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PESTICIDE DEVELOPMENT PROGRAMME IN INDIA

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INDIA

Technical report: Findings and Recommendations*

Prepared for the Government of India
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Wade Van Valkenburg,
consultant in pesticide formulations

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Vienna

* This document has not been edited.

C O N T E N T S

Explanatory Notes

UNDP (United Nations Development Programme) sponsors PDPI (Pesticid Development Programme India) under the management of HIL (Hindustan Insecticides Limited), a Government of India enterprise. As part of its sponsorship UNDP supports the visits to PDPI of technical experts such as the writer, Dr. Wade Van Valkenburg, a UNIDO expert on pesticide formulations.

PDPI has its offices and laboratories at the HIL research complex at Gurgaon in an industrial complex a short distance from New Delhi. This report covers the period of 21 November 1988 through 15 December 1988, which constitutes the writer's third mission and fourth visit to India under UNDP sponsorship. Previous visits were for the periods 21/2/86 - 8/4/86; 17/1/87 - 28/3/87 and 2/11/87 - 27/11/87.

Acknowledgement:

Although this is the author's fourth visit to India under UNDP sponsorship, it appears that each visit becomes more productive than previous visits. The trust, confidence and friendships previously established render an excellent foundation for continued progress.

I express my sincere appreciation and thanks for support and cooperation to the following people:

Shri S. Suri, Joint Secretary in the Deptt of Chemicals; Dr. S.P.Dhua , Chairman of HIL, Dr S. Khetan, General Manager,HIL R&D and PDPI and PDPI personnel, Dr. R.K. Khandal, Dr S Kumar, Dr. S.Y. Pandey, and Dr. P.K. Ramdas

Summary of Conclusions and Recommendations

Conclusions:

1. As phase I of UNDP support draws to a close PDPI has now established a "Centre of Excellence" for pesticide formulation technology.

2 PDPI's performance to date has been examined and approved by an UNDP examining committee which has recommended continued support of the project for a five year period.

3. PDPI , by consensus, has met four of its five objectives for Phase I and has delayed the fifth objective into the phase II period.

4 A new set of objectives, goals and plans have been formulated for phase II

5 The examining committee's constructive recommendations, the recommendations of experts and other interested parties are being studied and prioritised for incorporation into the phase II plans.

6. A new programme to offer analytical services at cost to the pesticide industry is being established. Such a move is worth while and should be encouraged to facilitate PDPI in meeting its objective of technical assistance to the pesticide industry.

Recommendations

1. PDPI should be continued for five years in a phase II programme.

2 Primary objectives for phase II should only be (a) the strengthening and improving of the pesticide industry in India and (b) acting as technology transfer agent on pesticide formulation technology for the RENFAP organisation.

3 PDPI should establish criteria for the annual measurement of its success in achieving its objectives. For example, if PDPI's primary objective is the technical upgrading of the pesticide industry in India they should report the number of industries assisted and the rupee value to the public of such assistance.

4 All suggested prospective goals for PDPI should be prioritized for action and incorporated into the phase II plan.

5 A plan of action should be initiated to meet these goals and objectives, said plan of action to be appropriate and consistent with available resources.

6 Technical services and the determination of the technical needs of the pesticide industry in India should be given the highest priority.

7 PDPI's activities should now be balanced between R&D and technical service with each group; analytical, formulation, minerals and pilot plant, initiating and carrying out a technical service programme.

8 Ascertainment of the technical needs of India's pesticide industry and the establishment of a PDPI technical service reputation should be accomplished prior to efforts to establish society status for PDPI.

9 Increased service should be administered to PDPI industrial members

10 The new Indian kaolin material which appears to be lighter, more neutral and lower in catalytic mineral elements should be investigated for compatibility with malathion. If the clay is substantially better than currently available materials, the clay should be characterised, patented and offered to private industry.

11 Since it appears that the formulation being developed for mosquito larvae control constitutes a significant scientific advance, a patent application should be persued aggressively.

