Investigation of the determination of allethrin isomers in mosquito coils by using different sources of reference standard

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Introduction

Mosquito is an epidemiologically important vector of Dengue hemorrhagic fever and Malaria. Several of protect to mosquito as hang up a mosquito net, cover clothes, use mosquito coil and use insecticide repellant. The mosquito is currently used in normalous households in Asia, African and South America. The major active ingredients of the mosquito coils in Thailand are allethrin isomers that is performed as 4 forms; d-allethrin, esbiothrin, s-bioallethrin and bioallethrin but only d-allethrin and esbiothrin is form used in mosquito coils. They are classified as hazardous substance type 3 according to Hazardous Substances Act, B.E.2535 and (3rd amendment), BE. 2551. Therefore the products have to be registered and permitted by the Thai FDA before produced or imported. In addition, the labeling should be perform of active ingredients and their concentration within the criteria as state in the notification of the Ministry of Public Health. At the present, the Bureau of Cosmetics and Hazardous substances provides the service of determination the allethrin isomers content by GC-FID. However, the selection of reference standard is one of important step and critical factor to get the accuracy of results of the determination. The purpose of this study is to investigate of the determination of allethrin isomers in mosquito coils by using different sources of reference standard. The test method are identification by HPLC and determination by GC.

Materials and Method

Standard

| Name | source | % purity | Cas no. |
|-------------------------------------|----------------|----------|-------------|
| D-allethrin | Manufacturer 1 | 93.00 | 584-79-2 |
| D-allethrin | Manufacturer 2 | 94.88 | 584-79-2 |
| d,d-trans allethrin (esbiothrin) | Manufacturer 3 | 97.10 | 260359-57-7 |
| esbiothrin | Manufacturer 4 | 97.50 | 84030-86-4 |

•Sample : Mosquito coils with d-allethrin (AI) = 6 samples (sample 1 − 6)

Mosquito coils with esbiothrin (AI) = $\frac{2}{2}$ samples (sample 7 – 8)

•Sample preparation: Extraction by soxhlet apparatus 4 hours and dilute with acetone to meet the calibration curve (0.05 - 0.8 mg/mL)

•Method description

| GC-FID condition | In-house method | |
|------------------|---|--|
| Column | HP-5 (J & W); Length 30 m x 0.32 mm i.d. x 0.25 um film thickness | |
| Detection system | FID | |
| Injection port | 290 °C | |
| Oven | 270 °C | |
| Detector | 300 °C | |
| Carrier gas | Helium | |
| Injection | 1 uL | |

| HPLC condition | CIPAC method | |
|--------------------|--|--|
| column | Two columns joined, each stainless steel, 250 mm x 4 mm (i.d.), packed with Sumicriral OA-2000I, 5 um n-hexane + ethanol, 1000 + 1 (v/v) | |
| Mobile phase | | |
| Flow rate | 1.0 mL/min | |
| Column temperature | ambient | |
| Detector | UV detector, 230 nm | |
| Injection volume | 2 uL | |

References

- WHO SPECIFICATION AND EVALUATIONS FOR PUBLIC HEALTH PESTICIDE. d-allethrin. World Health Organization; 2002

- WHO SPECIFICATION AND EVALUATIONS FOR PUBLIC HEALTH PESTICIDE. esbiothrin. World Health Organization; 2004

- CIPAC Handbook L, Collaborative International Pesticides analytical Council, 2006

