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PROCESS OF COMMINUTING IMPURE DDT

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This invention relates to insecticidal materials, and more particularly to an improved process for obtaining dispersible finely divided 1,1,1-trichloro-2,2-bis (p-chlorophenyl) ethane, herein-after called DDT for convenience.

DDT is of great value as an insecticide in various military and civilian applications. One of the principal modes of applying DDT is by dispersing it as a solid material in finely divided form in inert liquid or solid diluents. DDT of high purity, i. e., having a setting point above 106° C., can be comminuted in high velocity gas disintegrators, e. g., in a "Micronizer," to the desired small particle size in high yields. However, when technical or partially purified grades of DDT, which contain substantial proportions of impurities, particularly 1,1,1-trichloro-2(p-chlorophenyl)-2-(o-chlorophenyl) ethane, are disintegrated in a "Micronizer," a considerable amount of it, e. g., 10–15% or more, becomes packed into a solid cake or ring inside the grinding chamber. In addition to reducing the yield of DDT powder of the desired particle size, the solid cake of DDT reduces the pulverizing efficiency of the "Micronizer" to such an extent that the pulverizing operation must be interrupted frequently to remove this cake of DDT from the grinding chamber. This packing of impure DDT powder during "Micronization" is not substantially reduced by lining the inside of the grinding chamber with rubber or neoprene sheeting, an expedient which is effective in the "Micronization" of other crystalline organic compounds. Since DDT having a purity represented by material having a setting point of 102–106° C. is entirely satisfactory from the standpoint of its insecticidal properties, there exists a definite need for a process for reducing its particle size to the desired fineness in a high velocity gas disintegrator without losing excessive amounts of it by packing into a cake and without requiring frequent interruptions of the grinding to clean out the pulverizing chamber to restore its operating efficiency.

It is an object of this invention to provide a new method for comminuting DDT in a high velocity gas disintegrator. Another object is to provide a method for comminuting impure DDT having a setting point between 102–106° C. which reduces the amount of DDT powder which packs into a cake in the pulverizing chamber of the high velocity gas disintegrator. Other objects will appear hereinafter.

These objects are accomplished by the present invention of comminuting in a high velocity gas

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disintegrator impure DDT having a setting point of 102–106° C. in the presence of a salt of an acid having an ionization constant of at least 3×10^{-7} at 25° C.

5 The invention is carried out by uniformly blending DDT having a setting point of 102–106° C. with an aqueous solution of a salt of an acid having an ionization constant of at least 3×10^{-7} at 25° C., removing the water from the resulting paste, drying the coated DDT particles, and passing the dry DDT through a high velocity gas disintegrator such as that known commercially as a "Micronizer."

In a preferred embodiment of the invention the DDT is coated with a salt of an acid having an ionization constant of at least 3×10^{-7} which is also capable of promoting dispersibility of wet-
15 tability of the comminuted DDT.

The invention is illustrated by the following examples in which the proportions of the ingredients are expressed in parts by weight.

Example I

A mixture of 820 parts of DDT having a setting point of 103.5° C., 4.1 parts of sodium dodecyl sulfate, and 600 parts of water is blended uniformly into a smooth paste, which is then placed in a tray over which a current of air at 50° C. is circulated. After all the water is removed by evaporation, the resulting dry material is passed through an air "Micronizer." Only 1 part (0.12% of the weight of the total charge) of DDT becomes packed into a cake in the circular grinding chamber of the "Micronizer." This amount of caking does not reduce the operating efficiency of the "Micronizer." A sample of the same DDT "Micronized" without any anti-static agent forms a circular cake amounting to 15.6% of the total charge. This amount of caking prevents satisfactory comminution of the DDT, and the "Micronizer" must be shut down and cleaned out before satisfactory grinding can be continued.

Example II

45 A mixture of 820 parts of DDT having a setting point of 102.5° C., 4.1 parts of lauryl pyridinium chloride, and 600 parts of water is blended together, after which the water is removed as in Example I. The dry material is passed through an air "Micronizer." Only 19 parts (2.3% of the weight of the total charge) of DDT packs as a ring inside the "Micronizer." A sample of DDT of similar purity and size "Micronized" without any anti-static agent being present forms a ring amounting to 12.5% of the total charge.

Example III

A mixture of 820 parts of the DDT of Example II, 8.2 parts of lauryl pyridinium chloride, and 620 parts of water is blended together, after which the water is removed as in Example I. The dry material is passed through an air "Micronizer." Twenty-eight parts (3.4% of the weight of the total charge) of DDT packs into a solid ring inside the "Micronizer."

Example IV

A mixture of 820 parts of DDT of the type of Example I, 4.1 parts of sodium dibutylphenylphenoldisulfonate (the material known commercially as "Aresklene 400") and 600 parts of water is blended together, after which the water is removed as in Example I. The dry material is passed through an air "Micronizer." Only 13 parts (1.6% of the weight of the total charge) of DDT packs into a ring inside the "Micronizer."

