

ORIGINAL ARTICLE

Shelf life Studies on Bifenthrin 10 EC using GC-FID

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ABSTRACT

Modern pesticide formulation technology aims to prolong shelf-life as much as possible so as to optimize the biological activity of the pesticide and to give a product which is safe and convenient for use. However, Pesticides in sealed containers may change over time if the active ingredient changes chemically or the formulation of the pesticide breaks down. Therefore, pesticide store should be inspected regularly for signs of deterioration. Since quality and performance is the major constraint faced by the users, commercially available and widely used emulsifiable concentrate (EC) formulations of bifenthrin (10 per cent) was assessed with (GC-FID) for technical grade content in storage on the shelf in farm store room for the period of 30 months. The per cent active ingredient (a.i.) for bifenthrin from 0 month to 30 months were found in the range between 10.17 and 10.46 per cent (Permissible tolerance limit as per BIS Specification is 9.50 to 10.50). Keeping in view the expiry period of 2 years, the tested formulation sample was found stable and unaffected during the storage.

Keywords: Bifenthrin, GC-FID, EC formulation, BIS specifications.

1. INTRODUCTION

Pesticides are used globally and extensively for the control of various kinds of pests that cause harm to crops and livestock yields and reduces the farm productivity [1]. Several pesticidal formulations have been developed over the years with an aim to prolong shelf-life as much as possible. Most pesticides have an indicated shelf-life of at least two years from the time of manufacture, but Pesticides in sealed containers may change over time if the active ingredient changes chemically or the formulation of the pesticide breaks down. Therefore, it becomes necessary to take into account the time that pesticides may have been in transit between manufacture and

reaching the store. Bureau of Indian Standard specifications (BIS) is developed with the basic objective to ensure the percentage purity of active ingredient in both technical grades and formulations as per the labelled claim by the manufacturers, the suitability of the pesticide formulation for its intended use, safe and judicious use of pesticides and the shelf life of active ingredient so that consumers are not cheated into buying sub-standard, spurious products which have now flooded the market.

Pyrethroid insecticides, synthetic derivatives of pyrethrins, are used as wide-spectrum insecticides due to their high insecticidal potency, slow development of insect resistance, relatively low acute toxicity in mammals, not being persistent in the environment [2]. Bifenthrin is an insecticide in the pyrethroid family [3]. Pyrethroids are manmade versions of pyrethrins, which come from chrysanthemum flowers. Bifenthrin is broad spectrum pyrethroid insecticide. Products containing bifenthrin are used on cereals, cotton, corn, alfalfa, hay, grass seed, some fruits, ornamentals and vegetables [4]. Bifenthrin is a stomach and contact insecticide [5]. Bifenthrin is available as 2.5%EC, 10%EC, 10%FS, 20%FS, 10%SC, 40%SC in market. Bifenthrin is highly photo stable and has a low solubility and a correspondingly strong tendency to bind to soil. With this background, commercially available and widely used formulations of Bifenthrin 10% EC was assessed for technical grade content in storage on the shelf in farm store room for the period of 30 months. The results of the present studies will be further utilized for selection and also for its application.

2. MATERIAL AND METHODS

The expiry period of emulsifiable concentrate of bifenthrin is of two years from the date of manufacturing. In this study technical grade content of the formulation was studied periodically for the span of 30 months by keeping the interval of 6 months from the first sampling. The samples were tested at Zero (initial), 6, 12, 18, 24 and 30 months from the date of storage on shelf at ambient conditions of farm store room. Bifenthrin 10EC manufactured in June 2016 (100ml container) was used for the studies by starting the first sample analysis in first week of July 2016 which was considered as zero month sample. The last sample analysis was performed in second

week of January 2019. In each formulated compound, 20 containers were maintained on the rack in the store room at farm conditions of Nagpur during July 2016 to January 2019. At each sampling three tin containers were taken out and studied for technical grade content. The analytical methods adopted for studying the technical grade content is outlined below.

