots, and landscape and ornamental plants which comprises applying a slurry comprising water, a surfactant, and one or more particulate materials to the surface of said substrate to form a membrane comprised of one or more particulate layers and the surfactant, said layers comprising one or more particulate materials selected from the group consisting of calcium carbonate, calcined kaolin, hydrophobic treated hydrous kaolin, hydrophobic treated talc, mica, attapulgite, pyrophyllite, wollastonite, feldspar, sand, quartz, diatomaceous earth, baryte, ceramic, glass and organic microspheres, aluminum trihydrate, ceramic fibers, glass fibers, colorants, titanium dioxide, and mixtures thereof, said particulate materials being finely divided and hydrophobic, and wherein said membrane allows for the exchange of gases on the surface of said substrate.

39. The method of claim 38, wherein the particulate materials have a mean individual particle size below about 1 micron.

* * * * *

EXHIBIT 3



Surround® WP

Crop Protectant

Surround WP crop protectant forms a barrier film, which acts as a broad spectrum agricultural crop protectant for controlling damage from various insect and disease pests, a growth enhancer, and as a protectant against sunburn and heat stress.

ACTIVE INGREDIENT:

Kaolin 95.0%

OTHER INGREDIENTS: 5.0%

TOTAL: 100.0%

CONTAINS NON-PLANT FOOD INGREDIENT:

GUARANTEED ANALYSIS

Active ingredients

95% Kaolin

5% Inert ingredients

KEEP OUT OF REACH OF CHILDREN CAUTION/PRECAUTION

Si usted no entiende la etiqueta, busque a alguien para que se la explique a usted en detalle.
(If you do not understand this label, find someone to explain it to you in detail.)

FIRST AID	
If in eyes:	<ul style="list-style-type: none"> • Hold eye open and rinse slowly and gently with water for 15-20 minutes. • Remove contact lenses, if present, after the first 5 minutes, then continue rinsing eye. • Call a poison control center or doctor for treatment advice.
HOT LINE NUMBER	
Have the product container or label with you when calling a poison control center or doctor or going for treatment. You may also contact 1-800-877-1737 for emergency medical treatment information.	

The use of **Surround WP** in agricultural crop protection applications is covered by US Patents 6,027,740; 6,069,112; 6,110,867 and 6,156,327.

EPA Reg. No. 61842-18

EPA Est. No. 51036-GA-001

EPA Est. No. 72797-AL-001

Growth enhancer uses are not registered in California.

Product of U.S.A.
Manufactured for:
Tessengerlo Kerley, Inc.
2255 N. 44th Street, Suite 300
Phoenix, AZ 85008 USA
1-800-525-2803

NSV1SPUS0708



PRECAUTIONARY STATEMENTS

HAZARD TO HUMANS AND DOMESTIC ANIMALS

CAUTION

Causes moderate eye irritation. Avoid contact with eyes or clothing. Wash thoroughly with soap and water after handling. Remove contaminated clothing and wash clothing before reuse.

PERSONAL PROTECTIVE EQUIPMENT (PPE)

Applicators and other handlers must wear:

- Long sleeved shirt
- Long pants
- Socks and shoes
- Dust/mist-filtering respirator (MSHA/NIOSH approval number prefix TC-21C), or a NIOSH approved respirator with any N, R, P or HE filter.

When handlers use closed systems, enclosed cabs, or aircraft in a manner that meets the requirements listed in the Worker Protection Standard (WPS) for agricultural pesticides [40 CFR 170.240 (d) (4-6)], the handler PPE requirements may be reduced or modified as specified in the WPS.

Nuisance dust masks and goggles provide the best protection for harvesters especially when plants are shaken during harvest.

Follow the manufacturer's instructions for cleaning/maintaining PPE. If no such instructions for washables, use detergent and hot water. Keep and wash PPE separately from other laundry.

USER SAFETY RECOMMENDATIONS

Users should:

- Wash hands before eating, drinking, chewing gum, using tobacco, or using the toilet.
- Remove clothing/PPE immediately if pesticide gets inside. Then wash thoroughly and put on clean clothing.
- Remove PPE immediately after handling this product. As soon as possible, wash thoroughly and change into clean clothing.

ENVIRONMENTAL HAZARDS

For terrestrial uses: **DO NOT** apply directly to water, or to areas where surface water is present or to intertidal areas below the mean high water mark. **DO NOT** contaminate water when disposing of equipment wash-water or rinsate.

DIRECTIONS FOR USE

It is a violation of federal law to use this product in a manner inconsistent with its labeling.

DO NOT apply this product in a way that will contact workers or other persons, either directly or through drift. Only protected handlers can be in the area during application. For any requirements specific to your state or tribe, consult the state or tribal agency responsible for pesticide regulation.

DO NOT apply Surround® WP crop protectant through any type of irrigation system.

Use Surround WP in accordance with directions on this label.

AGRICULTURAL USE REQUIREMENTS

Use this product only in accordance with its labeling and with the Worker Protection Standard, 40 CFR Part 170. This standard contains requirements for the protection of agricultural workers on farms, in forests, nurseries, and greenhouses, and handlers of agricultural pesticides. It contains specific instructions and exceptions pertaining to the statements on this label about personal protective equipment. In nurseries and greenhouses workers are prohibited in the treated area and 25 feet in all directions of the nursery or the enclosed treated area until application is complete. **The restricted-entry interval (REI) is 4 hours from the time of application. DO NOT enter or allow worker entry into treated areas during the restricted-entry interval (REI) of 4 hours unless wearing appropriate PPE.** Personal protection equipment required for early entry workers are long-sleeved shirts, long pants, and shoes plus socks.

I. GENERAL INFORMATION

Surround® WP crop protectant forms a mineral-based particle film intended for protection of agricultural crops, plants in nurseries, and greenhouses. When **Surround WP** is applied to plants, a dry white film results. Many pests are listed as suppressed, which means that full control often is not achieved, and supplemental methods often are needed to enhance the level of control. **Thorough, uniform, and consistent coverage is essential throughout the infestation or stress period.**

Pre-harvest intervals (PHI): **Surround WP** may be applied up to the day of harvest.

