https://ijrps.com

ISSN: 0975-7538
Research Article

Analytical method for the determination of pesticides in soil by thin layer chromatography

B. Karthik kumar*[1], V.S. Thiruvengada rajan[1], N. Amruth kumar[1], S. Angala parameswari[1], C. Madhusudhana Chetty[2]

[1] Department of Pharmaceutical Analysis and Quality Assurance, Annamacharya College of Pharmacy, Rajampet - 516126, Andhra Pradesh, India

[2] Department of Bio-Technology, Annamacharya College of Pharmacy, Rajampet - 516126, Andhra Pradesh, India

ABSTRACT

The term pesticide as used throughout this report refers to chemical substances (i.e. conventional pesticides) that are biologically active and interfere with the normal biological processes of living organisms deemed to be pests, whether these are noxious plants or weeds, insects, mould or fungi. Monocrotophos is an organophosphorus insecticide with systemic activity and is used for the control of a wide range of chewing, sucking and boring pests. Chloropyrofus is a crystalline organophosphate insecticide that inhibits acetyl cholinesterase and is used to control insect pests. Acephate is an organophosphate foliar insecticide, is used primarily for control of aphids. Accurately weighed 10g of spiked soil and transfer in to 50ml volumetric flask and add 20ml of methanol then kept in ultrasonicator for 20min for efficient extraction. The above extracts were filtered through whatman filter paper. The filtrates were evaporated on a heating mantle at 40°C to dryness. The residue was dissolved in 1ml of Methanol. The type of pesticide present in the soil sample was determined TLC method by using Methanol–water (8:2, v/v) as a mobile phase. The R_f value of test sample (1) and standard sample (1) Monocrotophos are 0.92 and 0.88 respectively, the spots are identified under the short U.V light and the spot colour is blue in colour. Other test samples did not produce any spots, it indicates the absence of Chloropyrofus, Quinolphos and Acephate in the soil sample.

Keywords: Acephate; Chloropyrofus; Extraction; Monocrotophos; Quinolphos; Thin-layer chromatography; soil

INTRODUCTION

Pesticides are substances that help protect plants against molds, fungi, rodents, and insects. TLC is very widely used in a variety of pesticide studies, such as determination of quantitative structure activity relations (QSAR) that describe how the molecular structure, in terms of descriptors (lipophilic, electronic, steric), affects the biological activity of a compound. The term pesticide as used throughout this report refers to chemical substances (i.e. conventional pesticides) that are biologically active and interfere with the normal biological processes of living organisms deemed to be pests, whether these are noxious plants or weeds, insects, mould or fungi. The biological activity of pesticides is achieved by different modes of action. Pesticides help prevent crop loss and, potentially, human disease. According to the Environmental Protection Agency, there are currently more than 865 registered pesticides. Man-made pesticides are regulated by the U.S. Department of Agriculture. Monocrotophos is an organophosphorus insecticide with systemic activity and is used for the control of a wide range of chewing, sucking and boring pests (aphids, caterpillars, helicoverpa spp, mites, jassids, budworm, scale and stem borer) as well as locusts. Use is for cotton, beans (French), sunflowers, maize, millet, sorghum, soybeans, sweet corn, wheat, tobacco and for trees. Chloropyrofus is a crystalline organophosphate insecticide that inhibits acetyl cholinesterase and is used to control insect pests. Acephate is an organophosphate foliar insecticide, is used primarily for control of aphids, including resistant species, in vegetables (e.g. potatoes, carrots, greenhouse tomatoes, and lettuce) and in horticulture (e.g. on roses and greenhouse ornamentals). The method of TLC mainly based on the “adsorption phenomenon”. It is mainly used for the separation and identification of pesticide traces present in the soil both qualitatively and quantitatively. (Muller et.al., 2000) (Wasterman et.al., 1990) (Sandra Babic et.al., 1998) (Walsh et.al., 1973)

* Corresponding Author
Email: karthik.pharma49@gmail.com
Contact: +91-99985842949
Received on: 02-07-2011
Revised on: 05-09-2011
Accepted on: 10-10-2011

MATERIALS AND METHODS

Soil samples, Standard pesticide samples, Volumetric flasks, Beakers, Pipette, Sonicator, U.V scanner, Hand-

made TLC plates 7×2cm, Digital weighing balance, Methanol.