Example V

A mixture of 810 parts of the DDT of Example I, 4.05 parts of tallow-coconut oil soap, and 600 parts of water is blended together and then the water removed as in Example I, after which the dry material is passed through an air "Micronizer." Seventy-five (75) parts (9.3% of the weight of the total charge) of the DDT packs into a ring inside the "Micronizer."

Example VI

A mixture of 810 parts of the DDT of Example I, 4.05 parts of sodium chloride, and 600 parts of water is blended together and then the water removed as in Example I. Twenty-one parts (2.6% of the weight of the total charge) of DDT packs into a ring when the dry material is passed through an air "Micronizer."

The invention has been illustrated with particular reference to the "Micronization" of impure DDT in the presence of certain proportions of several specific salts and in a certain type of gas disintegrator; however, it is not limited to these specific embodiments. Other salts of acids having an ionization constant of at least 3×10^{-7} at 25° C. are also suitable for use in the comminution of impure DDT in high velocity gas disintegrators. Examples of other salts of this type which can be used include: potassium acetate, ammonium sulfate, magnesium chloride, trisodium phosphate, sodium benzene sulfonate, sodium petroleum sulfonate, sodium ethyl sulfate, potassium carbonate, trimethylamine hydrochloride, etc. These salts are believed to act as anti-static agents in preventing excessive packing of the DDT powder in the pulverizing chamber of the disintegrator. Salts of the stronger acids, e. g., salts of hydrochloric, sulfuric and sulfonic acids, are preferred since they are the most effective. Salts of acids having ionization constants less than 3×10^{-7} at 25° C., e. g., sodium silicate, and un-ionized materials such as sucrose are not effective in reducing packing of DDT in a "Micronizer."

Various proportions of these anti-static coatings can be used in the practice of this invention. The examples have illustrated the use of ½% and 1% (based on the weight of the DDT) of certain salts. Larger or smaller proportions, for example from 0.12 to 2%, however may be used if desired. Proportions of 1% and higher, however, are less effective than ½% of the same anti-static agent. Thus, when 1% and 2% of sodium chloride are substituted for the ½% of that salt

used in Example VI with DDT of the same purity as in that example, 6.2 and 11.0%, respectively, of the total charge of DDT passed through the "Micronizer" packs into a ring. Comparison of the results obtained in Examples II and III also shows the smaller amount of packing occurring when the lower percentages of the anti-static agent are used.

A separate dispersing agent such as for example, polyvinyl alcohol, methylcellulose, or the sodium salt of the oleic acid amide of N-methyl taurine known commercially as "Igepon T," may optionally be added to the dry mixture of DDT and anti-static agent to promote the dispersibility of the finely divided DDT. It is preferred, however, to use as the anti-static coating a salt of the above-defined type which is also capable of promoting dispersibility or wettability, e. g., salts such as sodium dodecyl sulfate, sodium petroleum sulfonate, lauryl pyridinium chloride and sodium dibutylphenylphenoldisulfonate.

The amount of water used in blending the anti-static agent with the DDT is not critical. An amount at least sufficient to produce uniform coating of the salt on the DDT particles should be used. Quantities of water amounting to 60-100% of the DDT give satisfactory results but larger or smaller quantities may be used if desired. The use of larger amounts of water will, of course, require the removal of larger amounts in the drying step, which would obviously be less economical.

This invention is of particular value in the manufacture of finely divided dispersible DDT powders since the present process provides a simple method for comminuting in a high velocity gas disintegrator such as a "Micronizer" an impure grade of DDT, which is insecticidally active, without causing frequent interruption of the grinding operation to clean out the pulverizing chamber to restore its operating efficiency.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A process which comprises comminuting in a high velocity gas disintegrator impure 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane having a setting point of from 102 to 106° C. in the presence of a salt of an acid having an ionization constant of at least 3×10^{-7} at 25° C.

2. A process which comprises uniformly blending to a paste impure 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane having a setting point of from 102° to 106° C. and an aqueous solution of a salt of an acid having an ionization constant of at least 3×10^{-7} at 25° C., removing the water from the paste and drying the particles of 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane coated with said salt, and passing the dried product through a high velocity gas disintegrator.

3. The process set forth in claim 1 in which the salt defined therein is a wetting agent.

4. The process set forth in claim 1 in which the salt defined therein is a dispersing agent.

5. The process set forth in claim 1 in which said salt is a hydrochloric acid salt.

6. The process set forth in claim 1 in which said salt is a sulfuric acid salt.

7. The process set forth in claim 1 in which said salt is a sulfonic acid salt.

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8. The process set forth in claim 1 in which said salt is sodium dibutylphenylphenoldisulfonate.

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