For fresh market crops that will not be washed or for field packed crops where a residual white film is not desired, make applications early-season only. White residue at harvest may be minimized if applications to smooth skin crops like apples stop when the fruit is approximately 1/4 of its expected size.

Plant Response Precautions: **Surround WP** keeps plant surfaces cooler and an advance or delay in maturity may result. Pome and stone fruit may have maturity delays of 3 to 7 days, especially in cool regions.

A. MIX INSTRUCTIONS:

For Agitating Sprayer Tanks

1. Slowly add **Surround WP** powder into the water in a recirculating sprayer tank, making sure to keep agitation brisk. **DO NOT** add **Surround WP** to a sprayer tank that does not have adequate agitation. A pre-mix tank can speed up loading operations if sprayer does not have mechanical agitation. Add directly into the mix basket if pump recirculation empties into the mix basket. If there is no mix basket, add **Surround WP** very slowly to the recirculating water. Avoid dumping **Surround WP** directly into the pump intake area as this could plug the filter or intake. Mix thoroughly.
2. Add tank mix pesticides, if any. See compatibility section below before adding any tank mix pesticides.
3. Continue agitation until all of the material is sprayed from the tank.
4. At the end of the application, spray until empty and flush system and nozzles with fresh water. Periodically check in-line strainer and clean if necessary. Properly dispose of rinse water.

For Non-agitating Sprayer Tanks, Such as Handheld and Backpack Sprayers

The following mixing sequence must be followed:

1. Use **Surround WP** powder at a rate of 1/4 to 1/2 lb of **Surround WP** powder per one gallon of water. One-half pound is approximately 3 cups. For sprayers difficult to shake, premix in a 5 gallon bucket per the directions below and pour suspension into sprayer.
2. Add **Surround WP** powder into 1/4 to 1/2 of the water that will be used in the batch to allow adequate space for vigorous shaking. Allow **Surround WP** to wet and sink into the water slowly.
DO NOT fill with a hose or shake the container while powder is floating on top of the water.
3. Mix thoroughly by shaking the closed container vigorously for 30 seconds.
4. Add tank mix pesticides, if any. See compatibility section below before adding any tank mix pesticides.
5. Add the remainder of the batch water and shake the closed container for an additional 30 seconds.
6. Shake the sprayer occasionally during application.
7. At the end of the application, spray until empty and flush system and nozzles or, blow air pressure out of the line and nozzle (usually by upending) and store in a cool place. Apply the leftover mix within two to three weeks to avoid spoilage. Rinse the sprayer and allow to air-dry before the next batch.

B. COMPATIBILITY:

Surround WP is not generally affected by most other insecticides, miticides, and fungicides. However, to ensure compatibility, test tank mixes before use. When mixing with other products, make up a small batch and observe slurry and film characteristics. Curdling, precipitation, spray beading and/or excessive run-off leading to lack of film formation, or changes in viscosity are signs of incompatibility. **Add tank mix pesticides after the Surround WP powder has been added.** Use of anti-foaming agents can interfere with proper coverage. Oil tank mixes can temporarily reduce the whiteness of the

film. Use adequate water on oversprays of products that require absorption into the plant to ensure wetting of the **Surround WP** film.

Tank mixing with other white mineral particulate products such as diatomaceous earth, or other sunburn materials, such as those containing wax, latex or polymer based materials, can lead to postharvest washoff problems. Applications of **Surround WP** over such products or oversprays of such products over **Surround WP** can also impair post harvest wash off.

C. GENERAL APPLICATION INSTRUCTIONS (see also, specific crop use instructions):

Rates: Rate is dependent on the amount of foliage that needs to be covered.

Concentration (the amount of **Surround WP** per 100 gallons of water): The best concentration of **Surround WP** is between 25 to 50 lbs **Surround WP** per 100 gallons, but concentrations of up to 100 lbs per 100 gallons are allowed for specific crop uses.

Coverage: Use sufficient spray volume to obtain thorough **near-drip** coverage. Two or more applications are desirable for complete coverage. For optimal performance as an insecticide, applications must coat all portions of plant that are to be protected, including both sides of the leaves. Apply an additional spray if coverage is insufficient. Spreading on waxy plant surfaces is usually better when the plant surface is warm.

Dwarf, semi-dwarf, and otherwise well-pruned trees will be easier to cover than large trees. Optimum efficacy often is more difficult to achieve in large trees due to increased difficulty achieving thorough coverage.

Applications to tree crops can be made with commercial air blast or high-pressure sprayers that provide enough air turbulence to coat both sides of the leaves, bark, and fruit. The best coverage is achieved at a tractor speed of less than 3 mph when using airblast sprayers.

Plant Color Change: Plant surfaces will typically turn a hazy white color after drying. Additional treatments will turn the plant surfaces a deeper white. This is normal, and indicates appropriate film formation.

Foliage Dryness: Applications to dripping wet foliage can provide inadequate coverage.

Under Hot, Dry Conditions: Best results are obtained with nozzles that produce a fine spray when using **Surround® WP crop protectant** under normal temperature and humidity conditions. Under very hot and dry conditions, increase volume of water and droplet size to improve deposition.

Spray Methods: Air blast, high-pressure handgun, or boom sprayers provide the best results. **DO NOT** apply by chemigation. Observe specific crop label instructions for directions regarding spray volume. **DO NOT** spray under windy or gusty conditions. Calibrate spray equipment per equipment manufacturer to deliver the required volume. At given concentrations, the flow rate of suspended **Surround WP** is similar to water. Strainers, preferably no finer than 40 mesh, in the spray system and behind each nozzle per normal practice help to reduce nozzle clogging.

When the dry foliage has lost its white appearance or when gently rubbing the treated area with a dark piece of cloth does not leave a white residue on the cloth, reapplication is necessary. Heavy rainfall, new growth, and wind erosion will affect film quality. Reapply to re-establish coverage after heavy rain as soon as the foliage is dry. However, reapplication often is not necessary if all target surfaces remain thoroughly coated and insect pressure is light. Excessively thick coatings can provide poor performance.

Overhead Irrigation and Overhead Cooling: Overhead irrigation is not preferred. **DO NOT** use with overhead cooling.

