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DETERMINATION GLYPHOSATE RESIDUE IN WATER BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

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ABSTRACT

A rapid, accurate and highly sensitive HPLC was used to estimate the residue of the herbicide glyphosate in water without any interference with other organic compounds. Tetrahydrofuran (THF) proved to be the best solvent for extraction the glyphosate residue even the lower concentrations. The extraction was carried out on reversed solid phase with 0.005 ppm as detection limit. Glyphosate residues in Al-Radhwania, Abo-Ghraib, Khan Dhari, Garmma and Zuobaa water were 0.05, 0.13, 0.17,18 and 0.09 ppm respectively.

INTRODUCTION

Searching for pesticide residues in plant, soil, water and others required sufficient equipments, certain solvents, experts and standards. Thus, the major task of the analysis process is to proved reliable and cost effective methods (3). However, both gas chromatography (GC) and high-performance liquid chromatography (HPLC) proved to be good options for pesticide monitoring (3, 5, 8 and 10). Since the disadvantage of gas chromatography is its limitation to volatile organophosphorous and thiocarbamite pesticides, hydroxyl derivatives could not be analyse directly by GC (9). Therefore, HPLC is quite suitable and better than GC when acidic pesticides with high polarities, low vitalities and thermal derividization steps were analyzed (6, 7). Meantime, acidic pesticides are strongly absorbed in UV region when liquid chromatography is used (2).

Glyphosate (Ground Up) might be the best well-known herbicide used in yards, gardens and other nonagricultural areas (1). To detect the residue of such herbicide, N-phospho nmethyl glycine should be determined in plants, soil and water. Therefore, the solid-phase extraction (SPE) mini column is usually used on the samples before running the analysis test. However, SPE had been widely used in food analysis for trace residues of pesticides and aflatoxins (4).

The study reported here is aimed to utilize the HPLC for accurate estimation of glyphosate in water of five different regions near Baghdad.

MATERIALS AND METHODS

Glyphosate residues in water were carried out on reversed phase, HPLC model LC-6A Shimadzu (Kyoto, Japan). The whole system was controlled by SCI-6A using UV- Visible spectrophotometer. The eluted GP was detected at 240 nm. Tetrahydrofuran (THF) and Ethyl acetate and dionized water HPLC grade were used without further purification.