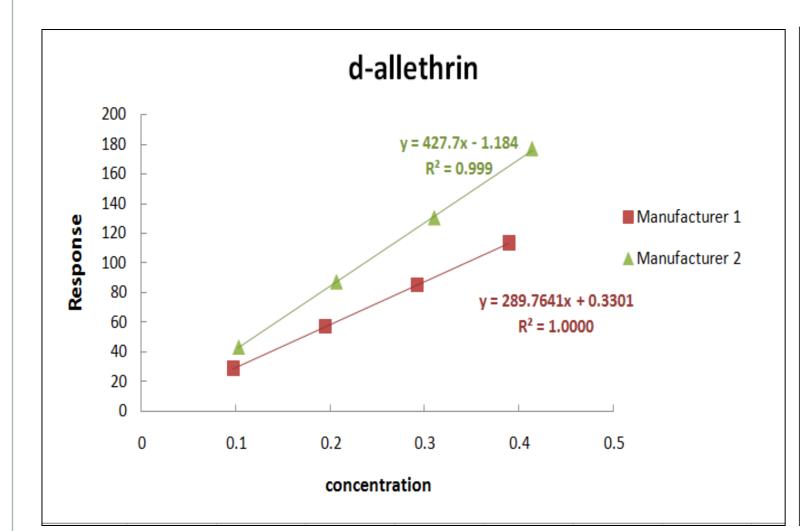
Results

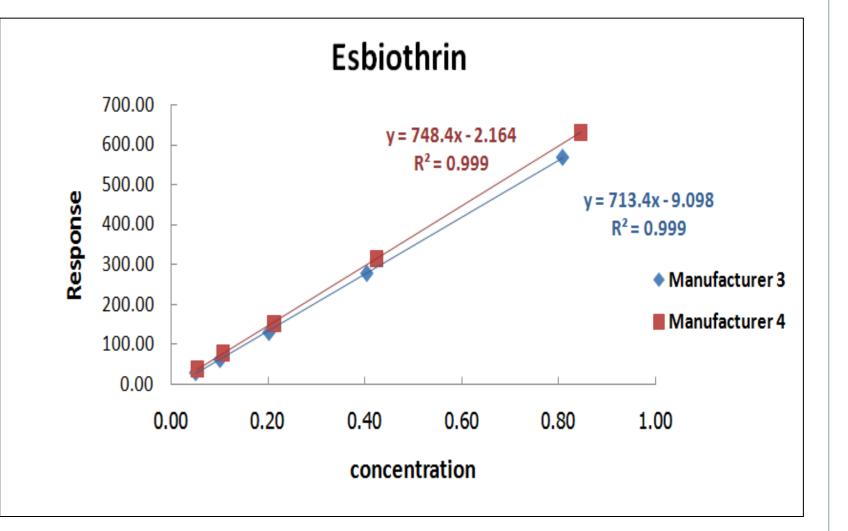
% content of d-allethrin and esbiothrin in mosquito coils samples by GC-FID determination

| Sample | % label (w/w) | % found of <u>d-allethrin</u> (w/w) | |
|----------|---------------|-------------------------------------|---------------------|
| | | Std. manufacturer 1 | Std. manufacturer 2 |
| Sample 1 | 0.3 | 0.633 | 0.342 |
| Sample 2 | 0.3 | 0.594 | 0.345 |
| Sample 3 | 0.3 | 0.658 | 0.354 |
| Sample 4 | 0.3 | 0.657 | 0.349 |
| Sample 5 | 0.3 | 0.526 | 0.306 |
| Sample 6 | 0.3 | 0.426 | 0.268 |

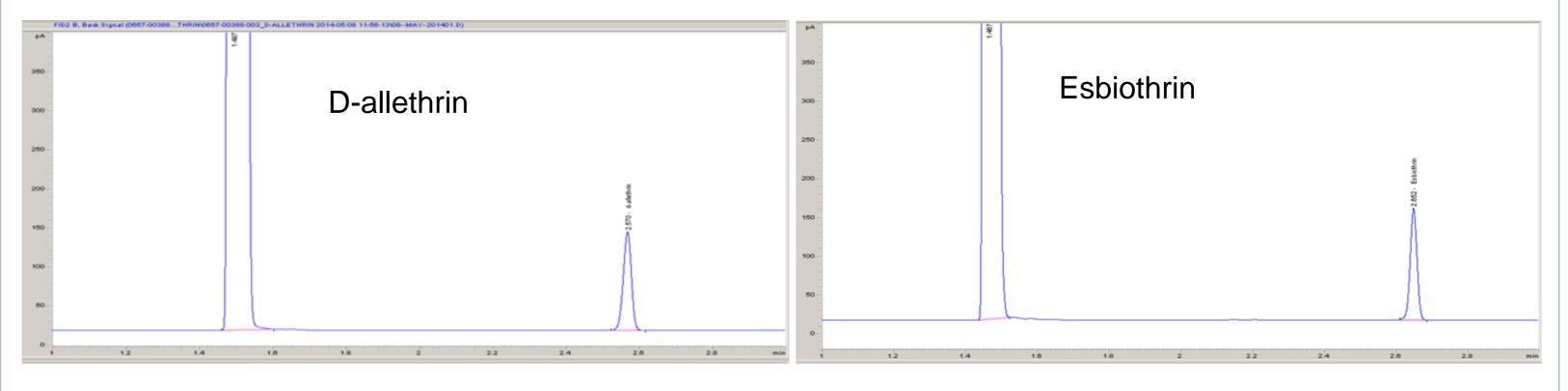
| sample | % label (w/w) | % found of <u>esbiothrin</u> (%w/w) | |
|----------|---------------|-------------------------------------|--------------------|
| | | Std.manufacturer 3 | Std.manufacturer 4 |
| Sample 7 | 0.15 | o.164 | 0.170 |
| Sample 8 | 0.15 | 0.155 | 0.155 |