12 PDPI should increase their efforts to transfer their formulations technology to the private industry.

13 New Hires : a minimum of four new employees should be considered for PDPI (a) a PDPI laboratory and technical service manager (2) a PDPI industrial membership administrator and (3&4) two technical service representatives. Now that Dr. Khetan has been elevated to the position of General Manager of the R&D complex he has inadequate time to administer the daily R&D and technical service activities of the staff. Creation and filling such a position will expedite programmes and facilitate planning.

Administration of the interface between PDPI and its membership is a full time operation. In the interest of developing this organisation a full time administrator should be explored.

Finally, the greatest unfilled need for PDPI is to develop a relationship that will learn the technical needs of the industry and to build a relationship that will bring in purchased R&D programmes for PDPI . If PDPI is to develop society status it must show that it can generate revenue to support at least half of its costs of operation. To expedite and accelerate this revenue generating programme two technical service representatives should be hired and an adequate rupee budget established to support their travel and interaction with the industry.

Technical service should be understood to mean responsiveness to any technical need of the pesticide industry. This does not mean merely demonstrating the new advanced formulation technology but means assistance with such problems as improved grinding of wettable powders, providing assisitance in stabilising existing malathion formulations, reduction of dust in a factory, better packaging, better emulsifiers, better ventillation and air flow and a host of other poroblems.

Many pesticide manufacturers in India will be reluctant to avail themselves of this service Their thoughts will be that any PDPI suggestion will cost them money and thereby cost them profits. They will fear that PDPI will notify the government of delinquencies and the result will be government interference with their operation.

But, some industries will listen, they will spend the money to improve the quality of their operations and the quality of their products. Once a company begins a quality programme the word spreads, reputations improve and educated and informed customers will buy only the quality products. Then the others must incorporate quality or they will not servive.

14 PDPI should install showers and eye wash fountains for the laboratories. The safety showers should be installed in the walkway area in the vicinity of the door of each laboratory.

Activities and Observations

I RENPAP

During the month of november PDPI conducted a workshop on the quality control of pesticide formulations. This workshop was well attended and focused on analytical methodology and hands on training utilising HPLC, GLC and other analytical techniques used in the analysis of pesticides.

It was obvious that this workshop had benefited from the recommendations and observations of previous workshops. Much more hands on training gave the attendees better training in the quality control of pesticides. It is this writer's opinion that the attendees were very well satisfied with the workshop.

Two lectures were given in this workshop. Titles and abstracts follow:

(a) Quality : The route to survival

Abstract : The history and necessity of the quality movement as a management tool was outlined. The seven steps of a quality programme were illustrated by an example (Marquette National Bank of St. Paul, Minessota) followed by the application of the quality management process to an analysis of the RENPAP programme.

(b) Quality: The consumer point of view.

The primary thrust of any quality proqoramme is customer satisfaction. The relationships were pointed out between analytical tests (which were enumerated on pesticidal formulations and raw materials) with customer expectations and satisfaction when they purchase and use a pesticide formulation.

II PDPI

A Technology Transfer

Additional tools are needed to assist in the transfer of pesticide formulation technology from PDPI to the pesticide industry. In the appendix to this report is a suggested format for a "Specification sheet" on one of the PDPI's developed formulations. A sheet such as this could contain the entire message when printed on two sides of a single sheet of paper. Such a fact sheet should be distributed to Indian pesticide companies to solicit interest in the technologies. Then each mailing should be followed up by a phone call or personal visit by a PDPI representative.

B Bacillus Sphaericus Formulation

The technology supporting this new formulation for the control of mosquito larvae is very significant. To this patent attorney the product has a high potential for patentability and a vigorous programme for filing a patent application should be pursued. A skeleton draft of a patent application outlining first principles of the invention, suggested examples and the framework of a claim structure has been written up and is appended to this report.