Active ingredient test: Bifenthrin 10%, BIS [6]

Material

For Bifenthrin 10% EC Gas chromatograph (GLC) equipped with FID with facilities for on column injection and coupled to a printer-plotter-cum-integrator is used for this determination

Method

Working condition for analysis:

Column	
Column Dimensions	Stainless steel, 2 m × 3 mm
Stationary phase	10 percent OV-101
Solid support	Chromosorb WHP 80 /100 mesh
Detector system	
Type	FID (Flame ionization detector)
<i>Temperature</i>	
Column oven	250° C
Injector	275° C
Detector	300° C
Carrier Gas	Nitrogen-30 ml/min Hydrogen-40ml/min Air-450 ml/lit
Retention time	Bifenthrin – 10.1 min Docosane – 5.3 min
Injection Volume	1 µl.
Microlitre Syringe	5/10 µl capacity
Reagent	
Acetone	AR grade or equivalent
Internal Standard	Docosane AR grade or equivalent
Bifenthrin Reference Standard	Of known purity

Procedure:**Preparation of Internal Standard Solution**

Weigh accurately 1.0 g of docosane into a 100 ml volumetric flask. Dissolve the solids completely in acetone and make up the volume up to the mark with acetone. Shake well to homogenize.

Preparation of Analytical Standard Solution

Weigh accurately 0.1 g of bifenthrin reference standard of known purity into a 50 ml volumetric flask. Dissolve in 10 ml of acetone. Add 10 ml of the internal standard solution and make up the volume up to the mark with acetone. Shake well to homogenize.

Preparation of Sample Solution

Weigh accurate quantity of a sample equivalent to about 0.1 g of bifenthrin sample into a 50 ml volumetric flask. Dissolve in 10 ml of acetone. Add 10 ml of the internal standard solution and make up the volume up to the mark with acetone. Shake well to homogenize.

Estimation

Inject 1 µl standard solution until the internal standard/reference substance area quotations of two successive chromatograms do not deviate from each other by more than 2 percent. Then use the following injection sequence:

C S1 S1, C S2 S2, C S3.....

where

C = standard solution; and

S = sample solution (1, 2, n).

Observations

From the chromatograms of the standard solution and sample solution, measure the peak areas of the internal standard and bifenthrin peaks, and compute the percentage of the bifenthrin as given below

Calculations

$$\text{Bifenthrin content, percent by mass} = \frac{M_1 \times A_1 \times A_3 \times P}{M_2 \times A_2 \times A_4}$$

where

M1 = mass of standard bifenthrin in standard solution, in g;

M2 = mass of sample taken for test in g;

A1 = peak area of bifenthrin in the chromatogram of standard solution;

A2 = peak area of bifenthrin in the chromatogram of sample solution;

A3 = peak area of internal standard in the chromatogram of standard solution;

A4 = peak area of internal standard in the chromatogram of in sample solution; and

P = percent purity of bifenthrin reference standard.

3. RESULTS AND CONCLUSIONS

The data on technical grade content in periodically drawn containers of bifenthrin 10 EC is given in table 1. This was the mean per cent of a.i. present in three samples tested at a time. On the first day of storage the technical grade content (a.i.) was 10.17 per cent. In subsequent sampling of 6, 12, 18, 24 and 30 months contents were, 10.85, 10.97, 11.01, 11.19 and 10.46 per cent respectively. Percent tolerance limits as specified by BIS (9.50 to 10.50).

Table 1: Storage shelf life of EC formulation of Bifenthrin10 EC

Sampling (Months after storage)	Recovered contents (%)			
	R I	R II	R III	Mean (± S.D.)
0(initial)*	10.48	10.44	10.46	10.46 (± 0.016)
6	10.43	10.41	10.44	10.43 (± 0.012)
12	10.36	10.42	10.38	10.39(± 0.025)
18	10.29	10.33	10.30	10.31(± 0.017)
24	10.25	10.28	10.25	10.26 (± 0.014)
30	10.16	10.18	10.15	10.17(± 0.012)
* Date of initial sampling : 14-06-2016				

The estimated contents of bifenthrin in the formulation were more or nearly equal to the standard requirement of 10 % as claimed on the label of the container. Hence an expiry period of 2 years was found most appropriate. The formulation of bifenthrin 10 EC can therefore, be considered suitable for spraying work within the period of expiry. Similar studies on pesticides with different formulations is carried by [7-9].

In storage shelf life studies of bifenthrin 10 EC, the sample remain unaffected for the technical grade content and emulsion stability over the period of 30 months. The per cent active ingredient (a.i.) for bifenthrin from 0 month to 30 months were found in the range between 10.17 and 10.46 per cent (Permissible tolerance limit as per BIS Specification is 9.50 to 10.50). The maximum temperature of farm store room during summer season was 45.3°C. Similarly the maximum relative humidity during rainy season was 93 per cent. The higher temperature and relative humidity are responsible for the deterioration of the pesticides in the stored containers. In the present studies, the above stated situation could not show any adverse effect on the EC formulation. Hence the expiry period of 2 years specified by the manufacturers hold good for the formulation.

Conflict of interest

No conflict of interest influenced in this research.

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