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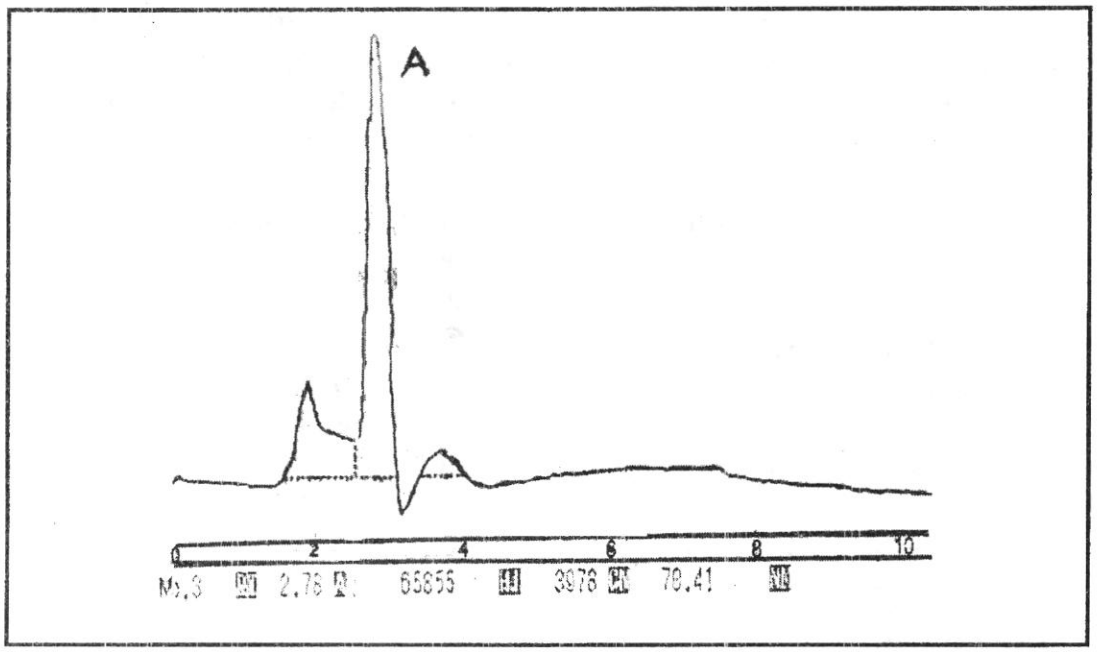
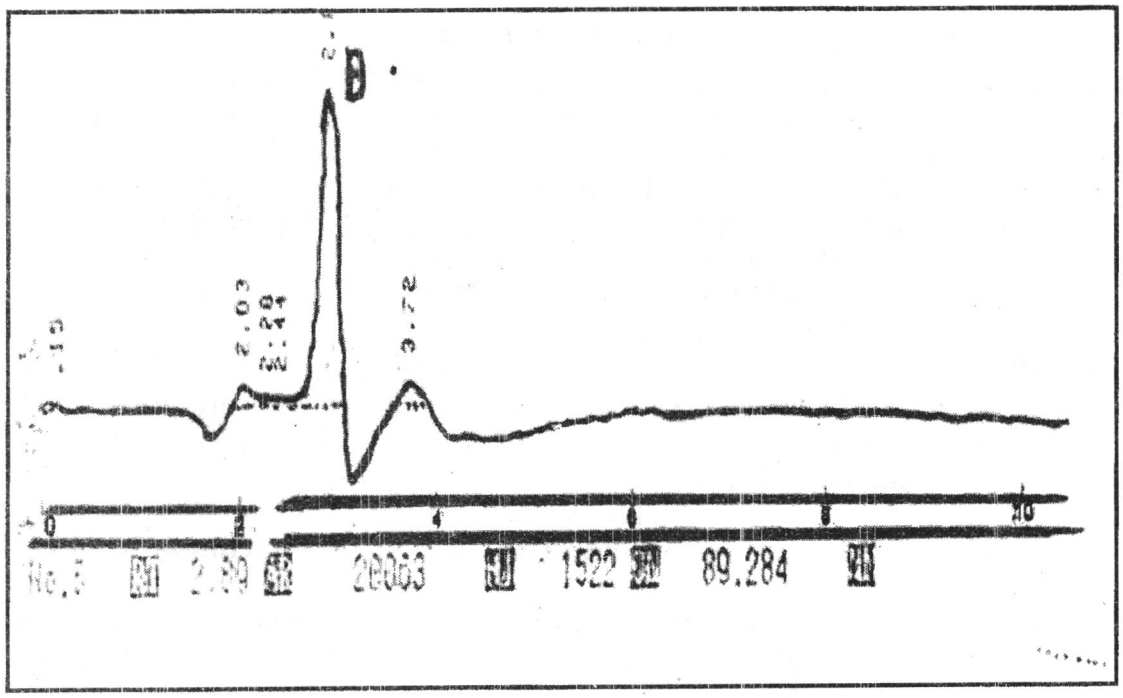
Meantime, the water during the experimental works was deionized. Water samples (1 liter/each) from five different locations near Baghdad (Radhwania, Abo-Ghraib, Khan Dhari, Garmma and Zuobaa) were collected after glyphosate had been used as weed herbicide. The water was pre-concentrated by passing it through the minicolumn cartridge Seppak C-18 (250 x 4.6 mm i.d) supplied with 5µm particle size to retain the glyphosate. The mobile phase was Tetrahydrofuran with flow rate 1ml/min. The glyphosate then was eluted by 5 ml of absolute ethanol. Twenty µl from each pre-concentrated water sample was injected in HPLC. The General Directorate obtained the standard Glyphosate from Aldrich Company (USA) for Animal Resource Laboratory/ Ministry of Agriculture.

