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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

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^{*} This document has not been edited.

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Acknowledgement

The author would like to acknowledge the help and assistance given to him by all the staff at the Pesticide Development Centre but particularly to Dr. Kawal Dhari the Director of the Centre for his help in all administrative and other general matters.

He would also like to thank Dr. Ramdas with whom he worked on a daily basis on matters relating to formulation technology.

Introduction

The Institute of Pesticide Formulation Technology (IPFT) was started as a joint UNDP, Indian Government sponsored project in close cooperation with HIL who provided the site and staff. This project has now been running for approximately 7 years and a wide range of experts have visited the site making recommendations for equipment and training in the areas of formulation development, analysis and biological testing.

Many of the recommendations were taken up and today, the Centre is very well equiped for the development of wide range of formulation type as well as for analysis of pesticide and their formulation.

Main objectives of visit

This was mainly follow up from several previous visits to help to consolidate tile background on existing formulation and to bring the Centre upto date a current European features.

The following is the basic programme drawn up by Dr. Ramdas and was basically designed to enable a review of progress to be made on some current major projects which had developed out of earlier work and also to help start up some new projects.

1. Isoproturon 50 SC

- 1.1 Review of the studies conducted on the development of Iso proturon 50 SC for M/s. Gharda Chemicals.
- 1.2 Suggestion for improvement

1.3 Editing of the technical report prepared

2. Isoproturon 75 WG

- 2.1 Review of the studies conducted on the development of Iso proturon 75 WG for M/s. Gharda Chemicals
- 2.2 Suggestions for improvement
- 2.3 Preparation of a standard specification of the product
- 2.4 Guidance on the scaling up studies

3. Copperoxychloride

- 3.1 Discussion with scientists of M/s. IOC Ltd., sponsor of the Project
- 3.2 Guidance on the development of the formulation

4. Spray granulator installation

4.1 Guidance on the installation / commissioning of the pilot plant model spray granulator.

5. Discussions

- 5.1 With Dr. gharda / Dr. Malte of M/s. Gharda Chemicals on the on- going projects and future possibilities
- 5.2 With Dr. Krishnamoorthy of M/s. Montari regarding the possible assistance that could be offered by IPFT on the development of new generation formulations.

Based on this program the discussions carried out are detailed below:

Isoproturon flowable (Item 1 to 1.2)

Much of the earlier work on this formulation had been consolidated and good quality formulations can now be produced. Work is continuing to try to use the cheaper locally available xanthan / guar gum mix to replace the use of xanthan gun alone, is giving very promising results.

It is very pleasing to see that the work is now being carried out with the objective of having a formulation suitable for passing on to M/s. Gharda Chemicals as a sponsored project.

Few additional recommendations could be made in this area as the IPFT have already good experience. However, it was possible to suggest the following slight modification.

In the course of preparing a flowable it is normal to just add the xanthan gum as a solution after milling. However, it is suggested that a small amount of the gum, between 10% and 30% of the weight of the gum, is added before milling.

Although it is known that some loss of structure occurs, this does ensure that some gum is well dispersed over the solid and thus helps to prevent aggregation and settling between the milling and the gelling stages. Care must be exercised to avoid this addition increasing the viscosity.

Trials were carried out on this basis and very promising results were achieved.

Isoproturon 75 DG (Item 2 to 2.4)

Once again the local team have maintained their expertise in the use of the laboratory Aeromatic for producing good quality water dispersible granules of Isoproturon. The main problem at present is that the same level of suspensibility cannot be achieved as with wettable powders.

The solution of the problem is important because M/s. Gharda Chemicals are also interested in this product with a view to export sales and for these markets a high quality is necessary.

Some discussions were carried out on this topic which led to the following suggestions.

i) Addition of a small quantity of xanthan gum at the preliminary milling stage.

The objective behind the proposal is to try to use the Xantham Gum as a protective colloid and thus help to prevent particle which have been over dried becoming aggregated to strongly. It was suggested that 0.02 - 0.03% level would be a useful start ing point.

ii) Addition of a small quantity of a polyethylene oxide

The reason for including a polyethylene oxide in the formulation again before milling is to try to arrange that a s the spray dries the particle interface is predominantly polyethlene glycol with little water present. Because of the high solids content of the mill base a level of only 1-2% is recommended at this stage but the effect of adding a further quality after milling must be examined.

Trials were organised to see if either of these proposition gave any improvement in the suspensibility of the product. Preliminary tests indicate at least that there is no adverse effect. This is continuing.