Aerial Application: **Surround WP** applied by air will reduce heat stress and sunburn damage if sufficient coverage can be achieved and maintained. Aerial applications often are not effective for controlling insects or diseases.

It is best when using aerial applications to take all precautions needed to minimize or eliminate drift, e.g., **DO NOT** spray under windy or gusty conditions. It is best to make applications not more than ten feet above the top of the largest plant unless a greater height is required for aircraft safety. It is best when making applications at low relative humidity to increase droplet size to compensate for evaporation.

Non-Target Surfaces: **DO NOT** spray where the resulting visible white film will be undesirable or cannot be washed off, such as porous wood, masonry, asphalt, and other valuable goods.

D. GROWTH ENHANCER, SUNBURN AND HEAT STRESS PROTECTANT:

When applied at given rates and frequencies, benefits such as increased plant vigor and improved yields typically occur on many crops. Under high ambient temperatures, **Surround WP** reduces canopy temperature and, therefore, can help to reduce heat and water stress. When **Surround WP** is used, many fruits have shown improved fruit color, soluble solids, smoothness, and size with less russet, dropping, sunburn, and cracking.

Sunburn Suppression: Apply to sunburn-prone fruit, leaf, or limb and trunk bark surfaces before conditions leading to sunburn occur. If initiating sprays for sunburn suppression where there have been no prior sprays, provide thorough coverage of all fruit or other plant surfaces prior to sunburn-causing conditions with one to two full rate applications 7 days apart. Depending upon the length of the high heat period, three to four applications in total often are needed, with subsequent applications every 7 to 21 days. Good coverage on typical semi-dwarf trees is best achieved with the initial one to two sprays at 50 lbs in 100 to 200 gallons per acre to achieve **near-drip** coverage on the fruit or other plant surfaces. However, if allowed (see specific crop group directions), up to 100 lbs/100 gallons are allowed when spray frequencies need to be reduced. Make subsequent applications at half to full rates if even coverage is maintained throughout the high heat period. Under windy conditions, **Surround WP** can be rubbed off by leaf movement making reapplication necessary.

Aerial Application: Aerial applications for sunburn and heat stress reduction are best made at concentrations of no more than 1 lb of **Surround WP** per 1 gallon of water. Use no less than 10 gallons per acre, but on trees 20 or more gallons per acre are preferred. Repeat applications 3 to 4 times per above intervals or as necessary to establish and maintain even coverage on fruit surfaces throughout the high heat period. See aerial applications under I C.

E. PACKING AND PROCESSING:

Washing is required unless only early season applications are made and the firm weathers off before harvest. Most residues wash off with packing line brushing and forced water sprays. An approved washing detergent is typically helpful if used in the packing line and/or wash tank. Perform a pre-harvest washing trial to determine if a washing detergent is necessary. Waxing further improves fruit appearance.

For fresh market apples that will not be waxed, such as apples for organic markets or specific export markets that DO NOT accept waxed apples or for washed crops where traces of white residue are not acceptable: Unless washing facilities are adequate, cease applications enough in advance of harvest to allow residue to weather off completely. For 'Red Delicious' and 'Braeburn' apple varieties DO NOT apply any later than two months prior to harvest.

II. CROP GROUPS

A. TREE FRUIT:

Volume: Apply to **near-drip**. **DO NOT** apply to run-off to avoid waste and poor coverage. For typical semi-dwarf trees in full foliage, it is recommended to use 100 to 200 gallons per acre. Adjust volume per Tree Row Volume to achieve near-drip for larger or smaller trees.

Concentration (the amount of **Surround WP crop protectant** per 100 gallons of water): It is best to use 25 to 50 lbs **Surround WP** per 100 gallons of water, but concentrations of up to 100 lbs per 100 gallons are allowed. Early applications typically use 37.5 to 50 lbs per 100 gallons of water per acre while follow-up applications may only require 25 lbs per 100 gallons of water per acre.

Pome Fruits
Such as apple, crabapple, quince, pear, and loquat

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Pear psylla	50'	<ul style="list-style-type: none"> On mature pear trees apply 100-200 gal/acre. Prebloom: Apply 3 applications, applying every 7-10 days starting at delayed dormant, but no later than green cluster bud. Petal Fall: Apply 3 applications, applying every 7-14 days starting at first petal fall. Spring populations can be reduced with a fall or winter application.

Insects Pear: Cutworms, pear midge, pear slug Apple: Apple sucker, climbing cutworm, eastern tent caterpillar, European apple sawfly, Gypsy moth, Japanese beetle, June beetle Pear and Apple: Grasshoppers, green fruit worm, leafrollers, Lygus bug, Mormon cricket, periodical cicada, stink bug, tarnished plant bug, thrips	25-50	Suppression only*. Start before infestation and continue at 7-14 day intervals. DO NOT widen respray interval past 14 days.
Apple or Pear Diseases Fabrea leafspot	25-50	Suppression only*. Apply prior to conditions favoring disease development and maintain coverage throughout the disease infection period. Supplements are generally necessary especially in high-pressure areas.
Over-wintering leafrollers, especially obliquebanded leafroller (OBLR)		Apply 2 sprays 7 days apart starting just prior to green tip or at initial emergence of leafroller larvae.
Leafhoppers ²		Apply first spray within 3 days of first petal fall or at initial infestation. Continue every 7-14 days during infestation period.
Lacanobia		<ul style="list-style-type: none"> • Oviposition: Apply at least one spray at start of oviposition. A second spray within 5-7 days improves efficacy. • Egg Hatch: Apply 2-3 sprays starting at initial egg hatch and continuing at 7-day intervals. • Using Surround WP for the later generation of lacanobia is allowable if fruit will be thoroughly washed prior to picking or packing.
Apple maggot		Apply 2 sprays 7 days apart before expected oviposition or first detection of infestation. Continue applications every 7-14 days to keep fruit completely covered during egg lay period.
Codling moth (first generation only), oriental fruit moth, plum curculio		Suppression only*. Apply at biofix or first detection. Continue applications every 7 days to keep fruit completely covered during egg lay period.
Sunburn and heat stress		See I D.
¹ Rates of 100 lbs in 100 to 200 gallons allowed on pear trees during prebloom for high infestation. ² Applications at petal fall can sometimes disrupt leaf miner parasitism, requiring control measures. *If complete control is needed, consider using supplemental controls.		