C. Kaolin Deposit

PDPI has a programme for the determination of the suitability of indigent mineral deposits for use in pesticide formulations. PDPI has identified one kaolin deposit that is of low density, neutral in pH and low in catalytic mineral moieties. There is a great need in India for discovery and use of a better diluent for use with malathion. This new diluent should be checked for compatibility with malathion and if found suitable should be promoted for use in the private sector.

D. Society and Technical Service

PDPI has 29 companies that are "members" of PDPI. PDPI also needs to provide sufficient services to their membership such that they will wish to continue their association with PDPI.

Therefore, it was suggested to PDPI that they offer one free technical service to their membership. Such service could assist PDPI learning more of the technical needs of private industry and have the additional benefit of cementing relations with their membership.

E. New Technologies

PDPI and HIL were put in contact with two new technologies. Mr. Joseph Haynes, President of ACD Inc, was introduced to Dr. Dhua. Mr. Haynes explained and offered to HIL new technology involving a stabilized propionic acid formulation that prevents the onset of aflatoxin on maize, ground nuts, wheat, etc. HIL is interested in the technology and is exploring it more fully.

Mr. A.J.A. Bud Brown, Senior Liaison Officer of Johnson Wax will meet with Dr Dhua the day this manuscript is being typed. He will offer to HIL a partnership for the sale and manufacture of household insect aerosols. These products, as well as the piopionic acid composition above, will be manufactured in India.

F. Public Relations

This writer was very fortunate to have three oppotunities on this mission to give some degree of publicity to the PDPI effort. On Thursday, December first, we were the invited guest on a national TV morning program hosted by Mr. Deepak Vohra. In two segments, seven minutes in total duration, Mr Vohra asked many questions relating to PDPI and the pesticides Industry in India.

In a 30 minute radio interview (Delhi Channel B) taped on 1/12/88 and aired on 4/12/88, Dr. Dhua, Mr. Noori (Afganistan) and this writer responded to questions on pesticides, PDPI, RENPAP, safety of food etc.

On thursday, December 8th, at this writer's invitation, Mrs. Patricia Haslach, agricultural attache in the American Embassy, visited the HIL complex in Gurgaon. She was very pleased to have learned about this program and will revisit Gurgaon with several of her associates at a future date.

G. Industrial Liaison

During this mission there were public announcements of three new pesticides being introduced into India. The three pesticides were methomyl, thiazophos and fenpropethrin. Indian commercial outlets for these material will be Coromandel, Rallis and Hoechst India. A draft letter to Coromandel and Rallis was submitted for management review. The letter proposes a joint R&D effort to develop a dispersible granule formulation for methomyl and an emulsion concentrate for fenpropethrin.

A two day trip to Bombay was scheduled on two occasions to visit pesticide manufactures and laboratories. Unfortunately, on both occasions, it was impossible to get confirmed seating on flights to Bombay and the trips were cancelled. This writer regrets very much the inability to make such contacts during this mission. PDPI must establish close relations with the pesticide industry in India and to learn of their technical needs. If this consultant should have the pleasure of another trip to India, a minimum of half of the mission should be spent in preprogrammed and advanced air travel booking to primary pesticide companies in all segments of India.

Summary

A review of the observations, recommendations and activities enumerated in this report illustrates the fact that the focus of this mission was on marketing, planning, public relations, patents, transfer and acquisition of technology, etc. There was little activity this time directed to the improvement of the technical expertise of the staff. As this program shifts to phase II this change in the activities of consultants will increase in importance.