Copper oxychloride (Item 3-3.2)

This is a project sponsored by M/s. Indian Oil Corpn Ltd (IOC) and being actively followed by the R.D. General Manager Dr. Bhatnagar. The main background to this project is that IOC sell spray oil for use in conjunction with copper oxychloride (CuO Cl2). The copper

oxychloride is used as an oil dispersible powder which is dispersed in the oil at about 17-20% by weight.

The interest of IOC in this project is in the possibility of extending the use of these oil by the having a ready to use formulation. Because IOC has no pesticide formulation background, the project was referred to IPFT.

As a starting point for this study a visit was made to the IOC R&D Centre at Faridabad. This visit included a presentation by the staff of the R.D. Centre on the formulation additives used in the oil industry.

The main additives used are the following:

- 1. Detergent such as sulphates and sulphonates
- 2. Dispersant succinimidet, thiophosphonates, lauryl, taurates and Mannich bases e.g. alkylene formaldehyde
- 3. Anti oxidants phenols e.g. BHT

Amines e.g. phenylene diamine

zinc dialhyl dithio phosphonates

- 4. Metal deactivators chelates
- 5. Antiwear-tricresyl phosphate

amine phosphates

xanthates most used are sulphur based for use at high temperature

7. Pour point depressants

Mostly copolymers - mixed vinyl/ methacrylate vinyl/svccinimide

8. Viscosity control - olefine copolymets

dispersant clays such as Bentone 38

In subsequent discussion it was felt that the most useful additives were the detergents, dispersants, viscosity control agents, and thickening clays. Sample of each of these products were taken away for trials at IPFT.

This presentation was followed by a visit to the research laboratories where there was a very impressive display of test equipment. Most of the equipment was specific to engine oil requirements. However there was also equipment which could be of interest to IPFT. In particular IOC have a scanning election microscope and an x-ray diffractometer. Both of

these could be useful to IPFT if IOC are willing and could give useful information in formulation development. The scanning miscroscope for looking at surface coatings on leaves etc and the x-ray diffractometer for detecting any morphological changes in active ingredients from different sources.

Following this visit a programme of work was started to see the effect of these various additives on a 20% CuOCl₂ formulation. Samples were prepared containing blends of the liquids i.e. a sulphonate, succinimide and a viscosity improver. These tests after 24 hrs showed that the sulphonate was a good flocculant and there was no hand settling whereas both the succinimide and the viscosity improver gave high wetting and hence hard settling.

Trials with the clay showed that although there was only a small increase in viscosity, good suspension of the CuOCl₂ was achieved with the clay alone.

It was apparent from these trials that work should be primarily directed towards optimising formulations based on the clay alone.

One further problem with the project was a lack of background data with reference to the ULV products use. To help overcome this the following check list of information was drawn up.

Rubber Spray Oil Formulations (Copperoxychloride OP in Oil - Applications).

Details Needed:

- 1.a) How the formulation is applied?
- b) Type of spraye. employed
- c) Nozzle diameter, flow rate
- d) Mesh size in the nozzle
- e) Pressure at which the formulation is pumped out
- 2 a) Copperoxychloride application rate per hectare
- b) Spray volume per hectare
- c) Droplet size range requirement
- 3a) Procedure for the preparation of the spray solution

- b) Specifications of the spraying solution
- c) Any other important characteristics from their experience
- 4. Please arrange tos upply samples of copperoxychloride OP and the spray oil
- 5.a) Have they ever tried any pre-formed oil flowable formulation of copperoxychloride before?
- b) If so can we have a sample and specifications?
- 6. Any other relevant experience in other areas where formulation of the type are used.

Although the questionnaire was specifically produced for this proejet it is a useful guide for other sprayed products and generally speaking the answers to these types of questions will be helpful in working on any project.

Dodine flowable to M/s Montari (Item No. 5.2)

Dr. V. Bhushan and Mr. Mukherjee from Montari Chemical Industries brought a mill base for a dodine flowable to be milled and the particle size determined at various stages of milling. They did not provide the composition of the formulation but said that they had used an emulsifier.

The sample was milled but with increasing milling the particles had obviously aggregated as the average particle size began to increase. Further milling did not improve this.

Examination of the sample in the Malvern showed severe flocculation and indications of some dewetting. Further discussion which took place and the following recommendations were made.

- i) Try variation of emulsifier concentration as the effect seemed to be one of poor weeting.
- ii) Try alternative wetting agents, particularly non-ionic ones since dodine is a zwitter ion, use of anionic materials could be a problem.
- iii) Try using a structuring agent such as xanthan or other gum

In addition it seems that very fine milling is not necessary in this case since the density of dodine is apparently very close to one.

