Original Article

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF MALATHION BY UV SPECTROSCOPY

R S Chandan[1], M Umesh[2*], N R Soundaryashree[3], Aparajita Neogi[4], Aparna AS[5], Eldo jacob[6], Divya scaria[7], Gana N P[8]
[1,2,3,4,5,6,7,8]Department of pharmaceutical analysis, JSS College of pharmacy, JSS University, Mysore-570015(KA), India.
Email: mumesh@jssuni.edu.in

Abstract

The aim of this study was to develop and validate a simple, accurate, cost-effective spectrophotometric method for determination of malathion pesticide. The developed method was validated in terms of accuracy, inter and intraday precision, linearity, limit of detection and limit of quantification as per ICH guidelines. Malathion was determined at 240nm in methanol by UV spectrophotometric method. The beers law obeyed in the range of 1-5 µg/mL (r2 =0.9995). The accuracy was found between 98-105%. The precision for interday and intraday results was found to be within the limits. Hence the method can be used for determination of malathion pesticide in water, vegetable samples.

Keywords: Malathion, Pesticides, UV spectroscopy.

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INTRODUCTION

Malathion (Diethyl 2-[(dimethoxyphosphorothioyl) sulfanyl] butanedioate) is one of the most commonly used organophosphate pesticides worldwide. It is used to control pests that infest agricultural crops, ornamentals, greenhouses, livestock, stored grain, forests, buildings, residences, and gardens. Contributing to its popularity is its relatively low acute mammalian toxicity. Malathion was the third most commonly reported pesticide in California, producing five times more occupational illness than the average pesticide [1]. Malathion itself is of low toxicity; however, absorption or ingestion into the human body readily results in its metabolism to malaoxon, which is substantially more toxic. Acute exposure to very high amounts of malathion will result in systemic symptoms whose severity will depend on the intensity of the exposure [2].

The literature survey reveals that only a few publications are related to the analysis of active substances in the pesticide formulation and their quality control. However, till date, only few analytical methods were reported for the estimation of malathion in pesticide formulations by using UV spectrophotometer. As a result, the objective of this research was to develop a novel, simple, rapid, and precise method for determining malathion in pesticide formulations. The proposed method has been applied successfully to determine malathion levels in water and vegetables.

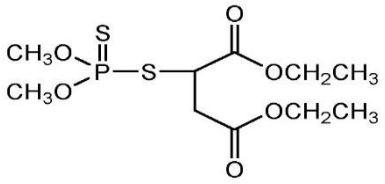

Figure 1: Structure of malathion

EXPERIMENTAL DETAILS

Materials

Malathion raw material was procured from sigma Aldrich, Bangalore, India. Formulation was purchased from local pharmacy. Methanol was procured from Merck life science Pvt Ld.

Instrumentation

Shimadzu 1700 UV-Visible spectrophotometer were used for absorption measurements and digital weighing balance (SHIMADZU AY220) were used for weighing. The drug and sample solution were sonicated using an ultra sonicator (Antech ultra sonicator).

Preparation of standard

Accurately weigh 10 mg of pure malathion in to 100 ml volumetric flask, dissolve and makeup the volume by using methanol and mark it as stock solution A (100 µg/ml). Pipette out 1 ml from the above stock solution A and transfer to 10 ml volumetric flask and make up the volume with methanol and shake well and consider this as B (10 µg/ml). From the above stock solution B, the standard stock solution of 1,2,3.4,5 ml was pipetted out and diluted with methanol to get the concentration from 1-5 µg/ml. The solution was sonicated then filtered by using 0.22µ membrane filter then absorbance was measured at 240nm.

Preparation of sample

A commercial formulation of Malathion (Malathion 50 EC) was prepared by serial dilution to get 10 µg/ml. The solution was sonicated then filtered by using 0.22µ membrane filter then absorbance was measured at 240nm.

RESULTS

Linearity

The linearity of the present method was evaluated by different concentration of insecticide under optimized conditions. For Malathion, the regression analysis data revealed a clear linear association over a calibration range of 1-5µg/ml. The equation for linear regression was observed to be Y=0.1962x +0.01.

