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INSECTICIDE AND METHOD OF MAKING

Charles B. Gnadinger, Minneapolis, Minn.

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This invention relates generally to the production of insecticidal substances, and particularly to a pyrethrum-soap insecticide, and method of producing the same.

To understand the problems concerned in the production of an ideal pyrethrum-soap insecticide, a discussion of the general subject is desirable. Oleo-resin of pyrethrum emulsified with liquid soap has been used as an insecticide. Extracts or pyrethrum in alcohol, acetone or petroleum oils, emulsified with soaps, have also been used. However, all of those substances are objectionable for one reason or another.

It is known that aqueous solutions of soap are hydrolized, becoming alkaline and thus destroying, by saponification, the activity of the pyrethrins which are esters. The more dilute the soap solutions, within certain limits, the greater the hydrolysis of the soap. In the ordinary dilute solution, therefore, the pyrethrins are quickly decomposed. The presence of alcohol also facilitates decomposition. It will be seen from the foregoing that the problem of producing an ideal pyrethrum-soap insecticide, is difficult.

The ideal pyrethrum-soap insecticide should contain a high concentration of pyrethrum, on which the toxicity principally depends; should contain the greatest possible amount of soap, so that sufficient wetting and spreading is obtained, when the product is used at high dilutions; should contain the minimum amount of water, to prevent hydrolysis of the soap, and resultant saponification of the pyrethrins, with corresponding destruction, or great reduction of insect toxicity; should be a homogeneous liquid, readily miscible with cold water; and the pyrethrum should be dissolved in an oil, preferably one having marked ovicidal and insecticidal properties.

I have extensively experimented with a large number of solvents and emulsifiers, in order to meet the above specifications for ideal soap-pyrethrum insecticides, and have finally succeeded in producing such substances.

Among other things, I have discovered that certain essential oils, such, for example, as steam distilled pine oil, and destructively distilled pine oil, are good solvents for the oleo-resin of pyrethrum. Also cedar oil, pine tar oil, various pine tar distillates are usable.

It is known that certain organic solvents such as ethylene dichloride, petroleum oils, and turpentine, dissolve the pyrethrins. I have found, however, that the amount of soap which can be incorporated with these different solvents varies greatly. Moreover, the type of product obtained when these solvents (or extracts of pyrethrum made with them) are mixed with soaps or other emulsifier varies markedly. For example, neither petroleum oils nor turpentine form homogeneous mixtures with potassium oleate, but the oils are emulsified, forming opaque mixtures which gradually separate on prolonged standing. The ideal insecticide should be, among other things, homogeneous.

Moreover, there are certain oily liquids which, when mixed with soap, become readily soluble in water. The best example is cresol which is almost insoluble in water but which becomes easily soluble when mixed with soap, as in the Liquor Cresolis Compositus of the United States Pharmacopoeia. Coal tar creosote oil with soaps forms milky emulsions when the degree of dilution is great. Soap emulsions of these oils are widely used as disinfectants.

I have found that pine oil, steam distilled pine oil and other essential oils form homogeneous mixtures with potassium oleate, and these mixtures are clear and have the appearance of true solutions and do not separate on standing. I have found that the amount of soap which can be incorporated is greatest for pine oil. The amount of soap which can be incorporated also depends upon the type of soap. Rosin soap is the least soluble of the soaps that I have investigated; potassium oleate is intermediate in solubility, while potassium-castor oil soap is the most soluble.

For the production of an excellent insecticide embodying all the properties above mentioned, the procedure is as follows: Extract with a volatile solvent, preferably using ethylene dichloride, 3000 pounds of pyrethrum flowers containing 0.90 per cent pyrethrins; other solvents such as naptha, petroleum ether and benzine can be used, or any volatile solvent which will properly extract the active principles of the flowers, that is the pyrethrins. The volatile solvent is then distilled off, in vacuum, to obtain an oleo-resin of pyrethrum. The weight of the oleo-resin obtained depends on the solvent used. With ethylene dichloride when using the amount mentioned, I obtain about 200 pounds of oleo-resin, and with petroleum ether about 110 pounds. The oleo-resin which contains substantially all the pyrethrins from the 3000 pounds of flowers (together with more or less inert matter, depending on the solvent used) is now dissolved in sufficient steam distilled pine oil or equivalent essential oil to make a total volume of 75 gallons. About 55 gallons of pine oil are required.

If the pyrethrum flowers contain more than 0.90 per cent pyrethrins, the total volume of the pine oil solution is made proportionately greater. If the flowers contain less than 0.90 per cent pyrethrins the volume of the pine oil solution is made proportionately less. The pine oil solution is thus standardized to contain in each gallon, the active principles of 40 pounds of pyrethrum flowers having a pyrethrin content of 0.90 per cent.

I next prepare a mixture of soap in pine oil as follows: Steam distilled pine oil or equivalent essential oil about 66 gallons; castor oil or equivalent vegetable oil, about 1038 pounds; potassium hydroxide or an hydroxide of an alkali metal, about 222 pounds (just sufficient to saponify the castor oil or equivalent oil as determined by titration); water, about 27 gallons. This soap is prepared with a minimum amount of heat, and after cooling to room temperture, about 75 gallons of the pine oil oleo-resin solution is added while mixing, to make a total volume of about 300 gallons.