This was a very useful piece of public relations work as both Dr. Bhushan and Mr. Mukherjee of Montari left with a very good impression of the ability and technical background of the

staff of IPFT. This was particularly important since previously they had been to ICI for similar work and had not got any useful advice.

General Discussions (Item 5)

General Discussion were held with various persons at IPFT and are generally summarised below:

Dr. Ramdas:

The main formulation expertise of the Centre is in flowable and water dispersible granule formulation and this is the area on which the formulation unit must concentrate. Additionally this should be expanded into suspoemulation and oil based flowables.

Work on concentrated emulsion and solubilised formulation is important as an area where IPFT could become a leader. However, this work is of less immediate interest.

Mr. Kumar and Mr. Sharma respectively.

Discussion with both these persons can be summarised together as both related to hazards and environmental safety.

Mr. Kumar requested information on safety training that is available in Europe as they are trying to introduce more safety training using videos, posters and booklets.

Both Mr. Sharma and Mr. Kumar were given an example of a stand alone effluent treatment unit and Mr. Sharma was given an example of an incinerator. The discussion covered safety manuals, safety data sheets, COSHH regulations and hazop studies. Information on all these aspects was requested and the author will try to provide this.

Visits

Visits were made to Indian Oil Company and the Indian Agricultural Research Institute.

The former visit is discussed above. The second visit was made at the invitation of Dr. Parmar to discuss in general term the work of both units. During the latter visit a short presentation on formulation technology was given.

General Conclusions

Despite the relatively short time of this visit some very useful work was carried out as detailed above. The most important feature was that the Formulation Team under Dr. Ramdas had maintained and improved on their technical experience particularly in the

fields of flowables and water dispersible granules. Further more the work with copper oxycholoride formed a good basis for developing expertise in oil based flowables for ULV application. This project is particularly useful as it combine both formulation and application.

Because of the change in source of funding it is now most imprant that IPFT acquire as many funded projects as possible. These provide both a source of income and publicise the work of IPFT. At the present time the targets should be the larger independent or semi independent formulators and the following list is fairly comprehensive.

Gharda Chemicals

Excel Industries

Montari Chemicals

Rallis India

NOCIL (Shell)

The smaller formulation units are probably not in a financial position to fund the projects and this is the reason for targeting the above list.

Further extension of the use of the pilot scale area by loaning the facilities to other companies could be a useful source of income for the Centre but there is still a lack of packaging equipment which would be necessary to operate this procedure properly.

The main area that needs further work is that of effluent treatment and safe working practice. In the laboratory it would be useful to have some safety posters for such things as using protective clothing. Additionally it is important to continue to emphasise the dangers of using solvents such as benzene and methanol in laboratories.

The discussions on effluent with Mr. Sharma were very useful and indicated the desire of IPFT to introduce some effluent treatment. This is very important as this is a serious lack in the facilities of the Centre. This is becoming an important area of concern since the local area around IPFT is gradually being developed and it is important that no contamination of ground water occurs.

Recommendations

- 1) The work on IPU WDG should take priority and proceed along the lines discussed to get better suspensibility.
- 2) No change in the proposed specification shall be made until the product has been made on the pilot plant scale. Aeromatic

- 3) High priority should be given to installing the pilot scale aeromatic and having available all the ancillary services.
- Sufficient materials must be availabel to enable long enough trials to be carried out on the Aeromatic. This quantity should be based on the recommendations of the supplier.
- Works should continue to optimise the level of bentone and succinimide in the copper oxychloride flowable to get a viscosity suitable to the method of application and which gives good storage stability in 18 months with no hard settling and less than 10% supernetant.
- 6) Carryout trials in conjunction with the application department to establish the vis cosity limits for the oil flowables.
- 7) If possible to build on the oil based flowables by working with other active ingredients such as carbaryl.
- 8) A further visit is made around March 1992 to coincide with a visit from the Aeromatic specialist who will commission the pilot unit. This could also be usefully combined with a Formulation Training workshop.

UNIDO COMMENTS

The findings and recommendation given in this report are very relevant since the expert has been associated with the project for many years and made a number of missions. His report definitely gives the impression that changes which have been taken place in the PFTI will be of great benefit to the pesticide industry. His recommendation for proper balance of applied and basic research is an important one so that the PFTI will be looked upon as an important institution both by industry, universities and other institutions.

It is also important that some international experts especially those who worked or working in multi-nationals and other reputable institutions should be used as spokenmen for PFTI by asking them to visit other institutions to promote links to make use of PFTI.

The PFTI is on the threshold of becoming an institute of recognition both in India and abroad. In order to facilitate we suggest separate budget is provided in proper maintenance of laboratories and the buildings and also solving the pernnial problem of unreliable power supply.