Horticultural Benefits

Surround WP often enhances fruit quality (see I D for general horticultural benefits) if applications start at petal fall and are continued until mid to late season. For petal fall applications, see footnote 2 above about leaf miner. Many pear cultivars, particularly Comice and Anjou varieties, have shown improved fruit color, smoothness, and size with less russet when **Surround WP** is used. Some apple cultivars have shown less bitter pit and corking in season-long programs.

Diseases

Surround WP sometimes can enhance the efficacy of wettable sulfur and/or lime-sulfur against scab, powdery mildew, sooty blotch, and flyspeck. See Product Guides for specific mixture directions. The use of wettable type sulfurs are preferred if tank mixing sulfur with **Surround WP**. **DO NOT** tank-mix elemental sulfurs with **Surround WP**.

Non-waxed Fresh Apples and Pears (also, see I E)

For fresh market apples and pears that will **not** be waxed, such as fruit for organic markets or specific export markets that **DO NOT** accept waxed fruit: It is best that **Surround WP** not be applied any later than two months prior to harvest as slight traces of white sometimes can still be visible after washing. This especially applies to 'Red Delicious' and 'Braeburn' cultivars.

Stone Fruit

Such as apricot, sweet or tart cherries, nectarine, peach, plum, pluot, plumcot¹, and prune

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Japanese beetle, rose chafer	25-50	Suppression only*. Start one week prior to expected infestation and follow with 3-4 applications at 5-7 day intervals.
Obliquebanded leafroller (OBLR), thrips		Suppression* only for OBLR. Start at pink bud and continue applications at 5-7 day intervals through jacket split.
Leafhoppers including sharpshooters		Suppression only*. Start before expected infestation, if possible. • Until harvest: Apply at 7-14 day intervals up to bloom. • Post harvest: Apply 2 or more applications at 7-14 day intervals.
Cherry fruit flies, grasshoppers, June beetle, leafhoppers, navel orange worm, oriental fruit moth		Suppression only*. Start before expected infestation, if possible, and continue at 7-14 day intervals.
Plum curculio		Suppression only*. Apply at 7 day intervals throughout egg laying period.
Sunburn and heat stress ¹	25-100	See I D.
¹ Rate of 100 lbs in 100 to 200 gallons are allowed for post-harvest sunburn and heat stress use.		
*If complete control is needed, consider using supplemental controls.		

Special Directions

Special Washing Considerations for Stone Fruit: For fresh market fruit, special washing is required; especially for fuzzy peaches. Most residues wash off with brushing and forced water sprays. An approved fruit cleaning detergent can be used in packing line and/or wash tank. Prior to brushing, a pre-soak in approved fruit cleaning detergent is usually needed for fuzzy peaches. A pre-harvest washing trial is a good practice to determine if a detergent is necessary. Waxing further improves fruit appearance. If fresh market stone fruit cannot be washed as noted above, discontinue sprays when the fruit are approximately 3/4 inch in diameter. Residues of **Surround WP crop protectant DO NOT** affect processed fruit quality.

If cherries are for fresh market, discontinue application when fruit are half size (approximately 1/4 inch) if no washing is available.

Citrus Fruits
Such as lemon, lime, grapefruit, mandarin, satsuma mandarin,
pummelo, mandarin, tangelos, kumquat, and oranges

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Thrips	50-75 ¹	Start at pre-bloom. Continue at 5-7 day intervals throughout bloom. Post-bloom continue at 7-14 day intervals through infestation.
Leafhoppers including sharpshooters		Suppression only*. • Apply every 7-14 days as infestation occurs. • Near-harvest: Apply at least 2 applications 7 days apart for pre-harvest control of sharpshooters.
Citrus psyllid, diapaupes root weevil, grasshoppers		Suppression only*. Apply every 7-14 days as infestation occurs.
Sunburn and heat stress		See I D.
150 lbs per 200 gallons per acre preferred for 12 foot trees. *If complete control is needed, consider using supplemental controls.		

B. NUT CROPS:

Volume: Apply to **near-drip**, not to excessive run-off. For 20-foot trees, it is best to use 200 gallons per acre. Large trees like walnut and pecan can require up to 300 gallons per acre.

Concentration (the amount of Surround WP per 100 gallons of water): It is best to use 25 to 50 lbs Surround WP per 100 gallons of water.

Tree Nuts
Such as almond, beech nut, Brazil nut, butternut, chestnut, cashew, filbert,
hickory nut, macadamia nut, pecan, walnut, and including pistachio

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Aphids such as pecan, black, and yellow aphid, codling moth, grasshoppers, husk fly, leafhoppers, naval orangeworm, stink bug	50-75 ¹	Suppression only*. Start at biofix or as infestation occurs. Apply every 7-14 days throughout infestation. For codling moth, apply a minimum of 2 sprays per generation.
Sunburn and heat stress		See I D.
¹ 50 lbs per 200 gallons per acre preferred. *If complete control is needed, consider using supplemental controls.		

c. Small Fruits:

Volume: Apply to **near-drip**. **DO NOT** apply to run-off to avoid waste and poor coverage. The volume of water/acre required will increase throughout the growing season in relationship to the increasing size of the crop and its foliage.

Concentration (the amount of **Surround WP crop protectant** per 100 gallons of water): It is best to use 25 to 50 lbs **Surround WP** per 100 gallons of water.

Berries

Such as blackberry, raspberry, dewberry, boysenberry, loganberry, elderberry, blueberry, ribes such as currant and gooseberry, and including cranberry

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Blackberry psyllid, grasshoppers, Japanese beetle, leafhoppers, leafrollers, plum curculio, rose chafer, thrips	25-50 ¹	Suppression only*. Begin applications after fruit set and prior to infestation, applying every 7-14 days.
Blueberry maggot		Suppression only*. Apply 2 sprays 7 days apart before expected infestation or first detection of infestation. Continue applications every 7-14 days during egg lay period.
Sunburn and heat stress		See I D.