RESULTS AND DISCUSSION

The isocratic separation of standard glyphosate (5ppm) on reversed phase C-18 column was optimized to obtain a baseline separation peak with retention time 2.78 minutes as shown in figure (1). The glyphosate concentrations in water samples were calculated by comparing the peak area of the sample with that obtained from the standard (11).The 5 ppm of standard glyphosate, which was adequate for this test, had been serially diluted with Tetrahydrofuran and chromatographed under the same condition to obtain the linearity with detection limit not more than 0.5 ppm. The method was valid since the concentrations used between 0.005 and 0.5 ppm. The separation method used in this investigation showed good linearity by plotting the relationship between the peak area and the concentrations used as showed in figure (2).

Concerning the glyphosate residues in those different locations, the herbicide was there in all location with high concentration (18 ppm) in Garmma site. It was 0.05, 0.13, 0.15 and 0.09 ppm in Radhwania, Abo-Ghraib, Khan-Dhari and Zuobaa sites respectively.





(A):Standard separated on C-18 column 4.6mmi.d×(250) column
(B):Glyophosate separated from water sample.
Mobile phase, Tetrahy drofuran (THF), flow rate 1ml/min, temperature 30°UV detection as 240nm.

Figure1: Chromatogram of standard glyophosate pesticide on

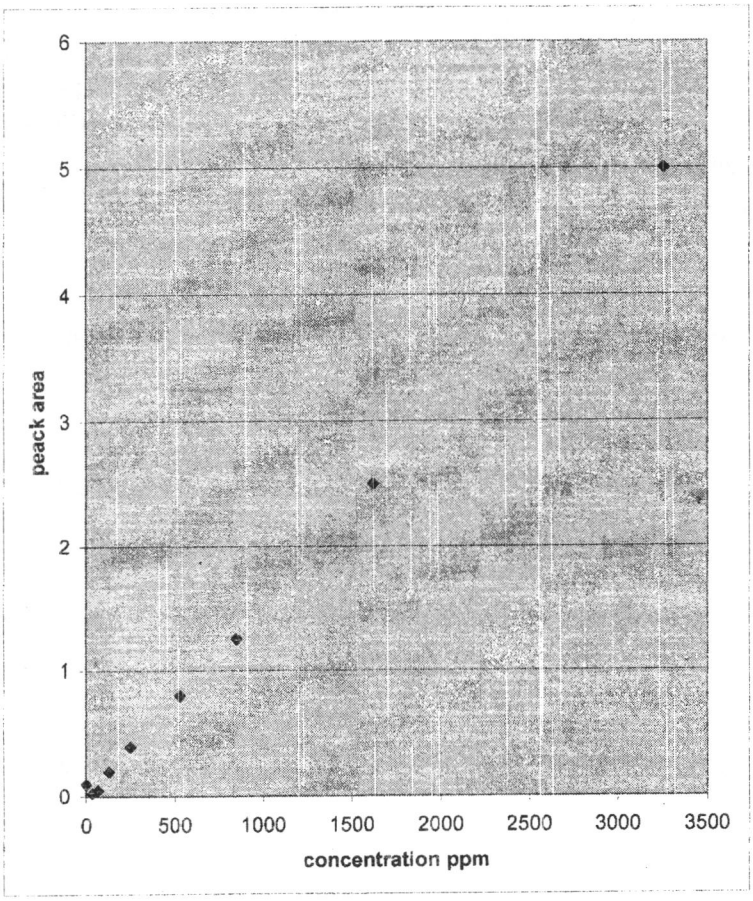


Figure 2: Calibration curve of Glyophosate

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تقدير بقايا مبيد الأدغال كلايفوسيت في الماء بوساطة جهاز السائل الكروموتوغرافي عالي الأداء

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الملخص

استخدم جهاز السائل الكروموتوغرافي عالي الأداء الذي يتصف بالسرعة والحساسية العالية في تقدير بقايا مبيد الأدغال كلايفوسيت بدون أية تداخلات مع المركبات العضوية الأخرى. أثبتت الدراسة أفضلية المذيب تتراهايدروفيوران لاستخلاص بقايا المبيد على الطور الصلب المعكوس وبكفاءة كشف بلغت ٠,٠٠٥ جزء بالمليون. أشارت النتائج إلى وجود ٠,٠٥، ٠,١٣، ٠,١٧، ١٨ و٠,٠٩ جزء بالمليون من المبيد في مياه مناطق الرضوانية، ابو غريب، خان ضاري، الكرمة وزوبع على التوالي.

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