Accuracy

Outcome of accuracy studies are listed in Table 2, which demonstrated that the percentage amount found was between 99.24 percent and 100.13 percent with % Relative standard deviation less than 2. The solutions were evaluated by the proposed process.

Precision

With the current relative standard deviation (RSD percentage) below 2.0 percent, the precision (intraday, interday, repeatability measurement) results showed excellent reproducibility. This revealed an incredibly precise system, Results of precision studies are listed in Table 3 and 4.

LOD and LOQ

The equation of linearity was established to be Y = 0.1962x + 0.01

The Limit of Detection and Limit of Quantitation were found to be 0.123µg and 0.4107µg/ml for Malathion

Repeatability

Repeatability was calculated by the six-fold study of 3µg/ml Malathion solution concentration and the percentage found was between 98 percent and 102 percent with percent RSD < 2 percent.

Robustness

The robustness of the method was calculated by measuring the absorbance on changing the wavelength range from 245-255nm. The results to be depicted in table 7.

Ruggedness

For the different concentration solutions, the absorbance was determined. For both of the products, the findings are in the appropriate range. Table 8 provides the results. The outcome showed that the RSD percentage was less than 2 percent.

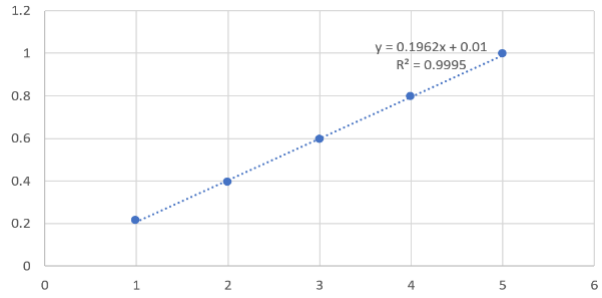

Figure 2: Linearity graph of Malathion

Table 1: Calibration Data of Malathion

SL. No	Conc. (µg/ml)	Abs Of Malathion
1	1	0.215
2	2	0.314
3	3	0.584
4	4	0.794
5	5	0.856

Regression EQN	0.1962x+0.01
Correlation Coefficient (R^2)	0.9995

Slope	0.1962
Intercept	0.01

Table 2: Accuracy Results for Malathion by UV Spectroscopy

Recovery levels	Amount of formulation	Amount of Pure drug	Total amount of drug	Abs	Difference	%Recovery	Mean Recovery
50%	10	2	12	0.378	0.064	96.96	98.48
	10	2	12	0.379	0.065	98.48	
	10	2	12	0.38	0.066	100	
100%	10	3	13	0.653	0.069	104	105.6
	10	3	13	0.654	0.070	106	
	10	3	13	0.655	0.071	107	
150%	10	4	14	0.862	0.068	103	99.48
	10	4	14	0.859	0.065	98.48	
	10	4	14	0.858	0.064	96.96	

Table 3: Results for Intraday Precision of Malathion by UV Spectroscopy

precision	Acceptance criteria	Concentration (µg/ml)			Mean Absorbance			%RSD		
Intraday morning	%RSD ≤2.0%	2	3	4	0.314	0.584	0.794	1.749	1.341	1.578
Intraday evening	%RSD≤2.0%	2	3	4	0.321	0.590	0.812	1.722	1.312	1.561

Table 4: Results for Interday Precision of Malathion by UV Spectroscopy

precision	Acceptance criteria	Concentration (µg/ml)			Mean Absorbance			%RSD		
Interday morning	%RSD ≤2.0%	2	3	4	0.301	0.551	0.698	1.699	1.299	1.509
Interday evening	%RSD ≤2.0%	2	3	4	0.312	0.563	0.721	1.733	1.299	1.506

Table 5: Results for LOD & LOQ of Malathion by UV Spectroscopy

LOD (µg/ml)	0.1232
LOQ (µg/ml)	0.4107

Table 6: Results for Repeatability of Malathion by UV Spectroscopy

Sl. No	**Concentration (µg/ml)**	**Absorbance**	**STDV**	**% RSD**
1	3	0.562	0.0077	1.3419
2	3	0.568		
3	3	0.586		
4	3	0.572		
5	3	0.582		
	Mean	0.574		