¹25 lbs per 50 gallons per acre preferred.
*If complete control is needed, consider using supplemental controls.

Special Directions

Apply on fresh market berries only up to the first three weeks after fruit set as trace residues can be difficult to remove after harvest. Application of plain water via normal sprayer prior to harvest can help to reduce **Surround WP** residues. Processing blueberries can be washed using detergent and water sprayers capable of rinsing away residues in the packing line. Minor residues of **Surround WP** on blueberries for processing are acceptable provided no other pesticides are also present.

Grapes

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Thrips	25-50 ¹	Apply 1-2 applications 7 days apart when pest reaches treatment threshold.
Rose chafer, omnivorous leafroller, grape leafroller, grape leaf folder, grasshoppers, grape leaf skeletonizer, Japanese beetle, June beetle		Suppression only*. Start at biofix or as infestation occurs, applying a minimum of 2-3 applications by applying every 7-14 days.
Leafhoppers including sharpshooter		Apply initial application as infestation occurs, applying at least 2-3 applications by applying every 7-14 days throughout the infestation.
Sunburn and heat stress		See I D.
¹ 25 lbs in 50 gallons per acre preferred. *If complete control is needed, consider using supplemental controls.		

Special Directions

Wine grapes: Harvest parameters can be altered and maturity can be delayed especially in white wine varieties. Closely monitor harvest parameters to determine optimal time to harvest. Changes in harvest parameters can affect final taste. Wine grapes sprayed up to veraison will have minimal adherence to berries. Applications after veraison will adhere more on grape berries.

Raisin grapes: It is best that raisin grapes be sprayed only up to bunch closing. Infestations can be sprayed up to first bloom, and again after harvest.

Table grapes: DO NOT spray table grapes from first bloom to harvest as white residue can remain on the rachis and fruit until harvest. Infestations can be sprayed up to first bloom, and again after harvest.

D. FIELD VEGETABLES:

Volume: Apply to **near-drip**. **DO NOT** apply to run-off to avoid waste and poor coverage. The volume of water/acre required will increase throughout the growing season in relationship to the increasing size of the crop and its foliage. Seedlings will need less water and a lower amount of **Surround WP crop protectant** than fully mature plants to uniformly coat their surfaces. Adjust the volume of water and rate used throughout the season based upon plant size. Lower volumes and rates are typically used only for immature plants.

Concentration (the amount of **Surround WP** per 100 gallons of water): It is best to use 25 to 50 lbs **Surround WP** per 100 gallons of water.

Legume Vegetables
Such as beans, pea, chickpeas, and soybean

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Bean leaf beetle, flea beetles, grasshoppers, Japanese beetle, leafhoppers, Mexican bean beetle, three cornered alfalfa hopper, thrips	25-50 ¹	Suppression only*. Start prior to infestation, applying every 7-14 days throughout infestation.
Sunburn and heat stress	25-100	See I D.
*If complete control is needed, consider using supplemental controls.		

Special Directions

Only spray beans that are difficult to wash, such as string beans, when beans are small. **DO NOT** apply to other field and garden vegetables if they are intended for the fresh market and cannot be adequately washed after harvest to completely remove **Surround WP** residues. Minor residues of **Surround WP** on field and garden vegetables for processing are acceptable provided no other pesticides are also present.

Root and Tuber Vegetables
Such as potato, garden beet, sugar beet, horseradish, radish, ginseng, rutabaga, carrot, ginger, sweet potato, yams, and turnip

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Flea beetles, grasshoppers, leafhoppers	25-50	Suppression only*. Start prior to infestation, applying every 7-14 days throughout infestation.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		

Fruiting Vegetables
Such as tomato, pepper¹ and including eggplant¹

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Cucumber beetles, flea beetles, grasshoppers, leafhoppers, thrips	12.5-50 ²	Suppression only*. Start prior to infestation, applying every 7-14 days throughout infestation.
Sunburn and heat stress	25-100	See I D.
¹ For fresh market apply only up to 1/4 of fruit size unless washing capabilities are sufficient. ² 12.5 lbs/25 gallons preferred up to fruit set. *If complete control is needed, consider using supplemental controls.		

Bulb Vegetables
Such as onions, garlic, leek, and shallot

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Thrips	25-50	Suppression only*. Start prior to infestation and apply every 5-7 days throughout infestation. Good coverage down into the plant crown is essential for efficacy.
Sunburn and heat stress	25-100	See I D.
*If complete control is needed, consider using supplemental controls.		

Cucurbit Vegetables
Such as cucumber, summer and winter squash, pumpkin, citron melon, muskmelon, and watermelon

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Cucumber beetle, grasshoppers	25-50	Suppression only*. Start prior to infestation, applying every 5-7 days, with the first two applications 3 days apart.
Powdery mildew		Suppression only*. Apply every 7-14 days as required to maintain coverage.
Sunburn and heat stress	25-100	See I D.
*If complete control is needed, consider using supplemental controls.		

Brassica (Cole) Leafy Vegetables
Such as broccolis, brussels sprouts, Chinese cabbages, cauliflower, collards, cabbages, mustard greens, kale, kohlrabi and including canola

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Flea beetles, grasshoppers	25-50	Suppression only*. Use only in early season to avoid residue at harvest. Apply every 7-10 days making sure to wet the plant surface completely.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		

Leafy Vegetables (Non-Brassica)
Such as arugula, celery, lettuces, parsley, rhubarb, and spinach

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Flea beetles, grasshoppers	25-50	Suppression only*. Use only in early season to avoid residue at harvest. Start prior to infestation. Apply every 3-5 days to maintain adequate coverage throughout infestation.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		

Asparagus

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Common and spotted asparagus beetle, grasshoppers	12.5-25	Suppression only*. Start prior to infestation. Apply every 7-10 days being sure to maintain coverage throughout infestation.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		

E. OTHER AGRICULTURAL CROPS:

Volume: Apply to **near-drip**. **DO NOT** apply to run-off to avoid waste and poor coverage. The volume of water per acre required will increase throughout the growing season in relation to the increasing size of the crop and its foliage.