Heat should not be employed, after the pyrethrum extract has been mixed with the soap. The use of excess alkali in making the soap should be avoided. For certain uses, potassium soaps should be used in preference to sodium soaps, since potassium is a plant food and sodium is not.

The emulsion obtained by the above procedure is clear, homogeneous, does not separate on standing, and has all the appearance of a true solution, and dissolves readily in cold water.

Pine oil and similar oils have considerable toxicity and repellent action to insects. Pine oil can be emulsified with soap containing a very low percentage of water, and is solvent for the pyrethrins.

The substance produced herein is a concentrated solution of pyrethrum in an essential oil such as pine oil which is emulsified with castor oil (or other vegetable oil) soap. The pyrethrins are first dissolved in pine oil or equivalent essential oil to prevent saponification. The product contains only 12 per cent of water which is not sufficient to cause hydrolysis of the 44 per cent of soap, and thus saponification of the pyrethrins by hydrolized soap is avoided.

I have used steam distilled pine oil, but oils such as destructively distilled pine oil, cedar oil, pine tar oil, various pine tar distillates and coal tar creosote oil can be used. If desired, derris extract, or rotenone can be added to the pine oil-pyrethrum extract.

Potassium-castor oil soap has been used as an emulsifier, but other substances such as rosin soap or soaps made from corn oil fatty acids, cocoanut oil fatty acids and the like can be used. Triethanolamine oleate can also be used instead of potassium or sodium oleate.

The above insecticide can be diluted with water for spraying plants, and the substances in this dilute solution remain in homogeneous suspension from which neither the pyrethrins, nor the pine oil, nor the soap separates. For aphids one part of the insecticide is diluted with 400 parts of water.

No spreader is added since the soap and pine oil serve the purpose. The pine oil also increases the toxicity. For leaf rollers and more resistant insects a dilution of one part of the insecticide to 100 parts of water may be used, and such a solution will not injure tender greenhouse plants. This insecticide is effective against aphids, leaf rollers and many other insects, and is also toxic to the eggs of insects.

My product is a liquid readily miscible with water which does not separate on standing, saponification of the pyrethrum is prevented, no additional spreader is required and it is more toxic to insects than ordinary soap pyrethrum mixtures having the same soap and pyrethrum content.

It is seen that the oil used in preparing my product should be one which can be emulsified with soap containing a very low percentage of water, which will take up a very large proportion of soap, and must be a solvent for pyrethrins. It is also desirable that the oil be toxic or repellent to insects. A standardized concentrated solution of pyrenthrins in this oil is prepared by dissolving oleo-resin of pyrethrum in it. A soap is then prepared in the oil-pyrethrum solution, of such concentration that the product can be greatly diluted with water without separation of the oil, pyrethrins, or soap, and still be extremely toxic to insects. The emulsion is stable.

It will be noted that I use a readily volatile solvent such as ethylene dichloride, naphtha, petroleum ether, or benzine for extracting the active principles of the pyrethrum flowers, so that the solvent may be distilled off without the use of excessive heat and thus obtain an oleo-resin of pyrethrum as the extract. In the appended claims, the term "extract of pyrethrum" is used to denote an extract of this type, that is, and extract without any substantial amount of the solvent present.

I claim as my invention:

1. The process of making an insecticide which consists in dissolving oleo-resin of pyrethrum in an oil of the group consisting of pine oil, cedar oil, pine tar oil and pine tar distillates, making a soap substantially free from alkali and containing an oil of the aforementioned group, and then mixing the oil solution of oleo-resin of pyrethrum with the oil soap to form a concentrated insecticide.

2. The process of making an insecticide which consists in dissolving oleo-resin of pyrethrum in pine oil, making an alkali free soap containing pine oil, and then mixing the pine oil oleo-resin of pyrethrum solution with the pine oil soap solution to form a concentrated insecticide.

3. The process of making an insecticide which consists in dissolving oleo-resin of pyrethrum in pine oil, making an alkali free vegetable oil soap containing pine oil, and then mixing the pine oil oleo-resin of pyrethrum solution with the pine oil soap to form a concentrated insecticide.

4. The process of making an insecticide which consists in dissolving oleo-resin of pyrethrum in pine oil, making a substantially alkali free castor oil soap containing pine oil, and then mixing the pine oil oleo-resin of pyrethrum solution with the castor oil soap containing pine oil to form a concentrated insecticide.

5. A stable and readily water soluble insecticide consisting of a pine oil solution of oleo-resin of pyrethrum and a substantially alkali free vegetable oil soap.

6. A stable and readily water soluble insecticide consisting of a pine oil solution of oleo-resin of pyrethrum and a substantially alkali free castor oil soap.

7. A stable and readily water soluble insecticide consisting of pine oil, oleo-resin of pyrethrum, and a substantially alkali free potassium castor oil soap.

8. A stable and readily water soluble insecticide consisting of a solution of oleo-resin of pyrethrum in an essential oil of the group consisting of pine oil, cedar oil, pine tar oil and pine tar distillates and a substantially alkali free vegetable oil soap.

9. A stable and readily water soluble concentrated insecticide consisting of pine oil, oleo-resin of pyrethrum, and a substantially alkali free potassium castor oil soap, said soap constituting at least substantially 44 per cent of the product.

CHARLES B. GNADINGER.