Experimental Work

The fresh sand sample (500 g) was collected from departmental medicinal garden and removes all the living material such as mosses, roots. Then the sample were underwent air drying of temperature of 40°C, until the loss in mass of the sample is not greater than 5 % (m/m) per 24h. The sample materials for storage should be kept without preservative under normal room conditions with minimal temperature and humidity fluctuations, shielded from incident light. (Karr CJ et.al., 2007) (Kellogg RL et.al., 2000)

Stock Solution

Stock solution of pesticide mixture was prepared by dissolving accurate amounts of powdered Acephate (N.N.Greenwood et.al., 1997) 0.01mg/ml of water and Monocrotophus (Bridget Stutchbury, 2008), Chloropyrofus (A.F.wells, 1984) and Quinolphos samples are taken as liquid standards.

Preparation of spiked soil samples

Spiked soil samples were prepared by adding 1 ml of standard mixture of pesticides to 10 g of soil. Additional methanol was added until the solvent completely covered the soil particles. The suspension was thoroughly mixed for 1 hour with shaking. The bulk of the solvent was slowly evaporated at room temperature. (Sandra Babic et.al., 1998)

Accurately weighed 10g of spiked soil and transfer in to 50ml volumetric flask and add 20ml of methanol then kept in ultrasonicator for 20min for efficient extraction. The above extracts were filtered through whatman filter paper. The filtrates were evaporated on a heating mantle at 40°C to dryness. The residue was dissolved in 1ml of Methanol. The type of pesticide present in the soil sample was determined TLC method. This sample preparation was done for every individual standard sample separately. (D.A.J. Weeds et.al., 1995)

Thin-Layer Chromatography

Most pesticide determinations are performed on silica gel TLC. The layers almost always contain a gypsum binder (designated as G layers). Layers often contain a fluorescent indicator or phosphor to facilitate detection of compounds that absorb 254 nm UV light as dark zones on a fluorescent background. TLC was performed on 7×2 cm handmade plates with a layer thickness of 0.25 mm. Aliquots (10 µl) of standard pesticide solution, of the soil extracts and of a blind extract (from nonspiked soil) were applied 10 mm from the lower edge of the plate as a spot. Mobile phases for the TLC of pesticides are less polar than the layer and are composed of aqueous–organic solvent mixtures or fully organic mixtures. Methanol–water (8:2, v/v) was used as a mobile phase. Capillary flow development with the mobile phase in a covered glass chamber. The plates were developed by ascending technique with previous chamber saturation at room temperature. After development, the plates were air dried. Spots were detected under 254 nm UV light. Many pesticides absorb short-wave UV light by inspection in a viewing box under illumination from a 254 nm lamp. (Sandra Babic et.al., 1998) (H.S.Rathore et.al., 1993) (J.sharma et.al., 1994). Pesticide identification is initially based on comparing R_f values compared with standard Fig.1

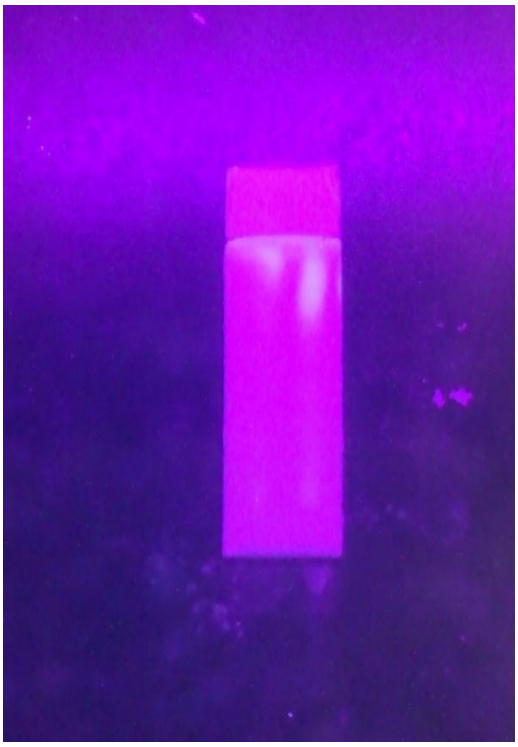

Figure 1: TLC plate under UV light

RESULTS

The TLC parameters for each sample are

Sample 1: Solvent front = 5.1cm

Distance travelled by the test sample1 = 4.7cm

Distance travelled by the standard sample 1 = 4.5cm (Monocrotophos)