Table 7: Results for Robustness of Malathion by UV Spectroscopy

Sl. No	**Wavelength (nm)**	**Absorbance**	**STDV**	**% RSD**
1	245	0.584	0.0082	1.4488
2	246	0.580		
3	247	0.577		
4	248	0.573		
5	249	0.570		
6	250	0.568		
7	251	0.564		
8	252	0.562		
9	253	0.560		
10	254	0.559		
	Mean	0.5124		

Table 8: Results for Ruggedness of Malathion by UV Spectroscopy

	Conc. (µg/ml)	**Trail 1**	**Trail 2**	**Mean**	**S. D**	**%RSD**
By changing the analyst	1	0.217	0.222	0.220	0.002	1.138
	2	0.413	0.401	0.407	0.003	0.731
	3	0.563	0.582	0.572	0.009	1.659
	4	0.701	0.711	0.706	0.005	0.708
	5	0.872	0.855	0.863	0.008	0.984
	1	0.210	0.212	0.211	0.001	0.473

By changing the instrument	2	0.398	0.384	0.391	0.007	1.790
	3	0.578	0.591	0.584	0.006	1.112
	4	0.689	0.674	0.681	0.007	1.100
	5	0.831	0.856	0.843	0.012	1.481

Conclusion

Although some techniques have been documented with UV-Visible spectrophotometric techniques for the production and validation of Malathion, efforts have been made to produce easy and reliable techniques for validation of Malathion. The validation process confirms that this is a suitable approach in the formulation for their quantification in QC labs.

REFERENCES

1. Brenner, L. Malathion. Journal of Pesticide Reform. 1992;12(4), 29-37.
2. Venugopal, N. V. S., Sumalatha, B., & Bonthula, S. Spectrophotometric determination of Malathion (an organo phosphorous insecticide) with Potassium bromate. Eurasian Journal of Analytical Chemistry. 2013; 8, 131-135.
3. Albuquerque, C. D., & Poppi, R. J. Detection of malathion in food peels by surface-enhanced Raman imaging spectroscopy and multivariate curve resolution. Analytica Chimica Acta. 2015; 879, 24-33.
4. Pandey, G. P., Singh, A. K., Deshmukh, L., Prasad, S., Paliwal, L. J., Asthana, A., & Mathew, S. B. A novel and sensitive kinetic method for the determination of malathion using chromogenic reagent. Microchemical Journal. 2014; 113, 83-89.
5. Velkoska-Markovska, L., & Petanovska-Ilievska, B. Rapid resolution liquid chromatography method for determination of malathion in pesticide formulation. Acta Chromatographica. 2020; 32(4), 256-259.
6. Musarurwa, H., Chimuka, L., & Tavengwa, N. T. Z-sep+ based QuEChERS technique for the pre-concentration of malathion pesticide in fruits followed by analysis using UV-Vis spectroscopy. Food Additives & Contaminants: Part A. 2020; 37(12), 2093-2108.
7. Gouda, A. A., Amin, A. S., El-Sheikh, R., & Akl, M. A. Sensitive spectrophotometric methods for determination of some organophosphorus pesticides in vegetable samples. Chemical Industry and Chemical Engineering Quarterly/CICEQ. 2010; 16(1), 11-18.
8. Sharma, D. K., Thakur, N., Sharma, A., & Raj, P. New Spectrophotometric Method for the Analysis of Commercial Malathion Formulation and Its Residues on Some Crop Produces and Environmental Samples. Journal of Advanced Scientific Research. 2020; 11(02), 131-136.
9. Rao, P. P., Seshaiah, K., & Naidu, P. R. (1983). Determination of malathion in formulations. Journal of Agricultural and Food Chemistry. 1983; 31(6), 1370-1371.
10. Pharmacopoeia, I. The Indian pharmacopoeia commission. Central Indian Pharmacopoeia Laboratory, Ministry of Health and Family Welfare, Govt of India, Sector, 23. 2007.