Concentration (the amount of **Surround WP crop protectant** per 100 gallons of water): It is best to use 25 to 50 lbs **Surround WP** per 100 gallons of water.

Cotton

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Boll weevil, cotton fleahopper, flea beetles, grasshoppers, thrips	25-50	Suppression only*. Start prior to infestation. Spray every 7-14 days with the first two sprays 7 days apart.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		

Cereal Grains and Nongrass Animal Feeds
Such as corn, popcorn, oats, barley, wheat, rice, sorghum, and alfalfa

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Armyworm, flea beetles, grasshoppers, leafhoppers, thrips	12.5-37.5	Suppression only*. Start prior to infestation. Apply every 5-10 days during infestation, with the first two applications 3 days apart. Tighten spray intervals during fast growing periods.
Sunburn and heat stress	25-100	See I D.
*If complete control is needed, consider using supplemental controls.		

Tropical Crops

Such as coffee, avocado, banana, guava, mango, passion fruit, starfruit, papaya, and pineapple

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Avocado looper, coconut bug, fruit flies, grasshoppers, leafhoppers, leafrollers, mango weevil, thrips	25-50	Suppression only*. Start prior to infestation. Apply minimum of 2 applications 7-14 days apart during application.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		

Special Directions

Initial application over waxy surfaces such as mango fruit can bead and not spread adequately.

Miscellaneous Crops

Such as artichoke, fig, hops, globe, kiwifruit, olives, okra, paw paw, peanut, persimmon, pomegranate**, strawberry and water chestnut

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Flea beetles, fruit flies, grasshoppers, leafhoppers, olive fruit fly, thrips	25-50	Suppression only*. Start prior to infestation. Spray every 7-14 days throughout infestation.
Sunburn and heat stress		See I D.
*If complete control is needed, consider using supplemental controls.		
**Not for use in CA unless accompanied by a Supplemental Label.		

F. NON-FOOD CROPS:**Nursery and Ornamental Plants and Trees**

Such as landscape plants, flowers or ornamental tree seedlings/saplings in nurseries or field plantings and forest trees** (deciduous or evergreen) in nurseries or field plantings

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Black vine weevil, diabrotica beetle, grasshoppers, leafhoppers including sharpshooters, Japanese beetle, thrips	6.25-37.5	Suppression only*. Start prior to infestation. Spray every 5-7 days throughout infestation.
Deer, rabbit		Suppression only*. Start prior to animal feeding and maintain coverage during feeding period. For plants on which Surround WP crop protectant does not adhere well, respray as soon as possible following rainfall or protection will be lost.
Sunburn and heat stress		See I D. Reduces transplant shock and heat stress when transplanting nursery stock to the field or garden. DO NOT dip transplants into Surround WP solution.
*If complete control is needed, consider using supplemental controls. ** Sunburn and heat stress only.		

Special Directions

Plants treated with **Surround WP** will appear white from application. If this is undesirable, **DO NOT** spray. On cut-flower plants, it is best to spray only the leafy foliage of the plant, so that cuttings are not affected.

Christmas Trees and Hybrid Poplar Plantations

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Sunburn and heat stress	25-75	See I D. Use 25-50 lbs in 100 gallons of water for best results. Spray 2 applications 14 days apart prior to heat event. Stop sprays 30 days prior to harvest.

G. GREENHOUSE GROWN PLANTS:

Edible and Non-Food Crops
Such as all crops listed on this label

PEST	LBS/ACRE	APPLICATION INSTRUCTIONS
Pests as listed under specific crop groups on this label.	6.25-37.5	Suppression only*. Start prior to infestation. Spray every 5-7 days being sure to maintain coverage, especially on new growth. Exercise extra care to cover undersides of leaves when targeting pests that inhabit leaf undersides.
*If complete control is needed, consider using supplemental controls.		

STORAGE AND DISPOSAL

DO NOT contaminate water, food or feed by storage or disposal.

PESTICIDE STORAGE: Store in a dry, sheltered location (away from food or feed). Product is slippery when wet. In case of spill or leak, avoid breathing dust, clean up and dispose of wastes in compliance with applicable Federal, State, and local regulations.

PESTICIDE DISPOSAL: Wastes resulting from the use of this product must be disposed of on site or at an approved waste disposal facility.

CONTAINER DISPOSAL: Completely empty bag into application equipment. Dispose of empty bag in a sanitary landfill or by incineration, or, if allowed by State and local authorities, by burning. If burned, stay out of the smoke.

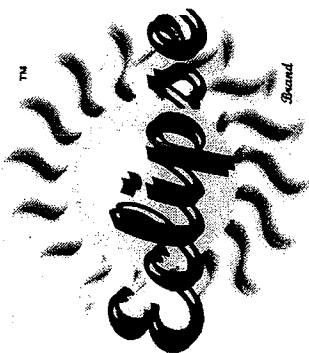
Information regarding the contents and levels of metals in this product is available on the internet at <http://www.aapfco.org/metals.htm>

Conditions of Sale and Warranty
<p>CONDITIONS OF SALE - LIMITED WARRANTY AND LIMITATIONS OF LIABILITY AND REMEDIES</p> <p>The directions on this label are believed to be reliable and must be followed carefully. Insufficient control of pests and/or injury to the crop to which the product is applied may result from the occurrence of extraordinary or unusual weather conditions, or the failure to follow the label directions or good application practices, all of which are beyond the control of Tessenderlo Kerley, Inc., or seller. In addition, failure to follow label directions may cause injury to crops, animals, man or the environment. Tessenderlo Kerley, Inc. warrants that this product conforms to the chemical description on the label and is reasonably fit for the purpose referred to in the directions for use, subject to the factors noted above which are beyond the control of Tessenderlo Kerley, Inc. Except as warranted by this label, Tessenderlo Kerley, Inc. makes no other warranties or representations of any kind, express or implied, concerning the product, including no implied warranty of merchantability or fitness for any particular purpose. To the extent allowed by applicable law, the exclusive remedy against Tessenderlo Kerley, Inc. for any cause of action relating to the handling or use of this product is a claim of damage, and in no event shall damages or any other recovery of any kind against Tessenderlo Kerley, Inc. exceed the price of the product which causes the alleged loss, damage, injury, or other claim. To the extent allowed by applicable law, Tessenderlo Kerley, Inc. shall not be liable and any and all claims against Tessenderlo Kerley, Inc. are waived, for special, indirect, incidental, or consequential damages or expense of any nature, including, but not limited to, loss of profits or income, whether or not based on the negligence of Tessenderlo Kerley, Inc. breach of warranty, strict liability in tort, or any other cause of action. Tessenderlo Kerley, Inc. and the seller offer this product, and the buyer and users accept it, subject to the foregoing conditions of sale and limitations of warranty, liability and remedies.</p>