Sample format

Formulation Specification Sheet

Carboxin 40 SC

Active Ingredient

(5,6 - dihydro - 2 - methyl-1 4-oxathi-ine-3-Carboxanilide)

Use

Carboxin is a systemic seed and foliar treatment fungicide which controls basidiomycete type of fungi, such as smuts on barley, oats and wheat

Composition

Carboxin	41.5%
Dispersing /wetting agent	7.0%
Suspending agent	0.35%
Humectant	9.0%
Water	Balance

Physical Properties

Appearance	Pinkish red stable suspension with a faint odor
Specific gravity	1.14
pH	7.0
Viscosity	750 cps
Setting	Less than 1%
Particle size	90% (5 um 50% (2 um

Registration Status Pending

Manufacturing Process

1. Blending
2. Wet milling
3. Packaging

Manufacturing cost per liter of 40% carboxin

Raw materials	125.61 Rs
Packaging	21.66
Utilities	1.00
Overhead	11.09
Total	159.36

Quantity form req./kg seed	2.825g
Cost/kg seed(before mark up to consumer)	Rs. 0.57
Cost savings over 75% wp	14%

Laboratory Bioassay for 100% control of loose smut in wheat

Carboxin 40 SC	1.130 g
Carboxin 75 wp	1.50

Features and Benefits of carboxin 40 SC over Carboxin 75 WP

1. Cheaper to customer for unit package
2. Freedom from dust
3. Greater ease of handling
4. Reduced mammalian toxicity
5. Reduced phytotoxicity
6. Non-flammable
7. Ease of dilution with water
8. Improved biological activity
9. Better and more uniform seed coating
10. More economical for user

Suggested letter to PDPI members

Member
Society of Pesticide Formulations

Dear

In an endeavour to learn more about our members and their pesticide formulations needs, PDPI is offering to society members a one time free service in any of the following areas;

Area (1) Quality Control check on one of your finished formulations. This quality control will include the following:

(a) % active ingredient

(b) measurement of critical physical properties appropriate to type of formulation

(c) Suitability for use in application euipment

(d) Bioefficacy

Area (2) Formultion stability studies

(a) determine formulation shelf life in and outside packaging container.

Duration 3 months
Temperture 50 C

Area (3) Safety inspection of manufacturing plant will include recommendations for improvement.

Area (4) Inspect manufacturing process and recommend methods of improvements.

A second phase of service is available if the member chose area (2) for his free service and investigation shows a problem. At the reuest of the member a formulation improvement development programme will be conducted with the member paying only 50% of the cost of services. Members are assured that the results of any of these investigations will be held confidential between PDPI and the member requestor. Such information will not be divulged to HIL, the Government of India or any other third party without the expressed permission of the member reuestor.

I look forward to your early response.

Very truely yours.

Dr. WADE VAN VALKENBURG, UNIDO REPORT SUPPLMENT

Skeleton Patent
Application

Title Water Habitant Insect Control Composition

Abstract The process of controlling water habitant insects involves the use of a composition consisting of an insecticide, a hydrophobic oil, an oil and water soluble aliphatic alcohol and an hydrophobic surface active agent; said composition being spread upon water containing insects, whereupon the resulting thin film of insecticide composition spontaneously breaks into micro particles which subsequently are ingested by the target insects.

Mosquito larvicidal formulations freuently are comprised of an insecticide solubilised into an oil based formulation which, when placed on water, spreads to a uniform film on the water. To affect biological activity the mosquito larvae must come in contact with the spread film when the larvae come to the surface of the water. The composition forms a film on the larvae and controls the insect through contact action.

Such mosquito larvae control film on water suffer the disadvantages that the film is frequently broken open by wind and/ or wave action. Such broken films allow the larvae to escape the lethal action of the insecticide. Other disadvantages include (1) thin films of such pesticides are easily degraded by sunlight and other environmental factors and (2) the film may deposit on the insect too small a quantity of insecticide to cause mortality of the insects.

Detailed Description of Invention:

We have discovered a unique insecticide composition which offers a unique process for the control of insects that live in or on water. The process for the control of insects is as follows

(1) The composition spontaneously spreads to a thin film on water;

(2) This thin film then spontaneously breaks into microparticles containing insecticides:

(3) Mosquito larvae and other water insects ingest the micro particles and succumb from the toxicity of the insecticde.

This process is illustrated in the three pictures A, B, and C.

