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RESEARCH ARTICLE

Effects of changes in Extrusion Process Variables upon Quality Parameters of Water Dispersible Granules formulation of Insecticide Deltamethrin

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Abstract

The effects of changes in extrusion process variables upon the quality parameters of Water Dispersible Granules (WG) of insecticide Deltamethrin were studied. Deltamethrin Water Dispersible Granules composition was optimized and different batches were prepared in the laboratory with changes in major process variables viz. amount of water added into extrusion powder, rotation speed of extruder and drying temperature. The effects upon the quality of WG with respect to major quality parameters i.e. suspensibility and dispersibility were studied. The amount of water added into extrusion powder shown remarkable effects on suspensibility and dispersibility. The samples prepared with less amount of water shown better suspensibility and dispersibility and vice-versa. The extruder rotation speed also affected the quality of WG. The samples prepared at low extruder speed shown better results of suspensibility and dispersibility. The variation in drying temperature shown negligible effect upon the quality of the product.

INTRODUCTION

Conventional Wettable Powder (WP) formulations of pesticides being used in agriculture and public health sectors contain very fine powder and thus have disadvantages like difficulty in dilution, contamination of non target area and inhalation risk to the user. The Water Dispersible Granules (WG) formulations are safe and promising alternative to minimize the problems associated with wettable powder formulations. WG of pesticides contain granules which readily disperse in water to form a suspension (GIFAP, 1989). The WG also known as dry flowables are free flowing, dustless granules of 1-2 mm size which disperse in water to form a sprayable suspension (Ramdas, 1996). These formulations are gaining popularity due to their various advantages like dust freeness, safe handling, easy dispersion, good suspensibility in the spray tank and good bio-efficacy (Bell, 1998).

WG formulations of pesticides are produced by different processes like Pan Granulation, Fluid-bed Spray Granulation and Extrusion (Knowles, 2008). The extrusion process is being widely used due to its economic and industrial feasibility. The preparation of extruded WG involves a three step process. The pesticide is mixed with surfactants and diluents and grinding is done to get a fine powder. Water is then mixed with the powder and extruded. The extruded granules are then dried and sieved to get the final product.

In the literature, sufficient data is not available about the effects of extrusion process variables upon quality of the product. In the present study, the effects of three major extrusion process variables viz. amount of water added into extrusion powder, rotation speed of screw extruder and drying temperature of granules upon the quality of water dispersible granules with respect to major quality parameters i.e., suspensibility and dispersibility have been

investigated. The studies were conducted on WG of insecticide Deltamethrin (IUPAC Name: (S)-alpha-cyano-3-phenoxybenzyl (1R, 3R) -3-(2, 2-dibromovinyl)-2, 2- dimethyl cyclopropane carboxylate). Deltamethrin is a widely used contact and stomach insecticide of synthetic pyrethroid group and is widely used to control mosquitoes, agricultural insects and locust.

2. MATERIALS AND METHODS

2.1 Materials

Deltamethrin (technical grade-98.0% a.i.) was obtained from Tagros Chemicals, (Chennai, India), wetting agent (sodium naphthalene sulphonate) was obtained from Jeevan Chemicals (Mumbai, India), dispersing agent (Potassium polycarboxylate) was obtained from Albright & Wilson Ltd. (Mumbai, India), Lactose monohydrate was obtained from Qualigens (Mumbai, India), China clay (levigated-micronized) was obtained from IAC Minerals (Chennai, India).

2.2 Preparation of Deltamethrin Water Dispersible Granules

Deltamethrin was mixed with inert ingredients as per optimized composition given in Table1. The role of each ingredient in the formulation is given in parenthesis in the table.

The mixture is finely grinded using a laboratory blade grinder to a fine powder of average particle size (mean diameter) of 4.89 microns. The ground powder (20gm) was taken in a 250ml beaker and different requisite quantities of water were added and mixed with a spatula. The wet powder was extruded through screw extruder (Model-Caleva Mini Screw) fitted with screen of 1mm perforations. The extruded granules were dried in the oven to less than 0.5% moisture in the final product of different batches.