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EXHIBIT 4



Product Overview

Optimum Sunscreen for Fruit and Produce

Most packer/shippers cull 40% of fruit and produce due to sunburn damage, and this problem will continue to rise due to climatic changes. Growers over the last 5 years have reduced sunburn damage up to 90% with Eclipse™ brand sun screen, which adds to the bottom line of increased returns.

Proven

Eclipse is a calcium carbonate and boron liquid suspension that has been proven effective in the prevention of sunburn and stress on produce. Eclipse is sprayed on through conventional sprayers or aerially applied throughout the season. Eclipse's unique sub-micron particle size is the key to its success. Applications throughout the season build in theory a protective coating of microscopic prisms to reflect the harmful sunrays, while at the same time relieves stress.

Ease of Use

Eclipse's superior formulation stays in suspension throughout. This allows for packaging in five gallon buckets all the way up to the farmer friendly 250 gallon shuttles. The shuttles alleviate the disposal of 2.5 gallon jugs and are convenient for spray sites. Eclipse blooms beautifully in the spray tank because of its particle size which in turn provides a uniform layer of protection for your fruit and produce from top to bottom. Please consult your field man on compatible products with Eclipse to save time and trips through your field.

Timing

The first applications of Eclipse are the most important! In the early stages of fruit and produce development, the cell walls are more susceptible to sunburn damage just like a baby's skin. Eclipse needs to be applied before the damage occurs and reapplied to the critical growth stages during the season to maximize pack out potential.

Highlights

- *Submicron Particle size: Eclipse stays in suspension due to superior formulation.
- *Farmer friendly packaging: Consistent formulation allows various package sizing.
- *Compatibility: Compatible with many products to decrease trips through the field.
- *Easy to remove: Packing houses love Eclipse because with a 5.5-6.0 PH, standard cleaning leaves no residue.
- *Total Fruit and produce production: Increasing color, increasing the integrity of cell structure, having reduced physiological problems, has all been attributed to Eclipse protecting the fruit from harmful UV. This simultaneously decreases transpiration from heat stress and increases fruit size and quality. The calcium load of Eclipse is available all season long.

Recommended General Applications

Depending on the growing season, four to six applications of Eclipse through the growing season will be needed. Additional applications may be required under extreme conditions, or rain events that may deteriorate the coverage of the crop.

Pome Fruit, Mangos, Avocados, and Pomegranates

Applications of Eclipse should start at 1/2" or 15mm diameter at a rate of 3 gallons/acre in 100 gallons of water or less as long as thorough coverage of the fruit is achieved. The second application should be applied 10-14 days later at a rate of 2.5 gallons/acre. Aerial applications can be made on these commodities and alternated with ground equipment for best results.

Subsequent applications of Eclipse should be made at 2-3 week intervals, or as needed to protect the new growth of the fruit at a rate of 2.5 gallons per acre in 100 gallons of water or sufficient water to insure thorough coverage.

Tomatoes, Peppers, Eggplants, and Melons

Applying Eclipse at a rate of 2-3 gallons/acre in 20-50 gallons of water to the plant is beneficial in preventing sunburn to the exposed portion of the plant. Subsequent applications using 2 gallons/acre should be made 7-10 days after the initial application or immediately after the initial harvest. Use low pressure on your sprayer to ensure proper coverage.

Cherries

Eclipse should be first applied at shuckfall at 3 gallons/acre in 100 gallons of water followed by the second application 10-14 days after the initial application at 2.5 gallons/acre in 100 gallons of water. The third application should be applied roughly 1-2 weeks prior to harvest to prevent sunburn and heat stress at 2.5 gallons/acre in 100 gallons of water. Post harvest application for prevention of doubles the following year apply Eclipse at a rate of 5 gallons/100 gallons of water within one of postharvest, two post harvest applications may be made if heat persists.

Tree nuts, Olives, and Figs

Eclipse should first be applied when fruitlets are 1/2" or 15mm diameter at a rate of 3 gallons/acre in 100 gallons of water or enough for thorough coverage. The second application should be applied 10-14 days later at a rate of 2.5 gallons/acre. Aerial applications can be made on these commodities and alternated with ground equipment for best results.

Onions, Garlic, and Potatoes

Ground application: Applying Eclipse at a rate of 2-3 gallons/acre in 20-50 gallons of water after the burn down of the foliage is beneficial in reducing sunburn damage to the exposed portion of the plant.

Aerial Application: Applying Eclipse at 2.5 gallons in 10 gallons of water is beneficial in reducing sunburn. An additional application of Eclipse can be applied 3-4 days after the initial application at 2.5 gallons/10 gallons of water.

Notice: Before using Eclipse, read and carefully observe the precautionary statements, directions for use, restrictions and all pertinent information on the label. Consult your fieldman for compatibilities.

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EXHIBIT 5



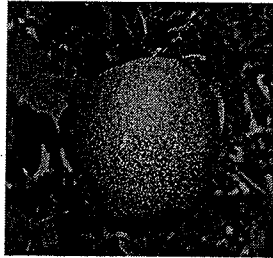
42 SPF for Sunscreen for Produce

Produce in the field is extremely susceptible to sun damage. An estimated 20-40% of produce in orchards may be damaged as a result of sunburn, and the problem continues to rise with global warming. With Eclipse, a newly engineered thin film sunshield, growers can reduce sunburn damage up to 90%, which significantly reduces product losses and increases returns.