In picture A the insecticide composition has been spread upon water and spontaneously forms the rivulettes containing very small nodules.

In picture B the nodules are growing and the connecting capillaries between the nodules become smaller and weaker.

In picture C the nodules have broken apart into separate microparticles containing an insecticidie. A mosquito larvae is shown at the water surface feeding on the microparticles.

The composition suitable for this insect control process has a number of advantages over compositions of the prior art. These advantages include:

(1) The composition spontaneously forms micro particles when placed in contact with any water surface.

(2) Because microparticles are formed the insecticide is degraded by sunlight at a much slower rate than will occur in insecticides cast on water in the form of a thin film.

(3) The taste of the insecticide is masked from the feeding insect by a thin film of oil which is edibly attractive to the insect.

(4) The size of the microparticles is appropriate for ingestion by water habitant insects.

The composition of matter, the specific ingredients and their physical properties are described below.

The composition : The composition must be a liquid at tempertures about 40 F and said liquid must spontaneously spread upon the surface of water. In order for any liquid to spread upon water there must be a favourable relationship between the surface tensions (surface energy) of the composition of water and of the interface between the composition and water.

The surface tension of the oily composition is identified as γ_o the surface tension of water as γ_ω and the interfacial tension between the two phases as $\gamma_{o/\omega}$. In order for the composition to spontaneously spread there must be a positivie spreading coefficient (S) which is defined as an inbalance of surface energies. The spreading coefficient is mathematically expressed as

$$S = \gamma_\omega - \gamma_o - \gamma_{o/\omega}$$

Thus for the insecticide composition in the instant invention, S must be positive.

The insecticide : The insecticide must be toxic enough to control the pest, must be a solid particulate material having a particle size of 5 microns or smaller and must be substantially insoluble in the oil phase of the composition. It must also be substantially insoluble in water and hydrolytically stable.

Some small solubility of the insecticide in the aqueous and oil phases is tolerable. In the oil phase solubility of the insecticide should be low enough so that the eventual microparticle will still be edibly attractive to the insect. Solubility in the aqueous phase should be fairly minimised to insure that the microparticles have an adequate period of biological activity. The insecticide may be present in the composition at a minimum of 0.1% to a maximum of 76%

The oil : The hydrophobic oil must be water insoluble and primarily comprised of aliphatic hydrocarbons. It may be comprised of a single oil or a combination of oils. The oils may be a vegetable oil type, petroleum distillate or a synthetic oil that is fundamentally hydrophobic or insoluble in water. It must have a specific gravity less than one. The composition will contain oils in a percentage by weight of from between five and thirty per cent.

Alcohol : The most important physical property of the alcohol is that it must have between the selected oil and water, a partition coefficient in the vicinity of one and optimally between 0.1 and ten. By the term partition coefficient one means the ratio of the solubility of the alcohol in the oil to the solubility of the alcohol in water. By indicating a partition coefficient in the vicinity of one it is stated that the alcohol must be approximately equal in solubility in both water and oil. This is a unique property which occurs with alcohols up to about four or five carbons. The higher molecular weight alcohols are more desirable as their solution into the aqueous phase takes place at a most desirable rate. The alcohol must be primarily aliphatic in nature, branched or unbranched, saturated or unsaturated and may be primary, secondary or tertiary. The alcohol is present in the composition at a percentage by weight of between five and thirty per cent.

The Surfactant : A surface active agent is defined as a molecule which tends to have a higher concentration on the surface of a liquid, with which the surfactant is mixed, than present in the bulk of the liquid. To facilitate this type of behaviour a surfactant is frequently comprised of two segments, one lipophilic (oil loving) in nature and one hydrophilic (water loving) in nature. An hydrophobic or lipophilic portion is frequently comprised of alkyl hydrocarbons or alkyl aromatics that contain a reactive alcohol or acid functionality. An increase in the degre of lipophilicity is accomplished when some propylene oxide is appended to an already lipophilic moiety. Hydrophilicity is accomplished by esterifying an alcohol or adding ethylene oxide to the molecule. A surfactant of choice is made by the Wyandote ChemicalCco and sold as Pluronic..... It's composition is. . . . . .