2.3 Suspensibility Analysis

Suspensibility (suspension stability) is the percent part of active ingredient suspended in a column of water after a given time. Suspensibility of the WG formulations has significant role in the application of spray suspension. If the suspensibility is upto the mark, spray tank mixing requirement is less and homogeneity is maintained in the suspension. It ensures uniform distribution of active ingredient on the target area, thus risk of less biological control in one part and phyotoxicity in another part is reduced. The suspensibility of the samples was analyzed by method no. MT168 of CIPAC Handbook (CIPAC, 2007)

2.4 Dispersibility Analysis

Dispersibility is the percentage of active ingredient dispersed in water after stirring the suspension. The spray tank mixing requirement is minimum if the WG formulation has good dispersibility. The dispersibility is a major quality parameter of WG formulations. The dispersibility of the samples was analyzed by method no. MT174 of CIPAC Handbook (CIPAC, 2007).

2.5 Particle Size Analysis

The particle size of the samples were analyzed by particle size analyzer (Model- Malvern Master Sizer MS20) using distilled water as dispersant.

3. RESULTS AND DISCUSSION

Table 1: Optimized composition of Deltamethrin Water Dispersible Granules

Ingredient	% w/w
Deltamethrin technical grade a.i. = 98.% (Insecticide)	25.5
Sodium naphthalene sulphonate (Wetting agent)	2.5
Potassium polycarboxylate (Dispersing agent)	8.0
Lactose monohydrate (Diluent)	22.5
China clay(Diluent)	q.s.
Total	100

3.1. Effect of variation in amount of Water added into Extrusion Powder upon quality parameters

Samples of WG were prepared with variations in water quantity added to extrusion powder from 5% to 12.5% (at increments of 2.5%). The other two process variable i.e. extruder rotation speed kept fixed at 70rpm and drying temperature kept fixed at 70^0C (3 hrs). The results are depicted in Table 2.

Table 2 : Effect of variation in amount of Water added into Extrusion Powder upon quality parameters

Amount of water (%)	Suspensibility (%)	Dispersibility (%)	Particle Size (mean diameter-microns)
5.0	85.07	88.32	5.32
7.5	79.78	83.01	10.23
10.0	75.71	79.60	12.85
12.5	74.40	77.34	17.82

The effect of amount of water added into extrusion powder was remarkable upon the suspensibility and dispersibilty of WG. The suspensibility was maximum i.e. 85.07% in the sample prepared with 5% water while suspensibilty was minimum i.e. 74.40% in the sample prepared with 12.5% water. Similarly the dispersibilty was maximum i.e. 88.32% in the sample prepared with 5% water while dispersibilty was minimum i.e. 77.34% in the sample prepared with 12.5% water.

The more amount of water added into extrusion powder might be catalyzing the particle agglomeration due to formation of more hydrogen bonding between the molecules. The particle size analysis results also support the suspensibility and dispersibility results. The particle size of sample having the highest suspensibility and dispersibilty is low i.e., 5.32µm while that of the sample having the lowest suspensibility and dispersibilty is high i.e., 17.82µm. Although all these samples were prepared from the same stock powder of 4.89 micron average particle size. The increase in the particle size may be due to the formation of strong agglomerates during extrusion and maximum in sample prepared with the highest quantity of water i.e. 12.5%. Thus, the minimum amount of water, just sufficient to extrude the powder provides better suspensibility and dispersibility product.

3.2 Effect of variation in Extruder Rotation Speed upon quality parameters

Samples of WG were prepared with variation in extruder speed from 70 rpm to 130 rpm (at increments of 20 rpm),the other two process i.e. drying temperature kept fixed at 70^0C (3 hrs) and amount of water added to extrusion powder kept fixed at 5%. The results are depicted in Table 3

Table 3: Effect of variation in Extruder Rotation Speed upon quality parameters

Extruder rotation Speed (rpm)	Suspensibility (%)	Dispersibility (%)	Particle Size (mean diameter-µm)
70	85.07	88.32	5.32
90	82.83	86.58	8.26
110	82.74	85.59	8.37
130	82.04	85.42	8.75

The suspensibilty was maximum i.e. 85.07% in the sample prepared at 70 rpm rotation speed of the extruder while suspensibilty is minimum i.e. 82.04 % in the sample prepared at 130 rpm rotation speed of the extruder. Similarly the dispersibilty is maximum i.e. 88.32% in the sample prepared at 70 rpm rotation speed of the extruder while dispersibilty is minimum i.e. 85.42% in the sample prepared at 130 rpm rotation speed of the extruder.