Highlights

- Submicron particle sizes
 - Product stays in suspension during spraying to give a uniform coat of protection from the bottom to the top of the tree.
- Easy to store, mix and spray
 - The proprietary liquid formulation will not settle out and provides a consistent concentration of product through the entire tank.
- Compatible with most other spray applications
 - Eclipse is compatible and can be mixed with most chemicals, which will help to improve returns by decreasing orchard trips.
- Easy to remove
 - Tests have proven that with standard brushes, Eclipse leaves no residue before waxing.
- Enables natural coloring and photosynthesis
 - Specific light wavelengths needed for plant health are not blocked by Eclipse - the harmful spectrum of natural light is blocked.

Proven



Eclipse is a calcium carbonate and boron colloidal liquid that has been proven effective in the control of sunburn on produce. Eclipse is sprayed directly on the produce surface throughout the season to build a protective coating that blocks harmful UV light, without decreasing photosynthesis.

Using Eclipse, growers have observed higher pack-outs, larger produce, better color and earlier harvest dates.

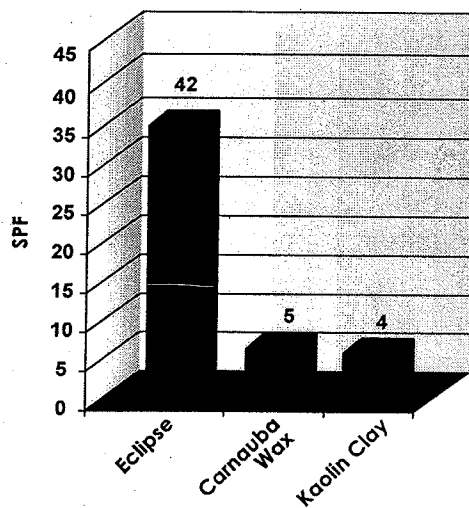
Economical

Eclipse can be sprayed at the same time with most other chemicals, saving time and orchard trips. The solution quickly tank mixes with minimal agitation, and with its small particle size, Eclipse stays in suspension during spraying to give a uniform coat of protection from the bottom to the top of the tree.

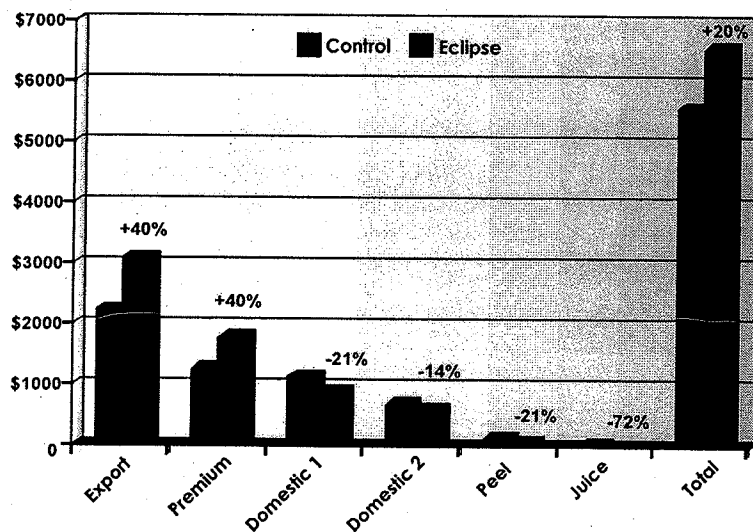
Safe

With Eclipse's food grade calcium carbonate structure, it can be applied anytime during the growing season. Application for organic pending.

Impact of Proprietary Liquid Structure



Eclipse Total Pack Out Value Added



2007 Univ. of Talca Granny Smith Apple Trial

Recommended Applications

General Application Guidelines

To protect most crops, four to six applications of Eclipse during the growing season are recommended. Additional applications may be required under conditions of persistent high temperature, increased UV exposure, rain or other circumstances that may deteriorate the coverage of the crop.

Pome Fruit and Citrus

Eclipse should first be applied when the fruit is 3/4" or 19 mm at a rate of 3 gallons/acre in 100 gallons of water or less as long as thorough coverage of the fruit is achieved. The second application should be applied 8 to 9 days later at a rate of 2.5 gallons/acre in 100 gallons of water or sufficient water to ensure thorough coverage.

Subsequent applications of Eclipse should be made at 2-3 week intervals, or as needed to maintain coverage of the fruit, at a rate of 2.5 gallons/acre in 100 gallons of water or sufficient water to ensure thorough coverage.

Tree Nuts, Olives, and Figs

Eclipse should first be applied when the fruiting bodies are 5/8 mm. Similar rates and application timing as in pome fruit and citrus should be followed.

Onions, Garlic and Potatoes

Ground Application: Applying Eclipse at a rate of 3 gallons/acre in 50 gallons of water after the "burn down" of the foliage is beneficial in limiting sun damage to the exposed portion of the plant.

Aerial Application: Applying Eclipse at a rate of 3 gallons/acre in 10 gallons of water is beneficial to protect exposed onions, garlic and potatoes from sun damage. If the crop is dried above the ground surface, Eclipse should be immediately applied to the exposed crop after removal from the soil. A reapplication of Eclipse is recommended 3-4 days after the initial application at a rate of 3 gallons/acre in 10 gallons of water.

Forestry Nursery Stock

Application of Eclipse at a rate of 10 gallons in 100 gallons of water per acre directly to nursery foliage before intense solar exposure (May 1 in North America) is beneficial in protecting the plant from stress. Additional applications should be applied at 30 day intervals at a rate of 5 gallons in 100 gallons of water to the acre. Immediately before transplanting, nursery stock can also be treated with a 10% solution of Eclipse to lessen the effects of heat stress.

Post Harvest Removal of Eclipse

It is generally accepted that Eclipse can be removed on a commercial packing line that includes a water filled dump tank with a Ph of 5.5 or less and a brush section for agitation. During and after agitation a fresh water rinse is recommended prior to any pre-packaging application of wax and/or fungicides.

Notice: This bulletin is not intended to provide adequate information for use of this product. Before using this product, read and carefully observe the precautionary statements, directions for use, restrictions and all pertinent information on the label.

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