Calibration Curve

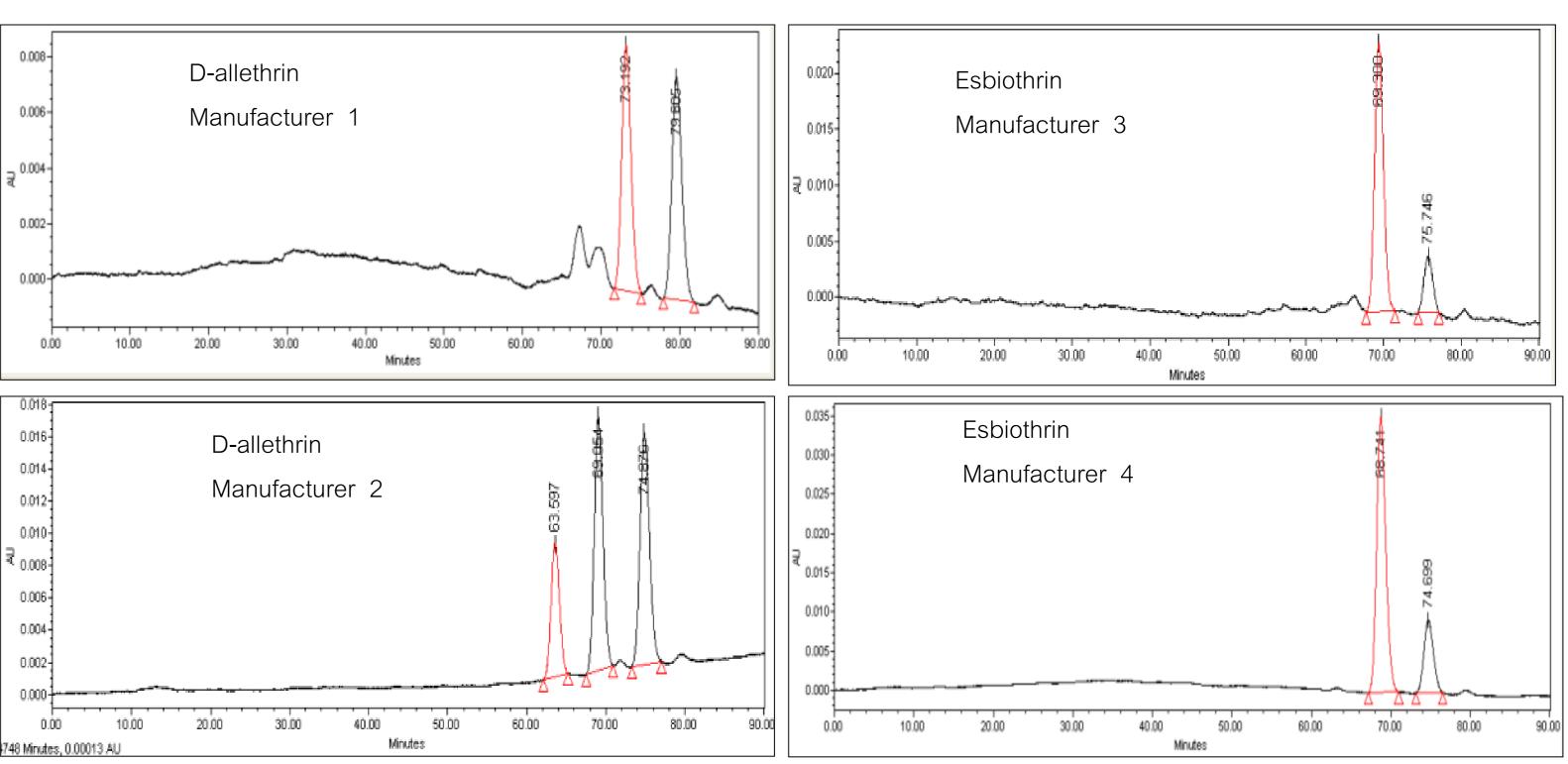




• GC - Chromatogram



HPLC - Chromatogram



Conclusions

- The identification of allethrin isomers by HPLC before select the reference standard helpful to increase the acuracy of quantitative results by GC and decrease the repeat of analysis.
- This study can be apply for analysis of allethrin isomer in other formulation.