Sample 2: Solvent front = 4.9cm

Distance travelled by the test sample 2 = no spot

Distance travelled by the standard sample 2 = 4.1cm (Chloropyrofus)

Sample3: Solvent front = 4.9cm

Distance travelled by the test sample 3 = no spot

Distance travelled by the standard sample 3 = 4.8cm (Quinolphos)

Sample4: Solvent front = 5cm

Distance travelled by the test sample 4 = no spot

Distance travelled by the standard sample 4 = 4.6cm (Acephate)

Table 1: Results from the TLC plates of the method

Samples	Distance travelled by sample (cm)	Distance travelled by standard (cm)	Distance travelled by solvent front (cm)	R_f Values for sample	R_f Values for standard
Monocrotophos	4.7	4.5	5.1	0.92	0.88
Chloropyrofus	-	4.1	4.9	-	0.83
Quinolphos	-	4.8	4.9	-	0.97
Acephate	-	4.6	5.0	-	0.92

DISCUSSION

The R_f value of test sample (1) and standard sample (1) Monocrotophos are 0.92 and 0.88 respectively, the spots are identified under the short UV light and the spot is blue in colour Table 1. Other test samples did not produce any spots, it indicates the absence of Chloropyrofus, Quinolphos and Acephate in the soil sample. This method is a useful tool to analyze the pesticides limit for better constituent yields.

CONCLUSION

From the above data we can conclude that the soil sample contain Monocrotophos pesticide sample traces and other samples like Chloropyrofus, Quinolphos and Acephate samples were absent. Monocrotophos pesticides are the initial or very low concentration pesticide mainly used for flowering plants and medicinal plants. Presence of Monocrotophos in the plant field shows the better yield of plant constituents and production.

REFERENCES

Bridget Stutchbury, New York Times March 30, 2008,Monocrotophos,Wikipedia

D.A.J. Weed, R.S. Kanwar, D.E. Stoltenberg and R.L. Pfeiffer. J. Environ. Qual. 24 (1995), pp. 68–79.

F. Wells, 'Structural Inorganic Chemistry, 5th ed., Oxford University Press, Oxford, UK, 1984.

H.S. Rathore and T. Begun. J. Chromatogr. 643 (1993), pp. 271–290.

J. Sherma. J. Planar Chromatogr., 7 (1994), pp. 265–272.

Karr CJ, Solomon GM, Brock-Utne AC. Health Effects of Common Home, Lawn, and Garden Pesticides. PediatrClin North Am. 2007;54(1).

Kellogg RL, Nehring R, Grube A, Goss DW, and Plotkin S (February 2000), Environmental indicators of pesticide leaching and runoff from farm fields. United States Department of Agriculture Natural Resources Conservation Service. Retrieved on 2007-10-03.

Muller, Franz, ed (2000). Agrochemicals: Composition, Production, Toxicology, Applications. Toronto: Wiley-VCH. p. 541. ISBN 3-527-29852-5.

N. N. Greenwood, A. Earnshaw, Chemistry of the Elements, 2nd ed., Butterworth-Heinemann, Oxford, UK, 1997.

S.K. Poole, T.A. Dean, J.W. Oudsema and C.F. Poole. Anal. Chim. Acta 236 (1990), pp. 3–42.

Sandra Babic, Mira Petrovic and Marija Kas telan-Macan, Volume 823, Issues 1-2, Journal of Chromatography A, October 1998, pages 3-9.

Walsh, L.M., and J.D.Beaton. 1973.soil testing and plant analysis. Soil sco.soc. Am., Madison. WI, USA.

Wasterman, R.L. (ed.) 1990. Soil testing and plant analysis. 3rd ed. Soil.sci.soc. Am. Madison. WI, USA.