The physical properties of surfactants are characterised by a numerical system called HLB; hydrophilic lipohile balance. It is a numbering system with the figures running from about one to twenty five. The high numbers are very hydrophilic while the low numbers are lipophilic. It has been ascertained that the surfactants of choice in the invention must have an HLB of seven or less. The surfactant will be present between one and thirty percent by weight.

Other Ingredients : The composition is not limited to the aforementioned ingredients. Other materials may be added such as suspension stabilizers, antioxidants, U.V. stabilizers, buffers and the like.

The above composition is compounded or processed, in the instance of a solid insecticide, by wet milling the insecticide, in the presence of an equivalent amount of the oil of choice, until the particle size of the insecticide is all substantially at five microns or below.

The balance of the ingredients are then gradually added by conventional mixing techniques.

It is believed but unproven that the process of forming discrete microparticles on the surface of water is accomplished by means of the following sequence of events:

(1) The insecticide composition is placed on the surface of water whereupon, because of the positive spreading coefficient, the oily liquid spreads to a thin film on the water.

(2) The alcohol begins to diffuse to the oil/ water interface and begins to solubilize in the aqueous phase. This tends to increase the interfacial tension, causing the spreading coefficient to become negative. This causes the film to break and the film on the surface to retract into rivulettes

(3) As the alcohol now continues to diffuse away from the oil/ water interface, the surfactant increases in concentration at the interface. This happens because the surfactant is insoluble in water and hence, as the alcohol solubilizes in the aqueous phase, the surfactant is left behind at the oil / water interface at an unstably high concentration. The rate of diffusion and dissolution of the alcohol will be greater in areas where the rivulettes have the highest radius of curvature. This means that the interfacial tension in these regions will be higher in the region of a higher radius of curvature than it is in the capillaries. This causes an imbalance of surface tension, called a "Marangoni effect", causing the oil in the capillaries to move to the nodules to equilibrate the surface energies.

(4) The nodules grow at the expense of the capillaries until the capillaries are depleted and the nodules become individual and discrete microparticles.

This invention is further illustrated but not limited by the following examples.

Note : Examples should illustrate the following:

Each example will be to illustrate a single point. Thus, to show oils that are satisfactory, make a single type composition where the insecticide, surfactant and alcohol are constant and the type of oil is varied. Show how this variation affects the formation of microparticles. Hence the following examples should be developed to illustrate the invention:

(1) Vary the oils. Then having selected the most viable oils, characterise them in terms of chemical and physical properties.

(2) Vary the alcohols to determine those that are most satisfactory. Again characterise them in terms of chemical and physical properties. Critically determine the minimum and maximum concentration of the most desirable alcohol. If possible measure the partition coefficient of the alcohol between the chosen oil and water. This might possibly be done through measurements of the index of refraction.

(3) Vary the surfactants as a function of HLB. Try several at the optimum HLB and characterise them in terms of structure and physical properties. Critically determine minimum and maximum concentrations.

(4) Vary the insecticide. First try several solids to see if there is any difference in performance. Secondly try some liquids. Does it make any difference whether or not the liquid insecticides are soluble or insoluble in the oil phase?

Will mosquito larvae refuse to eat an insecticide that has partial solubility in the oil? If the insecticide is a liquid, is it dispersed in the oil? Is particle size of the dispersion critical?

Suggested claim structure:

1. Claim to a process of controlling water habitant insects using a composition containing the four ingredients in general terms.

2. Claim to a composition of matter for the control of insects and describe composition in general terms.

3. Dependent claims describing a class of a specific ingredient in general terms.

4. Dependent claims giving examples of members of the class.

5. Dependent claims giving preferred individual ingredients.