The difference of approximately 3% between suspensibility and dispersibility of samples prepared at 70 and 130rpm rotation speed is although not very high but it is considerable. At high speed, the screw brings more amount of powder towards the screen, thus the increase in rotation speed enhances the product yield. But the small size (1mm) perforations on the screen prevent fast passing out of the granules. This results in compaction of the granules, thereby reducing suspensibility and dispersibility.

The particle size analysis results also correspond to the suspensibility and dispersibility results. The particle size of sample having the highest suspensibility and dispersibilty is 5.32µm while that of the sample having the lowest suspensibility and dispersibilty is 8.75µm. The increase in the particle size is due to the agglomerates formed by compaction. The lower extruder speed reduces compaction resulting in better suspensibility and dispersibility product.

3.3 Effect of variation in Drying Temperature upon quality parameters

Samples of WG were prepared with changing the drying temperature between 40^0C to 70^0C (at increments of 10^0C), more drying time is given at lower temperature and less drying time is given at higher temperature and time was

optimized to dry the samples to bring down moisture content less than 0.5% in the final product. The other two process variables i.e. extruder rotation speed kept fixed at 70rpm and water quantity added to extrusion powder kept fixed at 5%. The results of effect of changes in drying temperature upon quality parameters are given in Table 4

Table 4: Effect of variation in Drying Temperature upon quality parameters

Drying Temperature (^{0}C)	Drying Time (Hrs)	Suspensibility (%)	Dispersibility (%)	Particle Size (mean diameter-µm)
70	3	85.07	88.32	5.87
60	4	84.18	89.15	5.62
50	5	85.80	88.92	6.09
40	6	83.80	88.96	5.32

The variation in suspensibility of all the four samples is very less i.e. 1.27 % (85.07% - 83.80%) and in the same way, the variation in dispersibilty is also very less i.e. 0.64 % (88.96% - 88.32%). The variations of drying temperature of WG samples between 40^0C to 70^0C did not make remarkable change in suspensibility and dispersibilty of the products. The variation in particle size of the samples is also very less and it varied between 5.32µm to 6.09µm, this corresponds to suspensibility and dispersibility results. The negligible effect of drying temperature may be attributed to high melting points of inert ingredients and Deltamethrin . Thus for Deltamethrin WG preparation, the drying temperature may be increased upto 70^0C for faster drying without any adverse effect upon suspensibility and dispersibility of the product.

4. CONCLUSIONS

The changes in extrusion process variables shown remarkable effect upon the quality of extruded water dispersible granules of deltamethrin with respect to major quality parameters of suspensibility and dispersibilty. The less amount of water added to extrusion powder resulted in less agglomeration of particles, thereby provided better suspensibility and dispersibility of granules. The low extruder rotation speed reduced compaction resulting in better suspensibility and dispersibility of WG. The effect of variations of drying temperature was negligible upon the product quality.

REFERENCES

Bell, G.P. (1998) Water dispersible granules in Chemistry and technology of agrochemical formulations, edited by A. Knowles, London: Kluwer academic publishers; pp. 80-120

CIPAC Handbook, (2007) Physico-chemical methods for technical and formulated pesticides. Vol. F. Collaborative International Pesticide Analytical Council, Harpenden, U.K.

GIFAP(1989) Catalogue of pesticide formulation types and international coding system. Technical monograph no. 2. International group of national associations of manufacturers of agrochemical products , Brussels

Knowles, A. (2008) Recent developments of safer formulations of agrochemicals. Environmentalist, **28**, pp.35-44

Ramdas, P.K. (1996) Advances in pesticide formulations under Indian context in Agrochemicals for sustainable agriculture, edited by N. K Roy, New Delhi: APC Publications pp. 23-28